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Remedial Investigation Sampling and Analysis Plan for J-Field, Aberdeen Proving Ground, Maryland

Volume 2: Quality Assurance Project Plan

by S. Prasad, L. Martino, and T. Patton

Environmental Assessment Division,
Argonne National Laboratory, 9700 South Cass Avenue, Argonne, Illinois 60439

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FOREWORD

This document presents the Quality Assurance Project Plan for the characterization field work to be conducted as part of a remedial investigation (RI) to be carried out at J-Field, Aberdeen Proving Ground, Maryland, pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), as amended. The RI is to be conducted for the U.S. Army under the direction of the Directorate of Safety, Health, and Environment, Aberdeen Proving Ground. This report (Volume 2 of the *Remedial Investigation Sampling and Analysis Plan for J-Field, Aberdeen Proving Ground, Maryland*) is one in a series of documents prepared by Argonne National Laboratory to define the plans for RI activities at J-Field. Other documents in this series include a Remedial Investigation Work Plan (Benioff et al. 1995a); a Field Sampling Plan (Benioff et al. 1995b); and a Work Plan for the Focused Feasibility Study of the Toxic Burning Pits Area (Biang et al. 1995). Two other documents — an Ecological Risk Assessment and a Work Plan for the Feasibility Study — are in preparation.

NOTATION

The following is a list of acronyms and abbreviations, chemicals, and units of measure used in this document. Some acronyms used only in tables are defined in those tables.

ABBREVIATIONS AND ACRONYMS

AEC	U.S. Army Environmental Center
ANL	Argonne National Laboratory
AOC	area of concern
APG	Aberdeen Proving Ground
ASTM	American Society for Testing and Materials
BNA	base neutral acid extractable compound
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (as amended)
CLP	Contract Laboratory Program
CLPAS	Contract Laboratory Program Analytical Suite
COC	chain of custody
COE	U.S. Army Corps of Engineers
Cp/MS	Curie-point pyrolizer mass spectrometer
CRDEC	Chemical Research Development and Engineering Center
CRDL	contract-required detection limit
CRL	certified reporting limit
CRQL	contract-required quantitation limit
CTR	contract technical representative
CVAA	cold vapor atomic absorption
CWA	chemical warfare agent
DANC	decontaminating agent, noncorrosive
DNAPL	dense, nonaqueous-phase liquid
DOT	U.S. Department of Transportation
DQO	data quality objective
DSHE	Directorate of Safety, Health, and Environment (U.S. Army)
ECC	environmental chemistry coordinator
EDL	estimated detection limit
EDXRF	energy-dispersive X-ray fluorescence
EM	electromagnetic
EMD	Environmental Management Division (Aberdeen Proving Ground)
EMI	electromagnetic induction
EPA	U.S. Environmental Protection Agency
ERA	ecological risk assessment
FFS	focused feasibility study
FPD	flame-photometric detector

FS	feasibility study
FSP	Field Sampling Plan
GC	gas chromatography
GC/AID	gas chromatography/argon ionization detector
GC-ECD	gas chromatography with electron-capture detection
GC-FPD	gas chromatography with flame-photometric detection
GC/MS	gas chromatography/mass spectrometry
GFAA	graphite furnace atomic absorption spectroscopy
GPR	ground-penetrating radar
GPS	global positioning system
HASP	Health and Safety Plan
HPLC	high-pressure liquid chromatography
HPLC-EV/UV	high-pressure liquid chromatography with pulsed electrochemical detector and confirmation by ultraviolet detector
HRGC/LRMS	high-resolution capillary column gas chromatography/low-resolution mass spectrometry
HSO	health and safety officer
IC	ion chromatography
ICF	ICF-Kaiser Engineers
ICP	inductively coupled plasma spectroscopy
IC-UV	ion chromatography with an ultraviolet detector
IRDMS	Installation Restoration Data Management System
IRM	interim reference material
IRP	Installation Restoration Program
LOC	level of concern
LOEL	lowest observed effective level
MCA	multichannel analyzer
MDE	Maryland Department of the Environment
MDL	method detection limit
MPIC-EC/UV	mobile phase ion chromatography with combination ultraviolet and pulsed electrochemical detector
NIST	National Institute of Standards and Technology
OB	open burning
OD	open detonation
OSL	on-site laboratory
OVA	organic vapor analyzer
PB	Prototype Building
PDC	project data coordinator
PID	photoionization detector
PL	prime laboratory
PQL	practical quantitation limit

QA	quality assurance
QAO	quality assurance officer
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QC	quality control
RCP	Riot Control Burning Pit
RI	remedial investigation
RI/FS	remedial investigation/feasibility study
RL	referee laboratory
RPD	relative percent difference
RPDG	Robins Point Demolition Ground
RPTS	Robins Point Tower Site
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SARM	standard analytical reference material
SBDG	South Beach Demolition Ground
SBT	South Beach Trench
SOP	standard operating procedure
SOW	Statement of Work
TAL	Target Analyte List
TBD	to be determined
TBP	Toxic Burning Pits
TCL	Target Compound List
URL	upper reporting limit
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
USCS	Unified Soil Classification System
USGS	U.S. Geological Survey
UV	ultraviolet
UXO	unexploded ordnance
VTSR	verified time of sample receipt
WPP	White Phosphorus Burning Pits
XRF	X-ray fluorescence

CHEMICAL SYMBOLS AND ABBREVIATIONS

BHC	benzene hexachloride (insecticide)
BNA	base neutral acid extractable (organic compounds)
CH_4	methane
CK	cyanogen chloride
CN	chloroacetophenone
CS	o-chlorobenzylidine malononitrile/orthochlorobenzalmalononitrile
DCE	combined dichloroethylenes
12DCE	1,2-dichloroethylene
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DIMP	diisopropylmethylphosphonate
DM	adamsite
DMMP	dimethylmethylphosphonate
24DNT	2,4-dinitrotoluene
HMX	cyclotetramethylene tetranitramine
HNO_3	nitric acid
H_2SO_4	sulfuric acid
I-C_4H_8	isobutylene
MEK	methyl ethyl ketone (2-butanone)
NaI	sodium iodide
NaOH	sodium hydroxide
$(NH_4)_2S_2O_3$	ammonium thiosulfate
PAH	polyaromatic hydrocarbon
PCB	polychlorinated biphenyl
PETN	pentaerythritol tetranitrate
PVC	polyvinyl chloride
PWP	plasticized white phosphorus
RDX	cyclotrimethylene trinitramine
SVOC	semivolatile organic compound
TCE	combined trichloroethanes
112TCE	1,1,2-trichloroethane
TCLEA	1,1,2,2-tetrachloroethane
TCLEE	tetrachloroethylene
TCPU	bis(2,4,6-trichlorophenyl)urea
TETRYL	*n*-methyl-*n*-2,4,6-tetranitrobenzoaniline
TEX	toluene, ethylbenzene, and xylenes
246TNT	2,4,6-trinitrotoluene

TOC	total organic carbon
TOX	total organic halogen
TPH	total petroleum hydrocarbon
trans-12DCE	trans-1,2-dichloroethylene
TRCLE	trichloroethylene
VOC	volatile organic compound
VX	*o*-ethyl S-(2-diisopropylaminoethyl methylphosphonothioate) = methylphosphonothioic acid, a nerve agent
WP	white phosphorus

UNITS OF MEASURE

cm	centimeter(s)
Ci	curie(s)
pCi	picocurie(s)
d	day(s)
°C	degree(s) Celsius
eV	electron volt(s)
°F	degree(s) Fahrenheit
ft	foot (feet)
ft^2	square foot (feet)
g	gram(s)
kg	kilogram(s)
mg	milligram(s)
µg	microgram(s)
ng	nanogram(s)
gal	gallon(s)
in.	inch(es)
L	liter(s)
mL	milliliter(s)
m	meter(s)
mm	millimeter(s)
µm	micrometer(s)
µmho	micromho(s)
nm	nanometer(s)
mi	mile(s)
mrem	millirem(s)
oz	ounce(s)
ppb	part(s) per billion
ppm	part(s) per million
yd	yard(s)
yr	year(s)

1 INTRODUCTION

1.1 PURPOSE AND SCOPE

This document is the Quality Assurance Project Plan (QAPjP) (Volume 2 of the Sampling and Analysis Plan [SAP]) for remedial investigation (RI) activities at J-Field, Aberdeen Proving Ground (APG), Maryland. The QAPjP is intended to ensure that the RI sampling and analysis activities described in the Field Sampling Plan (FSP) (Volume 1 of the SAP) are appropriately scoped and performed to obtain quality data. The QAPjP has been prepared in accordance with the guidelines established by the U.S. Environmental Protection Agency (EPA) (1980a, 1984a, 1989a); U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) (1990), predecessor to the U.S. Army Environmental Center (AEC); and Argonne National Laboratory (ANL 1988; Pentecost and Doctor 1990). The procedures presented in this QAPjP are derived in part from ICF-Kaiser Engineers (1992a). Health and safety precautions and procedures for the J-Field RI are presented in the Health and Safety Plan (HASP) for the J-Field activities.

1.2 REPORT ORGANIZATION

Section 1 summarizes the purpose and scope of the QAPjP and outlines the organization of the report. Section 2 discusses the scope and objectives of the RI activities, including the ecological risk assessment, site background, and field operations. A tentative field schedule and summaries of on-site and off-site laboratory analyses are also provided.

Section 3 outlines the project organization and details the responsibilities of ANL and its subcontractors. Section 4 lists data quality objectives (DQOs) for Stage I RI activities and sets quality assurance (QA) objectives for accuracy, precision, representativeness, completeness, and comparability.

Section 5 discusses sample labeling, sample containers, and sample preservation. References to appropriate standard operating procedures (SOPs) and other resources that outline field sampling procedures are given. The types of quality control (QC) samples and frequency of collection are also discussed. Section 6 describes chain-of-custody (COC) procedures, requirements for packaging and handling samples to be shipped off-site for analysis, sample storage during screening for chemical warfare agents (CWAs), and requirements for disposal of investigation-derived waste.

Section 7 discusses calibration procedures (including frequency of calibration) for field instruments and specifies requirements for calibrating laboratory instruments. Section 8 describes field screening procedures and analytical procedures both for on-site and off-site laboratories.

Section 9 discusses procedures and requirements for data validation, reduction, and reporting. Section 10 summarizes the types and frequency of QC samples to be used for field

sampling and laboratory analysis. Section 11 describes procedures for conducting performance and system audits for field sampling and laboratory analyses. (A system audit checklist is provided in Appendix B.) Section 12 specifies requirements for preventive maintenance for field and laboratory equipment.

Section 13 discusses the requirements for recording sampling and field investigation data. Requirements for laboratory records are also discussed. Section 14 discusses statistical techniques to be used in assessing the quality of data collected throughout the J-Field RI. This discussion includes data accuracy and precision, confidence limits, and estimation of trends.

Section 15 lists corrective action protocols to be followed if problems arise during sample collection and analysis. Section 16 discusses periodic internal QA reports to be made to ANL management and provides a list of documents and deliverables to be submitted to the Directorate of Safety, Health, and Environment (DSHE) and to the AEC (formerly USATHAMA). Documents referenced in this report are listed in Section 17, and a list of preparers is given in Section 18.

Appendix A presents selected SOPs prepared for implementation during the RI activities at J-Field. Additional SOPs listed in Appendix A are developed by the U.S. Army Corps of Engineers (COE) Waterways Experiment Station (COE 1993). These SOPs are periodically updated and are accessible through the Internet. Appendix B is a sample system audit checklist.

2 PROJECT DESCRIPTION

2.1 SITE BACKGROUND

J-Field encompasses about 460 acres at the southern end of the Gunpowder Neck Peninsula in the Edgewood Area of APG (Figure 2.1). Since World War II, the Edgewood Area of APG has been used to develop, manufacture, test, and destroy chemical agents and munitions. These materials were destroyed at J-Field by open burning and open detonation (OB/OD). For the purposes of this project, J-Field has been divided into eight geographic areas or facilities that are designated as areas of concern (AOCs): the Toxic Burning Pits (TBP), the White Phosphorus Burning Pits (WPP), the Riot Control Burning Pit (RCP), the Robins Point Demolition Ground (RPDG), the Robins Point Tower Site (RPTS), the South Beach Demolition Ground (SBDG), the South Beach Trench (SBT), and the Prototype Building (PB). The site background and environmental setting are summarized in Sections 1 and 2 of the FSP (Volume 1 of the SAP).

2.2 PROJECT SCOPE

The scope of this project is to conduct a remedial investigation/feasibility study (RI/FS) and ecological risk assessment to evaluate the impacts of past disposal activities at the J-Field site. Sampling for the RI will be carried out in three stages (I, II, and III) as detailed in the FSP. A phased approach will be used for the J-Field ecological risk assessment (ERA).

2.3 PROJECT OBJECTIVES

2.3.1 Remedial Investigation

The RI activities at J-Field will be conducted in three stages. Stage I soil/sediment sampling activities are designed so that the results, combined with existing data, can be used to determine locations and depths free from CWAs or unexploded ordnance (UXO) and to choose sampling locations for off-site Contract Laboratory Program (CLP) analyses. In addition, these activities can be used to help determine the nature and extent of contamination sources. Stage I soil/sediment activities may require several rounds or phases of sampling in which the area sampled is progressively extended until sampling shows little or no contamination. The Stage I soil/sediment sampling stops with this last phase as both the lateral extent and depth of the contaminated region are delineated by the results of this and previous rounds of Stage I sampling. The Stage I phasing is potentially applicable to sampling activities for soil, sediment, and soil gas. The existing soil-gas data, which show soil-gas contamination out to the edge of the areas surveyed, exemplify the need for this use of phasing.

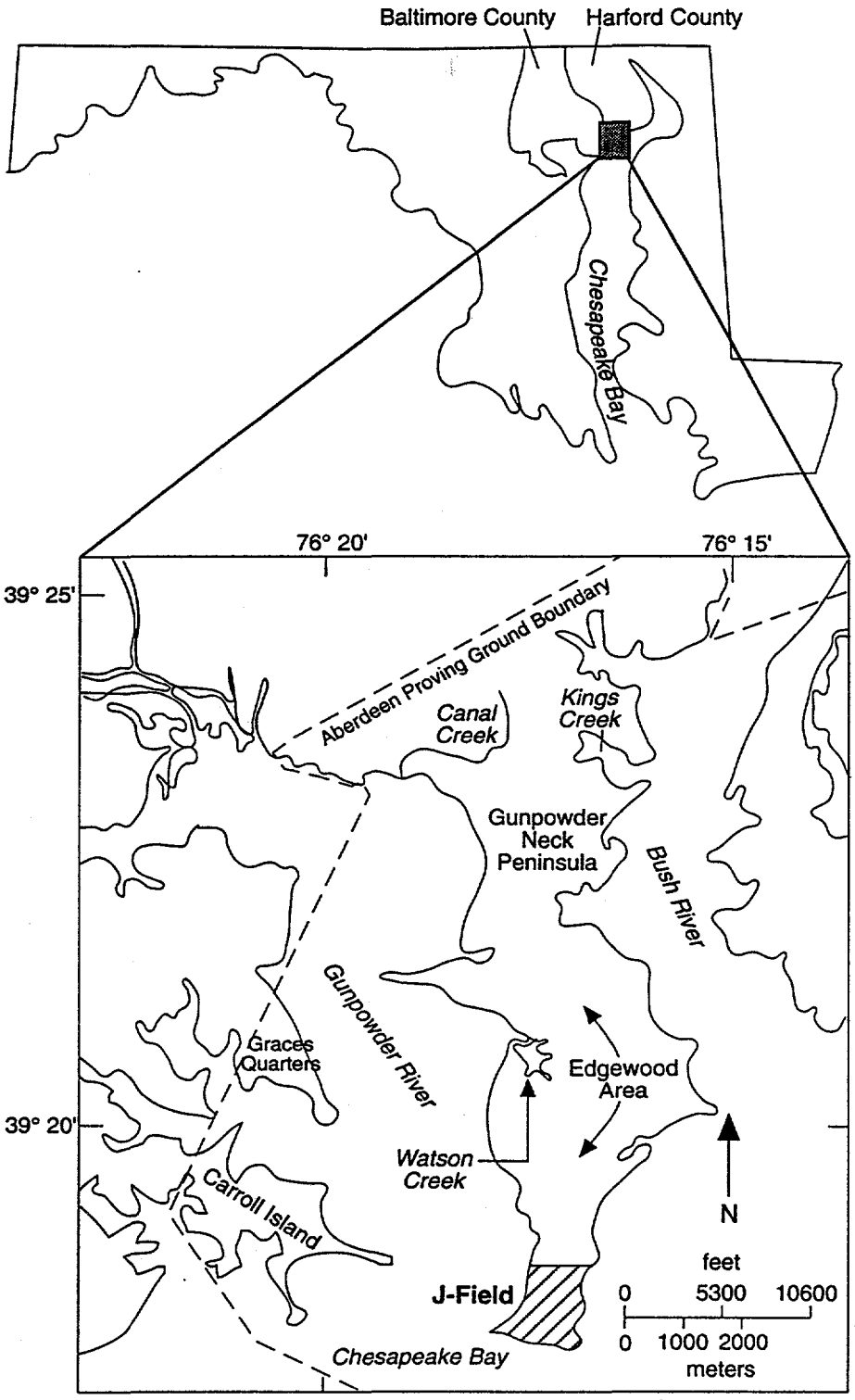

FIGURE 2.1 Location of J-Field in the Edgewood Area at APG (Source: Adapted from Hughes 1993)

The goal of Stage II activities for soil and sediment sampling is characterization of contamination within the areas delineated by the phases of Stage I sampling (and/or verification that apparently uncontaminated areas are "clean") to the extent needed to carry out human health and ecological risk analyses. Both the analytes and the choice of sampling locations will be based on the Stage I results. At this time, it is assumed that analytical methods will include off-site analyses of soils and sediments with CLP and AEC methods yielding data of EPA analytical levels III, IV, or V.

Stage II sampling activities for soil, sediment, surface water, and groundwater will consist of collecting additional samples of environmental media and collecting additional samples from existing wells. Stage I results will be used to determine the location and number of additional samples and additional monitoring wells (if any) and any changes in the analytical suites. The groundwater, soil, and sediment samples for Stage II will be analyzed off-site to provide EPA level III, IV, or V data. These data will be combined with the Stage I data to determine the extent of areas of contaminated surface water or of plumes of contaminated groundwater, to determine the extent of contaminated soil and sediment, and to characterize the contamination within those areas or plumes.

In Stage III, sampling and analyses strategies will be developed, if necessary, to ensure that the desired DQOs for remedial alternative selection have been achieved in the previous stages.

Essentially no background samples will be collected in any of the stages. Instead, data collected by APG as part of a sitewide background study and other background data collected by APG contractors will be used.

2.3.2 Ecological Risk Assessment

An ERA will be conducted at J-Field to answer the following questions:

1. Are contaminants in J-Field environmental media now producing demonstrable ecological effects at the population, community, or ecosystem level and, if so, what are the extent and magnitude of the effects?

2. Are contaminated environmental media at J-Field directly toxic to biota and, if so, what type of organisms are affected and where do they live?

3. Are contaminants in environmental media at J-Field capable of producing effects on biota through bioaccumulation, food chain effects, or other secondary mechanisms and, if so, what is the pathway of exposure and the extent and magnitude of risk?

The results of the ERA will also help to determine if any remedial action is necessary and to evaluate the risk that remedial actions may pose to the biological community.

As part of Phase 1 of the ERA, biotic surveys will be conducted at contaminated sites within J-Field and at reference sites to determine whether ecological effects from contamination are evident and whether the media from the primary areas of contamination at J-Field are toxic to biota. The surveys will include terrestrial, open-water, and wetland habitats. In addition, residual analysis of biota will be conducted to assess short-term and long-term exposures to contaminants. The results of the biotic surveys and residual analyses will be an important part of the pathway analysis that will take place during Phase 3 of the ERA. Contaminated areas will be identified by an on-site analytical suite and rapid toxicity screening as described in the FSP.

Phase 2 will consist of in-situ and laboratory toxicity testing to determine which organisms are affected by contaminated environmental media and to assess the extent of contamination and the magnitude of its effect. Phase 3 will consist of pathway analysis and other risk assessment procedures appropriate to the nature of effects determined in Phases 1 and 2. The activities to be conducted as part of the ERA are summarized in the Ecological Risk Assessment Work Plan.

2.4 FIELD OPERATIONS

2.4.1 Schedule

Field activities to be conducted during Stage I of the RI/FS include conducting land surveys and establishing grids; surveying geophysical conditions; surveying soil-gas levels; installing a tide gauge; installing groundwater monitoring wells; measuring groundwater and tidal levels; and collecting environmental media samples. The field activities are expected to begin in February 1993 and continue until July 1995. Quarterly groundwater sampling is already in progress (by the U.S. Geological Survey [USGS]) and will be extended for at least two to three more quarters (by ANL).

2.4.2 Sampling Activities

The first round of Stage I will include the following activities:

- UXO/CWA surveys and field surveys of the AOCs;
- Video inspection of the several USATHAMA and Princeton Aqua Science wells;
- The abandonment, if necessary, of damaged or unusable wells (in accordance with Maryland Department of the Environment [MDE] well-abandonment procedures);
- Installation of a tidal gauge near the RPTS AOC;

- Performance of a topographic survey and the generation of a site map for each AOC;
- Performance of a variety of geophysical surveys, including (but not limited to) ground-penetrating radar (GPR), electrical conductivity, electrical resistivity depth soundings, magnetics, seismic reflection, and seismic refraction;
- Implementation of a groundwater and surface water monitoring program using CLP-certified methods;
- Installation of a monitoring well; and
- Analysis of soil, soil-gas, and sediment samples, with field screening and field analytical methods augmented with the off-site analyses of samples with CLP-certified methods. (Tables 2.1 through 2.5 define the chemical analytical methods, parameters, matrix type, and number of samples [including QC samples] to be collected for chemical analysis.)

The geophysical surveys and soil-gas surveys will be used to locate buried UXO or CWA containers and other buried items, to help verify the locations of former OB/OD areas, and to aid in the exploration of unknown contamination sources. The soil-gas surveys will also be useful because of the reported widespread use of DANC (decontaminating agent, noncorrosive) as a decontaminating agent for CWA. (DANC contained about 90-95% 1,1,2,2-tetrachloroethane [TCLEA] [Nemeth 1989].)

TABLE 2.1 Summary of Off-Site Laboratory Chemical Analysis Strategy for Groundwater Samples

Analyte	Method[a]	Number of Samples	Number of QC Samples		
			Duplicates	Rinse Blanks	Ambient Field Banks
Full complement of organic constituents (Target Compound List)[b]					
Volatile compounds	CLP (OLM01.0): GC/MS	48	5	5	5
Semivolatile compounds	CLP (OLM01.0): GC/MS	48	5	5	5
Polychlorinated biphenyls (PCBs)/Aroclors	CLP (OLM01.0): GC/MS	48	5	5	5
Pesticides	CLP (OLM01.0): GC/MS	48	5	5	5
Inorganic constituents (Target Analyte List)[b]					
Total metals	CLP (ILM02.0): ICP GFAA (As, Pb, Se, Tl), CVAA (Hg)	48	5	5	5
Cyanide	Colorimetry[c]/titration	48	5	5	5
CWA degradation products	GC-FPD, HPLC-EV/UV, IC-UV, MPIC-EC/UV, GC/MS	48	5	5	5
Explosives and explosives-related compounds	HPLC-UV	48	5	5	5
Total organic halogen (TOX)	EPA Method 9020 (SW 846)[d]	48	5	5	5
Total organic carbon (TOC)	EPA Method 9060 (SW 846)	48	5	5	5
Conductivity	EPA Method 9050 (SW 846)	48	5	5	5
Major cations and anions	TBD	48	5	5	5
Radioactivity (gross alpha and beta)	EPA Method 9310 (SW 846) and/or EPA 1979	48	5	5	5

[a] Abbreviations:

CLP = Contract Laboratory Program,
CVAA = cold vapor atomic absorption,
GC-FPD = gas chromatography with flame photometric detection,
GC/MS = gas chromatography/mass spectrometry,
GFAA = graphite furnace atomic absorption spectroscopy,
HPLC-EV/UV = high-pressure liquid chromatography with a pulsed electrochemical detector and confirmation by ultraviolet detector,
ICP = inductively coupled plasma spectroscopy,
IC-UV = ion chromatography with an ultraviolet detector,
MPIC-EC/UV = mobile phase ion chromatography with combination ultraviolet and pulsed electrochemical detector,
TBD = to be determined.

[b] CLP SOW ILM 02.0; low or medium concentration.

[c] USATHAMA, Method D-THAMA-METH.2/TRIN.1.

[d] EPA (1986a).

TABLE 2.2 Summary of Stage I On-Site Analysis Strategy for Surface Soil Samples

Analyte	Method[a,b]	Number of Samples	Number of QC Samples		
			Duplicates	Rinse Blanks	Ambient Field Blanks
CWAs					
Gross screening (on-site)	TBD	122	12	12	12
Sensitive screening (off-site)	TBD	TBD	TBD	TBD	TBD
Volatile organic compounds (VOCs)	Headspace analysis EPA Method 3810 (SW 846)[c]	122	12	12	12
Soil Gas — VOCs					
Active	Portable GC-ECD	122	12	12	12
Passive	Petrex survey	122	12	12	12
Total petroleum hydrocarbons (TPHs)	Colorimetric immunochemistry	122	12	12	12
Total polyaromatic hydrocarbons (PAHs)	Colorimetric immunochemistry	122	12	12	12
Metals	X-ray fluorescence (XRF)	122	12	12	12
Nitroaromatics — explosives	Colorimetry[d]	122	12	12	12
PCBs	Colorimetric immunochemistry	122	12	12	12
Toxic chemicals	MicroTox (bioluminescence)	122	12	12	12
Soil microbes	MICKIT (bacterial count)	122	12	12	12
Radioactivity (gross alpha and beta)	EPA Method 9310 (SW 846)	20	2	1	1

[a] Abbreviations: TBD = to be determined; GC-ECD = gas chromatography with electron-capture detection.

[b] See also SOPs (Appendix A).

[c] EPA (1986a).

[d] USATHAMA, Method D-THAMA-METH.2/TRIN.1.

TABLE 2.3 Summary of Stage I On-Site Analysis Strategy for Subsurface Soil Samples

Analyte	Method[a]	Number of Samples	Number of QC Samples: Duplicates	Rinse Blanks	Ambient Field Blanks
CWAs					
Gross screening (on-site)	TBD[b]	131	13	13	13
Sensitive screening (off-site)	TBD	TBD	TBD	TBD	TBD
VOCs	Headspace analysis EPA Method 3810 (SW 846)[c]	131	13	13	13
TPHs	Colorimetric immunochemistry	131	13	13	13
Total PAHs	Colorimetric immunochemistry	131	13	13	13
Metals	XRF	131	13	13	13
Nitroaromatics — explosives	Colorimetry[d]	131	13	13	13
PCBs	Colorimetric immunochemistry	131	13	13	13
Toxic chemicals	MicroTox (bioluminescence)	131	13	13	13
Soil microbes	MICKIT (bacterial count)	131	13	13	13

[a] See also SOPs (Appendix A).

[b] TBD = to be determined.

[c] EPA (1986a).

[d] USATHAMA, Method D-THAMA-METH.2/TRIN.1.

TABLE 2.4 Summary of Stage I On-Site Analysis Strategy for Sediment Samples (including surface sediment and sediment boring samples)

Analyte	Method[a]	Number of Samples	Number of QC Samples		
			Duplicates	Rinse Blanks	Ambient Field Blanks
CWAs					
Gross screening (on-site)	TBD[b]	65	6	6	6
Sensitive screening (off-site)	TBD	TBD	TBD	TBD	TBD
VOCs	Headspace analysis EPA Method 3810 (SW 846)[c]	65	6	6	6
TPHs	Colorimetric immunochemistry	65	6	6	6
Total PAHs	Colorimetric immunochemistry	65	6	6	6
Metals	XRF	65	6	6	6
Nitroaromatics — explosives	Colorimetry[d]	65	6	6	6
PCBs	Colorimetric immunochemistry	65	6	6	6
Toxic chemicals	MicroTox (bioluminescence)	65	6	6	6
Soil microbes	MICKIT (bacterial count)	65	6	6	6

[a] See also SOPs (Appendix A).

[b] TBD = to be determined.

[c] EPA (1986a).

[d] USATHAMA, Method D-THAMA-METH.2/TRIN.1.

TABLE 2.5 Summary of Off-Site Laboratory Chemical Analysis Strategy for Surface Water Samples

Analyte	Method[a]	Number of Samples	Number of QC Samples: Duplicates	Rinse Blanks	Ambient Field Banks
Full complement of organic constituents (Target Compound List)[b]					
Volatile compounds	CLP (ILM02.0): GC/MS	44	4	4	4
Semivolatile compounds	CLP (ILM02.0): GC/MS	44	4	4	4
PCBs/Aroclors	CLP (ILM02.0): GC/MS	44	4	4	4
Pesticides	CLP (ILM02.0): GC/MS	44	4	4	4
Inorganic constituents (Target Analyte List)[b]					
Total metals	CLP (ILM02.0): ICP GFAA (As, Pb, Se, Tl), CVAA (Hg)	44	4	4	4
Cyanide	Colorimetry[c]/titration	44	4	4	4
CWA degradation products	GC-FPD, HPLC-EV/UV, IC-UV, MPIC-EC/UV, GC/MS	44	5	4	4
Explosives and related compounds	HPLC-UV	44	4	4	4
TOX	EPA Method 9020 (SW 846)[d]	44	4	4	4
TOC	EPA Method 9060 (SW 846)	44	4	4	4
Conductivity	EPA Method 9050 (SW 846)	44	4	4	4
Major cations and anions	TBD	44	4	4	4
Radioactivity (gross alpha and beta)	EPA Method 9310 (SW 846) and/or EPA 1979	44	4	4	4

[a] Abbreviations:

CLP = Contract Laboratory Program,
CVAA = cold vapor atomic absorption,
GC-FPD = gas chromatography with flame photometric detection,
GC/MS = gas chromatography/mass spectrometry,
GFAA = graphite furnace atomic absorption spectroscopy,
HPLC-EV/UV = high-pressure liquid chromatography with a pulsed electrochemical detector and confirmation by ultraviolet detector,
ICP = inductively coupled plasma spectroscopy,
IC-UV = ion chromatography with an ultraviolet detector,
MPIC-EC/UV = mobile phase ion chromatography with combination ultraviolet and pulsed electrochemical detector,
TBD = to be determined.

[b] CLP SOW ILM 02.0; low or medium concentration.

[c] USATHAMA, Method D-THAMA-METH.2/TRIN.1.

[d] EPA (1986a).

3 PROJECT ORGANIZATION AND RESPONSIBILITIES

3.1 PROJECT ORGANIZATION

As the principal investigator for the J-Field RI/FS, and in accordance with the conditions set forth by APG, ANL will implement the J-Field RI Work Plan and maintain overall control of RI field activities performed by ANL subcontractors. The ANL organizational structure for the RI project is shown in Figure 3.1. All agreements and modifications regarding implementation of the RI Work Plan will be made between APG and ANL, and ANL and APG will perform all contract services required to implement the Work Plan, including field operations, laboratory testing, and data management.

3.2 RESPONSIBILITIES

3.2.1 Argonne National Laboratory

The ANL program managers and project manager will form the ANL program management team. John Ditmars and Andrew Anderson will be the ANL program managers. They will be authorized to commit ANL's resources to accomplish the project objectives and will represent ANL in all contractual matters with APG. The program managers will ultimately be responsible for all contractual matters and for ANL and subcontractor performance.

The ANL project manager, Lou Martino, will report directly to the ANL program managers. He will be responsible for the day-to-day direction and management of ANL's field, laboratory, and office activities related to the APG project and will oversee the activities of ANL's subcontractors. He will use his authority to coordinate and integrate the activities of other investigators working directly under the control of the APG project officer, including (but not limited to) the USGS, Roy F. Weston, Inc. (Weston), the EPA, and ICF-Kaiser Engineers. Martino will be authorized to procure necessary support services and equipment on behalf of APG, and subject to APG approval, to implement the FSP, the QAPjP, and the RI Work Plan. He will also be responsible for staffing, scheduling, and reporting all ANL activities.

The ANL project manager will be supported by staff and leaders of the RI team, the Feasibility Study (FS)/Focused Feasibility Study (FFS) team, and an ANL contracting officer, contract technical representative (CTR), and several subcontractors.

The ANL project quality assurance officer (QAO), Surya Prasad, will be responsible for all aspects of QA/QC during the implementation of the RI Work Plan. He will serve as liaison between the ANL project manager and the APG program QAO, all subcontractors, and contract laboratories. He will report directly to the ANL program managers when corrective action is required as a result of system and performance audits.

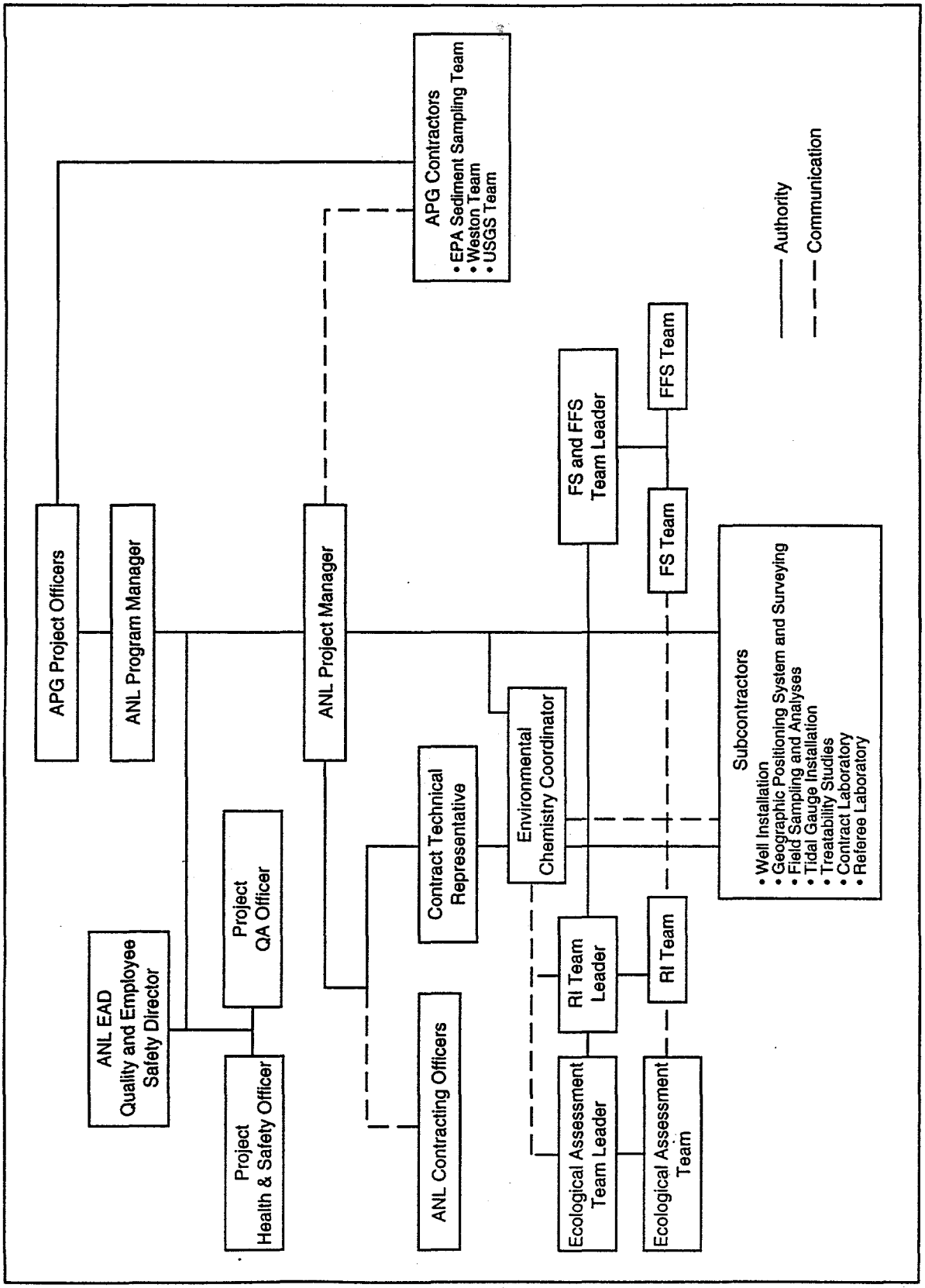

FIGURE 3.1 ANL Organizational Chart for the Remedial Investigation, J-Field, Aberdeen Proving Ground

An environmental chemistry coordinator (ECC) will be responsible for coordinating the RI sampling efforts, including collection, CWA screening, and analysis. The ECC will also serve as a liaison between the project QAO and the project manager. The RI team leader will be responsible for ensuring that all sampling and analysis activities are conducted according to the approved FSP and for preparing the RI report. The ERA team will implement the Ecological Risk Assessment Work Plan. The FS/FFS team leaders will be responsible for the FS and FFS, including ensuring that the RI results meet relevant and defensible DQOs (EPA 1981, 1984a, 1987a), and for FS and FFS report preparation.

Also as part of its RI responsibilities, ANL will establish, with the assistance and collaboration of the Chemical Research Development and Engineering Center (CRDEC) at APG, an on-site laboratory (OSL) at APG to perform certain Stage I analyses. The functions of the OSL are outlined in Section 3.2.2. The ECC, the RI field team leader, and the laboratory staff will form a core team for day-to-day laboratory operations, and the ANL project manager will have ultimate responsibility for the overall management of the OSL.

A designated ANL staff member will be authorized by the ANL contracting officer to function as the project CTR. The CTR will be responsible for directing and monitoring the activities of all ANL subcontractors and will be the primary liaison between the ANL contractors and the project manager.

The ANL project health and safety officer (HSO) will be responsible for all health and safety aspects related to RI Work Plan activities. The HSO will serve as the liaison between the ANL project manager, the related safety officers within APG, and the HSOs of all subcontractors. The HSO will report to the ANL contracting officer should corrective action be required of an ANL subcontractor. The HSO will also report directly to the ANL program managers should corrective action be required for activities conducted by ANL staff members.

3.2.2 Analytical Laboratories

The OSL to be set up by ANL at J-Field will provide initial screening and extraction of environmental samples for a variety of chemical and radiological parameters collectively referred to as the Stage I analytical suite. Part of this activity will be to screen for CWA in soil and sediment. These analytical activities, described as Stage I testing during RI, will assist in determining the design of the sampling plan for Stage II efforts, which will involve analyses of solid environmental media samples at off-site analytical laboratories with strict QA/QC procedures. No "referee" laboratory will be set up for confirmation of OSL analysis.

Two off-site analytical laboratories, hereafter referred to as the prime laboratory and the referee laboratory, will provide analytical services for aqueous environmental media samples (including groundwater and surface water) during RI Stages I, II, and III and for solid environmental media samples (including soil and sediment) during Stages II and III. The function of the prime laboratory will be to provide comprehensive testing of

environmental media samples for a variety of chemical parameters. The referee laboratory will provide analytical services similar to the prime laboratory, but only on a selected number of samples that are also tested at the prime laboratory. Data derived from the referee laboratory will be compared with data from the prime laboratory in support of the project QA protocols.

4 DATA QUALITY OBJECTIVES

Data quality objectives are qualitative and quantitative objectives developed by ANL that specify the type and quality of data to be collected during the RI activities at J-Field. The initial evaluation of J-Field, presented in the RI Work Plan, indicated that additional data are needed to characterize the J-Field site. Therefore, the first objective of the RI field activities at J-Field is to determine the nature and extent of contamination in the eight AOCs. Section 4.1 presents specific DQOs for Stages I and II field activities designed to initiate the task that will accomplish this objective.

The data collected during site characterization will also be used in the risk assessment that will determine if site contaminant levels pose any threats to human health and the environment. The results of the risk assessment will ultimately guide decisions on the need for remedial action. Section 4 of the RI Work Plan outlines the DQOs for the J-Field RI activities.

4.1 DATA QUALITY OBJECTIVES FOR STAGE I FIELD ACTIVITIES

In Stage I of the three-stage sampling approach, soil and sediment samples analyzed on-site will be limited to EPA analytical level I and II analyses. Additional details are given in the Section 5 of the RI Work Plan and in Section 3.2 of the FSP (Volume 1 of the SAP). A subset of the soil and sediment samples will be analyzed on-site, and surface water and groundwater samples collected during Stage I will be sent off-site for analysis at EPA levels III and IV. This action is possible because of the relatively rapid breakdown of CWA in water and because a subset of the soil and sediment samples to be collected are expected to be free from CWA. The DQOs for Stage I sampling activities are summarized in Tables 4.1 and 4.2. The DQOs for Stage II sampling activities are summarized in Table 4.3. Stage III DQOs will be developed as necessary to ensure that sufficient data exist to support the remedial alternative selection as part of the FS.

The following elements are addressed in DQO summary tables and are discussed in more detail throughout the FSP and this QAPjP:

- The *objectives* of collecting data from the media associated with each field activity.
- The *data types* required to meet chemical and physical objectives. Included is the estimated number of data or samples that will be collected to meet the objective.
- A description of the *sampling method* to be used for each type of data.

TABLE 4.1 DQOs for Stage I Surveys and Hydrogeological Investigations at J-Field

DQO	Topographic Surveying and Grid System Establishment	Geophysical Survey	Soil-Gas Survey	Hydrogeologic Investigation
Objective	Global positioning system (GPS) or conventional survey methods will be used to map the topography of each AOC and to provide locational information for surveys, soil sampling, and monitoring well installation.	Electromagnetic, total-intensity magnetic, and ground-penetrating radar (GPR) electromagnetic survey measurements will be made to define boundaries of known burial trenches and to identify any yet-undiscovered trenches. Borehole electromagnetic induction (EMI) will be used to determine the vertical distribution of conductive zones. Surface electromagnetic (EM) surveys will be conducted to obtain information on contaminant plume movement. Seismic reflection, seismic refraction, and electrical resistivity data will be used to characterize the leaky confining unit. Other remote sensing data, such as thermal imagery, near-red, and infrared, will be used to evaluate the J-Field site for any other potential contamination source areas.	Active and passive soil-gas sampling will be conducted at the TBP, WPP, the Prototype Building, and RCP areas to define the extent of contamination and to identify new areas with elevated levels of soil-gas contaminants. These results will also be used to establish surface soil sampling locations.	ANL will install one new monitoring well at J-Field, downgradient of the TBP. This well will be constructed with a well screen located just above the surface of the confining layer. Groundwater samples will be collected and analyzed to determine the nature and extent of contamination in the surficial aquifer. Water levels will be measured periodically to construct a groundwater elevation map.

TABLE 4.1 (Cont.)

DQO	Topographic Surveying and Grid System Establishment	Geophysical Survey	Soil-Gas Survey	Hydrogeologic Investigation
Chemical Data	None.	None.	A minimum of 165 active and/or passive soil-gas samples will be collected. Analytes will include chlorinated solvents (12DCE, 112TCE, TRCLE, TCLEE, and TCLEA); phthalates; benzene; and toluene, ethylbenzene, and xylenes (TEX).	Groundwater samples will be analyzed for TCL (VOCs, semivolatile compounds, PCBs, and pesticides), CWA degradation products, TAL (metals, dissolved metals, and cyanide), explosives, TOX, TOC, phosphate, sulfate, and radioactivity.
Physical Data	Details to be determined.	Geophysical surveys will be interpreted by the J-Field team and will be used to guide the placement of soil gas monitoring points, surface and sub-surface soil sample locations, and/or monitoring wells.	None.	Temperature, pH, specific conductance, redox, dissolved oxygen, and turbidity will be measured before, during, and after presample purging. Other general parameters will be analyzed as stipulated in Section 3.4.3 of the FSP. Water-level measurements will be taken within a 24-hour period from all wells, accurate to 0.01 ft.
Sampling Method	None.	***Chemical Data:*** No chemical data will be collected. ***Physical Data:*** Biased, non-intrusive.	***Chemical Data:*** Biased, intrusive. ***Physical Data:*** No physical data will be collected.	***Chemical Data:*** Biased, intrusive, environmental, discrete. ***Physical Data:*** Biased, intrusive, environmental, discrete.
Data Use	Site characterization.	Site characterization.	Site characterization.	Site characterization, risk assessment.

TABLE 4.1 (Cont.)

DQO	Topographic Surveying and Grid System Establishment	Geophysical Survey	Soil-Gas Survey	Hydrogeologic Investigation
Analytical Data Level	All data will be equivalent to EPA level I.	All data will be equivalent to EPA level I.	All data will be equivalent to EPA level II.	AEC- and CLP-certified analyses will be equivalent to EPA levels III and IV. All other data (measurements of temperature, pH, conductivity, and water levels) will be equivalent to EPA level I.
Analytical Method — AEC, CLP,[a] and ASTM[b]	No chemical or physical analytical data are required for surveying and grid establishment.	No chemical or physical analytical data are required for geophysical surveys.	Soil-gas samples will be analyzed by noncertified methods.	***Chemical Testing:*** see Table 2.1. ***Physical Testing:*** TBD.[c]
Typical Detection Limit	None.	None.	Typically 1 ppm.	Varies depending upon environmental media and matrix interference effects.
Quality Control Samples	None.	None.	Duplicates, others to be determined.	Trip blanks, rinse blanks, duplicates, filter blanks, ambient blanks, method blanks, internal laboratory standards.
Background Samples	None.	None.	None.	None.

[a] CLP = Contract Laboratory Program.

[b] ASTM = American Society for Testing and Materials.

[c] TBD = to be determined.

TABLE 4.2 DQOs for Stage I Soil, Surface Water, and Sediment Sampling and for Tidal Monitoring

DQO	Subsurface Soil Sampling	Surface Water/Sediment Sampling	Surface Soil Sampling	Tidal Monitoring
Objective	Subsurface soil samples will be collected and analyzed to determine the nature and extent of contamination.	Surface water samples will be collected from marshes near the AOCs. In addition, tidal areas offshore of the Gunpowder Neck will be sampled. The results of the walkover inspection and surveys at each AOC will be used to locate any intermittent surface water features indicating that surface water drainage is a possible contaminant migration pathway.	Surface soil samples will be collected and field X-ray fluorescence (XRF) metal scans performed at the AOCs and PAOCs to locate areas of contamination. UXO/CWA screening will be required.	A tidal gauge equipped with a digital recorder will be installed in the Chesapeake Bay, about 200-300 yd east of Robins Point, to help determine the impact of tidal fluctuations on the aquifers and to determine groundwater flow velocity.
Chemical Data	Subsurface soil samples will be analyzed on-site for CWA and UXO (screen) and the Stage I analytical suite.	Surface water samples will be analyzed for the CLPAS.[a] Sediment samples will be analyzed on-site for CWA and UXO (screen) and the Stage I analytical suite.	Surface soil samples will be analyzed on-site for CWA (screen) and the Stage I analytical suite minus the MICKIT analyses. Gross beta and gamma spectrometry measurements will be made on some samples.	None.
Physical Data	Map coordinates for all sampling locations will be generated. General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.	Map coordinates for all sampling locations will be generated. General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.	Map coordinates for all sampling locations will be generated. Visual characterization of soils. General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.	The tidal gauge station will continuously record water levels in the Chesapeake Bay with a digital recorder for approximately 3 years.

TABLE 4.2 (Cont.)

DQO	Subsurface Soil Sampling	Surface Water/Sediment Sampling	Surface Soil Sampling	Tidal Monitoring
Sampling Method	***Chemical Data:*** Biased, intrusive, environmental, discrete. ***Physical Data:*** No physical data will be collected.	***Chemical Data:*** Biased, intrusive, environmental, discrete. ***Physical Data:*** No physical data will be collected.	***Chemical Data:*** Biased, intrusive, environmental, discrete. ***Physical Data:*** No physical data will be collected.	***Chemical Data:*** No chemical data will be collected. ***Physical Data:*** Biased, intrusive, discrete.
Data Use	Site characterization, risk assessment.	Site characterization, risk assessment.	Site characterization, risk assessment.	Site characterization.
Analytical Data Level	On-site analyses of the Stage I analytical suite equivalent to EPA levels I and II. AEC and CLP-certified analyses will be equivalent to EPA level IV for subsurface soil.	AEC- and CLP-certified analyses will be equivalent to EPA level IV for surface water and sediment. For sediment, on-site analyses equivalent to EPA levels I and II.	On-site analyses of the Stage I analytical suite equivalent to EPA levels I and II. AEC and CLP-certified analyses will be equivalent to EPA level IV for surface soil. Visual inspection will be equivalent to EPA level I.	All data will be equivalent to EPA level I.
Analytical Method — AEC, CLP, and ASTM	***Chemical Testing:*** see Table 2.3.	***Chemical Testing:*** see Tables 2.4 and 2.5.	***Chemical Testing:*** see Table 2.2.	No chemical or physical analytical data are required for tidal gauge measurements.
Typical Detection Limit	TBD[b]	TBD	TBD	None.

TABLE 4.2 (Cont.)

DQO	Subsurface Soil Sampling	Surface Water/Sediment Sampling	Surface Soil Sampling	Tidal Monitoring
Quality Control Samples	Trip blanks, rinse blanks, duplicates, filter blanks, ambient blanks, method blanks, internal laboratory standards.	Trip blanks, rinse blanks, duplicates, ambient samples, method blanks, internal laboratory standards.	Rinse blanks, duplicates, method blanks, ambient samples, internal laboratory standards.	None.
Background Samples	APG is developing a sitewide background study.	APG is developing a sitewide background study.	APG is developing a sitewide background study.	None.

[a] CLPAS = Contract Laboratory Program Analytical Suite.

[b] TBD = to be determined.

TABLE 4.3 DQOs for Stage II Soil, Subsurface Soil, Sediment, and Monitoring Well Sampling

DQO	Surface Soil Sampling	Subsurface Soil Sampling	Sediment Sampling	Hydrogeologic Investigation
Objective	Surface soil samples will be collected at the AOCs to define the extent of contamination. UXO/CWA screening will be required.	Subsurface soil samples will be collected and analyzed to determine the nature and extent of contamination. UXO/CWA screening will be required.	Sediment samples will be collected from marshes near the AOCs. In addition, sediment borings will be drilled near the TBP. The results of the walkover inspection and surveys at each AOC will be used to locate any intermittent surface water features indicating that surface water drainage is a possible contaminant migration pathway. CWA and UXO screening will be required on sediment samples.	A subset of the monitoring wells will be sampled to further site characterization efforts. If needed, additional monitoring wells will be installed and sampled upon approval of well site and construction specification by EPA and MDE.[a]
Chemical Data	Subsurface soil samples will be analyzed on-site for CWA (screen) and the CLPAS. Gross beta and gamma spectrometry measurements will be made on some samples.	Subsurface soil sampling sites will be analyzed on-site for CWA and UXO (screen) and the CLPAS.	Sediment samples will be analyzed on-site for CWA and UXO (screen) and the CLPAS.	Groundwater samples will be analyzed for TCL (VOCs, semivolatile compounds, PCBs, and pesticides), CWA degradation products, TAL (metals, dissolved metals, and cyanide), explosives, TOX, TOC, phosphate, sulfate, and radioactivity.

TABLE 4.3 (Cont.)

DQO	Surface Soil Sampling	Subsurface Soil Sampling	Sediment Sampling	Hydrogeologic Investigation
Physical Data	Map coordinates for all sampling locations will be generated.	Map coordinates for all sampling locations will be generated.	Map coordinates for all sampling locations will be generated.	Temperature, pH, specific conductance, redox, dissolved oxygen, and turbidity will be measured before, during, and after presample purging. Other general parameters will be analyzed as stipulated in Section 3.4.3 of the FSP. Map coordinates for any additional wells will be generated.
	Visual characterization of soils.	General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.	General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.	
	General parameters will be analyzed as stipulated in Section 3.2.2 of the FSP.			Water-level measurements will be taken before the sampling event from all wells, accurate to 0.01 ft.
Sampling Method	***Chemical Data:*** Biased, intrusive, environmental, discrete.	***Chemical Data:*** Biased, intrusive, environmental, discrete.	***Chemical Data:*** Biased, intrusive, environmental, discrete.	***Chemical Data:*** Biased, intrusive, environmental, discrete.
	Physical Data: No physical data will be collected.	***Physical Data:*** No physical data will be collected.	***Physical Data:*** No physical data will be collected.	***Physical Data:*** Biased, intrusive, environmental, discrete.
Data Use	Site characterization, risk assessment.	Site characterization, risk assessment.	Site characterization, risk assessment.	Site characterization, risk assessment.

TABLE 4.3 (Cont.)

DQO	Surface Soil Sampling	Subsurface Soil Sampling	Sediment Sampling	Hydrogeologic Investigation
Analytical Data Level	AEC- and CLP-certified analyses will be equivalent to EPA level IV for surface soil sampling. Visual inspection will be equivalent to EPA level I.	AEC- and CLP-certified analyses will be equivalent to EPA level IV for subsurface soil sampling. Visual inspection will be equivalent to EPA level I.	AEC- and CLP-certified analyses will be equivalent to EPA level IV for sediment.	AEC- and CLP-certified analyses will be equivalent to EPA levels III and IV. All other data (measurements of temperature, pH, conductivity, and water levels) will be equivalent to EPA level I.
Analytical Method — AEC, CLP, and ASTM	***Chemical Testing:*** see Table 2.2. ***Physical Testing:*** TBD[b]	***Chemical Testing:*** see Table 2.3. ***Physical Testing:*** TBD	***Chemical Testing:*** see Tables 2.4 and 2.5. ***Physical Testing:*** TBD	***Chemical Testing:*** see Table 2.1. ***Physical Testing:*** TBD
Typical Detection Limit	TBD	TBD	TBD	TBD
Quality Control Samples	Rinse blanks, duplicates, method blanks, ambient samples, internal laboratory standards.	Trip blanks, rinse blanks, duplicates, filter blanks, ambient blanks, method blanks, internal laboratory standards.	Trip blanks, rinse blanks, duplicates, ambient samples, method blanks, internal laboratory standards.	Rinse blanks, duplicates, method blanks, ambient samples, internal laboratory standards.
Background Samples	APG is developing a sitewide background study.	APG is developing a sitewide background study.	APG is developing a sitewide background study.	APG is developing a sitewide background study.

[a] MDE = Maryland Department of the Environment.

[b] TBD = to be determined.

- The *uses* for which data are being collected (described in terms of general-purpose categories, e.g., site characterization and risk assessment).

- The identification of an appropriate *EPA analytical level* for the analysis (or measurement) being performed.

- The *analytical method* (USATHAMA, CLP, or American Society for Testing and Materials [ASTM]) that will be used to analyze samples.

- The *typical detection limit* requirements for the chosen analytical methods. Detection limits will always be lower than the levels of concern (LOCs), where available. Tables 4.4 and 4.5 list LOCs for groundwater and surface water. The LOCs chosen for the sampling event were calculated for the protection of human health and aquatic life on the basis of the EPA Water Quality Criteria (EPA 1988a). LOCs are not available for soil, sediment, or biological samples.

- The *types of quality control samples* that will be collected in association with each sampling media. Quality control procedures for field sampling are discussed in Sections 5 and 10 of this QAPjP, and analytical methods are discussed in Section 8.

- The *background locations* and number of background samples to be collected. These items are currently deferred to the APG background study to be conducted by the AEC.

4.2 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The following subsections establish QA objectives for accuracy, precision, representativeness, completeness, and comparability that apply to both on-site and off-site field and laboratory analyses. Objectives for these parameters are established to ensure that the field and laboratory data collected during the RI meet the requirements specified in the DQOs.

4.2.1 Accuracy

Accuracy is the degree of agreement of a measurement (or an average of measurements of the same characteristic), X, with an accepted reference or true value, T. Accuracy is usually expressed as the difference between two values, X – T, the difference as a percentage of the reference or true value, 100(X – T)/T, or sometimes, as a ratio, X/T. Accuracy is a measure of bias in a measurement system (EPA 1980a).

Sampling accuracy will be quantitatively assessed through the evaluation of trip blank, rinse blank, field blank, and filter blank data. This information indicates whether

TABLE 4.4 Groundwater and Surface Water Analyte Levels of Concern (LOCs) for Human Health[a]

Analyte	Concentration (µg/L)			
	Water Ingestion		Fish Ingestion	
	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]
TCL[c] Volatile Organic Compounds				
Benzene	–[d]	0.67	–	40
Bromodichloromethane	–	–	15.7	–
Bromomethane	–	0.19[e]	–	15.7[e]
Carbon tetrachloride	–	0.42	–	6.94
Chlorobenzene	488	–	–	–
Chloroform	–	0.19	–	15.7
Chloromethane	–	0.19[e]	–	15.7[e]
Dibromochloromethane	–	0.19[e]	–	15.7[e]
1,2-Dichloroethane	–	–	–	243
1,1-Dichloroethylene	–	0.033	–	1.85
trans-1,2-Dichloroethylene	–	–	–	–
1,2-Dichloropropane	–	–	–	–
cis-1,3-Dichloropropene	87	–	14,000	–
trans-1,3-Dichloropropene	87	–	14,000	–
Ethylbenzene	2,400	–	3,280	–
Tetrachloroethylene	–	0.88	–	8.85
Toluene	15,000	–	424,000	–
1,1,1-Trichloroethane	19,000	–	1.03×10^6	–
1,1,2-Trichloroethane	–	0.60	–	41.8
Trichloroethylene	–	2.8	–	80.7
TCL[c] Semivolatile Organic Compounds				
Acenaphthene	20	–	–	–
Benzylbutylphthalate	–	–	–	–
4-Bromophenyl phenyl ether	–	–	–	–
bis(2-Chloroethoxy)methane	–	0.19[e]	–	15.7[e]
bis(2-Chloroethyl)ether	–	–	–	1.36
bis(2-Chloroisopropyl)ether	–	–	–	–
4-Chloro-3-methylphenol	–	–	–	–
2-Chloronaphthalene	–	–	–	–
2-Chlorophenol	–	–	–	–
4-Chlorophenyl phenyl ether	–	–	–	–
1,2-Dichlorobenzene	470	–	2,600	–
1,3-Dichlorobenzene	470	–	2,600	–
1,4-Dichlorobenzene	470	–	2,600	–
2,4-Dichlorophenol	3,090	–	3,090	–
Diethylphthalate	–	–	1.8×10^6	–

TABLE 4.4 (Cont.)

Analyte	Concentration (µg/L)			
	Water Ingestion		Fish Ingestion	
	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]
TCL[c] Semivolatile Organic Compounds (Cont.)				
2,4-Dimethylphenol	–	–	–	–
Dimethylphthalate	350,000	–	2.9×10^6	–
Di-*n*-butylphthalate	44,000	–	150,000	–
Di-*n*-octylphthalate	–	–	–	–
2,4-Dinitrophenol	–	–	–	–
4,6-Dinitro-2-methylphenol	–	–	–	–
bis(2-Ethylhexyl)phthalate	21,000	–	50,000	–
Fluoranthene	188	–	54	–
Hexachlorobenzene	–	0.021	–	0.00074
Hexachlorobutadiene	–	0.45	–	50
Hexachlorocyclopentadiene	206	–	14,800	–
Hexachloroethane	–	–	–	8.74
Isophorone	5,200	–	520,000	–
Naphthalene	–	–	–	–
Nitrobenzene	19,800	–	–	–
2-Nitrophenol	–	–	–	–
4-Nitrophenol	–	–	–	–
N-Nitroso-diphenylamine	–	7.0	–	16.1
N-Nitroso-dipropylamine	–	–	–	–
Pentachlorophenol	1,010	–	–	–
Phenol	3,500	–	–	–
Toxaphene	–	0.026	–	0.00073
1,2,4-Trichlorobenzene	–	–	–	–
2,4,5-Trichlorophenol	–	–	–	–
Pesticides				
Aldrin	–	0.0012	–	0.00079
alpha-BHC[f]	–	0.013	–	0.031
beta-BHC	–	0.023	–	0.0547
delta-BHC		–	–	
gamma-BHC (Lindane)	–	0.017	–	0.0625
Chlordane	–	0.022	–	0.00048
4,4-DDE	–	–	–	–
4,4-DDT	–	0.0012[g]	–	0.000024[g]
Dieldrin	–	0.0011	–	0.000076
Endosulfan I	138[h]	–	159[h]	–
Endosulfan II	138[h]	–	159[h]	–
Endrin	1	–	–	–
Heptachlor	–	0.011	–	0.00029
Toxaphene	–	0.026	–	0.00073

TABLE 4.4 (Cont.)

Analyte	Concentration (µg/L)			
	Water Ingestion		Fish Ingestion	
	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]	Threshold Toxicity Protection[b]	10^{-6} Cancer Risk[b]
PCBs/Aroclors				
PCBs		0.013	–	0.000079
TAL[c] Inorganics				
Antimony (total)	146	–	45,000	–
Arsenic (total)	–	0.025	–	0.0175
Beryllium (total)	–	0.0039		0.117
Cadmium (total)	10	–	–	–
Chromium, trivalent	179,000	–	3.4×10^6	–
Chromium, hexavalent	50	–	–	–
Copper	1,000	–	–	–
Cyanide	200	–	–	–
Lead	50	–	–	–
Mercury	10	–	0.146	–
Nickel	15.4	–	100	–
Selenium	10	–	–	–
Silver	50	–	–	–
Thallium	17.8	–	48	–
Zinc	5,000	–	–	–
Explosives				
1,3-Dinitrotoluene				
2,4-Dinitrotoluene	–	0.11	–	9.1
2,6-Dinitrotoluene	–	–	–	–
Nitrobenzene	19,800	–	–	–

[a] Based on a table, EPA Water Quality Criteria for Protection of Human Health (August 1988), reported in EPA (1988a).

[b] Threshold toxicity is the concentration limit at which adverse effects are manifested in organisms as a result of exposure; 10^{-6} cancer risk is the concentration of which a lifetime exposure will lead to a 10^{-6} risk of cancer.

[c] TCL = Target Compound List; TAL = Target Analyte List.

[d] Data not available.

[e] Value is for nonspecific halomethanes.

[f] BHC = benzene hexachloride.

[g] Value is for nonspecific DDT.

[h] Value is for nonspecific endosulfan.

Source: EPA (1988a).

TABLE 4.5 Groundwater and Surface Water Analyte Levels of Concern (LOCs) for Aquatic Life[a,b]

	Concentration (µg/L)			
	Freshwater		Marine	
Analyte	Acute	Chronic	Acute	Chronic
TCL[c] Volatile Organic Compounds[d]				
Benzene	5,300	–[e]	5,100	700
Bromodichloromethane	11,000	–	12,000	6,400
Bromomethane	11,000	–	12,000	6,400
Carbon tetrachloride	35,200	–	50,000	–
Chlorobenzene	1,120	763	1,970	–
Chloroform	28,900	1,240	–	–
Chloromethane	11,000	–	12,000	–
Dibromochloromethane	11,000	–	12,000	–
1,2-Dichloroethane	118,000	20,000	113,000	–
1,1-Dichloroethylene	11,600	–	224,000	–
trans-1,2-Dichloroethylene	11,600	–	224,000	–
1,2-Dichloropropane	23,000	5,700	10,300	3,040
cis-1,3-Dichloropropene	6,060	244	790	–
trans-1,3-Dichloropropene	6,060	244	790	–
Ethylbenzene	32,000	–	430	–
Tetrachloroethylene	5,280	840	10,000	450
Toluene	17,500	–	6,300	5,000
1,1,1-Trichloroethane	18,000	–	31,200	–
1,1,2-Trichloroethane	18,000	9,400	–	–
Trichloroethylene	45,000	21,900	2,000	–
TCL[c] Semivolatile Organic Compounds[d]				
Acenaphthene	1,700	520	970	710
Benzylbutylphthalate	940	3	2,944	3.4
4-Bromophenyl phenyl ether	360	122	–	–
bis(2-Chloroethoxy)methane	11,000	–	12,000	6,400
bis(2-Chloroethyl)ether	360	122	–	–
bis(2-Chloroisopropyl)ether	360	122	–	–
4-Chloro-3-methylphenol	30	–	–	–
2-Chloronaphthalene	1,600	–	–	7.5
2-Chlorophenol	4,380	2,000	–	–
4-Chlorophenyl phenyl ether	360	122	–	–
1,2-Dichlorobenzene[b]	1,120	763	1,970	–
1,3-Dichlorobenzene[b]	1,120	763	1,970	–
1,4-Dichlorobenzene[b]	1,120	763	1,970	–
2,4-Dichlorophenol	2,020	365	–	–

TABLE 4.5 (Cont.)

Analyte	Concentration (µg/L)			
	Freshwater		Marine	
	Acute	Chronic	Acute	Chronic
TCL[c] Semivolatile Organic Compounds[d] (Cont.)				
Diethylphthalate	940	3	2,944	3.4
2,4-Dimethylphenol	2,120	–	–	–
Dimethylphthalate	940	3	2,944	3.4
Di-*n*-butylphthalate	940	3	2,944	3.4
Di-*n*-octylphthalate	940	3	2,944	3.4
2,4-Dinitrophenol	230	150	4,850	–
4,6-Dinitro-2-methylphenol	230	150	4,850	–
bis(2-Ethylhexyl)phthalate	940	3	2,944	3.4
Fluoranthene	3,980	–	40	16
Hexachlorobenzene	1,120	763	1,973	–
Hexachlorobutadiene	90	9.3	32	–
Hexachlorocyclopentadiene	7.0	5.2	7	–
Hexachloroethane	980	540	940	–
Isophorone	117,000	–	12,900	–
Naphthalene	2,300	620	2,350	–
Nitrobenzene	27,000	–	6,680	–
2-Nitrophenol	230	150	4,850	–
4-Nitrophenol	230	150	4,850	–
N-Nitroso-diphenylamine	5,850	–	3.3×10^6	–
N-Nitroso-dipropylamine	5,850	–	3.3×10^6	–
Pentachlorophenol	55	3.2	53	34
Phenol	10,200	2,560	800	–
Toxaphene	1.6	0.013	0.07	–
1,2,4-Trichlorobenzene	1,120	763	1,970	–
2,4,6-Trichlorophenol	500,000	970	–	–
Pesticides				
Aldrin	3.0	–	1.3	–
alpha-BHC[f]	100[d]	–	0.34[d]	–
beta-BHC	100[d]	–	0.34[d]	–
delta-BHC	100[d]	–	0.34[d]	–
gamma-BHC (Lindane)	100[d]	–	0.34[d]	–
Chlordane	2.4	0.0043	0.09	0.004
4,4-DDE	1,050[d]	–	14[d]	–
4,4-DDT	1.1	0.001	0.13	0.001
Dieldrin	2.5	0.019	0.71	0.0019
Endosulfan I	0.22	0.056	0.034	0.0087
Endosulfan II	0.22	0.056	0.034	0.0087
Endrin	0.18	0.0023	0.037	0.0023
Heptachlor	0.52	0.003	0.053	0.0036
Toxaphene	1.6	0.013	0.07	–

TABLE 4.5 (Cont.)

Analyte	Concentration (µg/L)			
	Freshwater		Marine	
	Acute	Chronic	Acute	Chronic
PCBs/Aroclors				
PCBs	2.0	0.014	10	0.03
TAL[c] Inorganics				
Antimony	9,000[d]	1,600[d]	–	–
Arsenic, trivalent	360	190	69	36
Arsenic, pentavalent	850[d]	48[d]	2,319[d]	13[d]
Beryllium	130[d]	5.3[d]	–	–
Cadmium	3.9[g]	1.1[g]	43	9.3
Chromium, trivalent	1,700[g]	–	10,300[d]	–
Chromium, hexavalent	16	11	1,100	50
Copper	18[g]	12[g]	2.9	2.9
Cyanide	22	5.2	1	1
Lead	82[g]	3.2[g]	140	5.6
Mercury	2.4	0.012	2.1	0.025
Nickel	1,400[g]	96[g]	140	7.1
Selenium	260	35	410	54
Silver	4.1[g]	0.12	2.3	–
Thallium	1,400[d]	40[d]	2,130[d]	–
Zinc	320[g]	47	170	58
Explosives[d]				
1,3-Dinitrotoluene	330	230	590	370
2,4-Dinitrotoluene	330	230	590	370
2,6-Dinitrotoluene	330	230	590	370
Nitrobenzene	27,000	–	6,680	–

[a] Based on a table, EPA Water Quality Criteria for Protection of Aquatic Life (September 1986), reported in EPA (1988a).

[b] For some compounds, values listed for nonspecific compounds were used. For example, for 1,2-, 1,3-, and 1,4-dichlorobenzene, the values for nonspecific dichlorobenzene were used.

[c] TCL = Target Compound List; TAL = Target Analyte List.

[d] Insufficient data to develop criteria; values presented are the lowest observed effective levels (LOELs).

[e] Data not available.

[f] BHC = benzene hexachloride.

[g] Where the criterion is hardness dependent, the value was calculated using a hardness of 100 mg/L.

Source: EPA (1988a).

contamination has been introduced during the sampling event or during sample transit. The rinse blank data will be collected at 10% of the total number of samples per media (where applicable) and will provide an assessment of decontamination efficiency and the potential for cross contamination to occur during the field investigation. One trip blank will be added to each cooler containing aqueous samples for analysis of volatile organic compounds (VOCs) and will itself be analyzed for VOCs. Trip blank data will provide an indication of whether VOCs have been introduced into the samples during shipment. Ambient field blanks will be collected at a frequency of 10% of the total number of samples to provide data on contamination entering from the ambient air. Filter blanks will be collected with the filters used for groundwater samples for dissolved or total metals analysis at a frequency of one per lot of filters.

Accuracy in the analytical laboratory will be assessed through the evaluation of the percent recoveries associated with reference samples (e.g., matrix spikes, surrogates, continuing calibration checks). Potential sample contamination contributed by the laboratory environment will be discerned through the evaluation of the laboratory method blanks. Method blanks will be processed at the beginning of each analytical run by the laboratory to determine whether internal laboratory sources of contamination could affect the integrity of the sample.

The criterion for the evaluation of blank contamination applies to any blank (field and laboratory) associated with the samples and states that no contamination should be in the blank. If contamination is detected in a blank sample, all data associated with the blank will be evaluated to determine if there is an inherent variability in the data for the lot or if the problem is an isolated occurrence not affecting all samples in the lot. In cases where more than one blank is associated with a given sample, qualification will be based on a comparison with the associated blank having the highest concentration of the contaminant.

Sample results greater than the certified reporting limits (CRLs) but less than five times the amount detected in any blank will be reported as undetected for inorganic constituents. The criterion for organic contamination will depend on whether the contamination is a common laboratory contaminant. For organic constituents, the sample result will be reported as undetected when the compound concentration is greater than the CRL but less than 10 times the amount in any blank for common laboratory contaminants (e.g., methylene chloride, acetone, toluene, 2-butanone [methyl ethyl ketone, or MEK], carbon disulfide, and common phthalate esters).

The accuracy of field measurements can be inferred from the calibration logs generated before and after measurements. The goal regarding level of accuracy for field measurements of this project is established at 80%. Calibrated readings will be compared with initial readings obtained the day after calibration. Readings that vary by more than 20% will be identified, and corrective action will be taken (discontinuing use of instrument and obtaining a replacement). The previous day's results will be qualified. Samples will be reanalyzed when results cannot be qualified or as otherwise deemed necessary. Accuracy of field measurements will be qualitatively controlled through the use of SOPs developed to

standardize the collection of measurements and samples. Consistent proper calibration of all equipment throughout the field exercises, as described in this QAPjP, will assist in the accuracy of measurements. Field documentation and QA audits will be used to establish that protocols for sampling and measurement follow appropriate SOPs.

4.2.2 Precision

Precision measures the reproducibility of measurements under a given set of circumstances. Specifically, it is a quantitative measure of the variability of a group of measurements compared with their average value. Precision is usually stated in terms of standard deviation, but other estimates, such as relative percent difference (relative standard deviation), range (maximum value minus minimum value), or relative range, are also used. The overall precision of measurement data is a mixture of sampling and analytical factors. Analytical precision is easier to control and quantify than sampling precision, mainly because a laboratory is a controlled, and therefore a measurable, environment. Sampling precision is unique to each site, making it harder to control and quantify. Precision will be evaluated by calculating the relative percent difference (RPD). The smaller the RPD, the more precise the measurements:

$$RPD = \frac{XA - XB}{XM} \times 100 \quad , \tag{4.1}$$

where

XA and XB are duplicate analyses, and

XM is the mean value of duplicate analyses XA and XB.

Sampling precision will be assessed by evaluating duplicate samples collected at a frequency of 1 duplicate sample for every 10 samples collected for each environmental medium. A duplicate sample is defined as a sample that is collected from the same source and under identical conditions as the original sample. The duplicate is used as a check for field and laboratory procedures. The RPD will be assessed for detected analytes in all duplicate samples. A level of 75-80% precision (i.e., RPD $\leq$25%) will be the goal for the J-Field RI/FS; those analytes with an RPD greater than 25% will be identified.

Analytical precision is determined by replicate analyses performed on each sample matrix. For organic parameters, a matrix spike and matrix spike duplicate are analyzed. For inorganic parameters, sample duplicates are analyzed. The RPD will be calculated for each analytical parameter. A level of 75% precision (i.e., RPD $\leq$25%) is expected for analyses for organic parameters. A level of 80% precision (i.e., RPD $\leq$20%) for water duplicates will be adopted as the criterion for inorganic duplicates. If these criteria are not met, the sampling techniques, sample media, and analytical procedures will be carefully examined as described above to identify the cause of the high RPD and to evaluate the usefulness of the

data. In addition, control charts will be used to monitor variations in the precision of analytical methods and to provide inferences on method performance.

4.2.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are properly selected and that a sufficient number of samples are collected.

Representativeness is addressed in the FSP (Volume 1 of the SAP) by describing the types of samples to be collected, the sample locations, and the rationale used to determine sampling locations. Field handling protocols (e.g., storage, handling in the field, and shipping) have also been designed to protect the representativeness of the collected samples. Proper field documentation and QA audits will be used to establish that protocols have been followed and that sample identification and integrity have been maintained. Field sampling, handling, and documentation protocols are detailed in the SOPs developed by the COE (1993) and ANL. (See Appendix A of this document.)

4.2.4 Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid. The overall completeness goal for all data collected during the RI work at J-Field is 80-100% to ensure that a sufficient number of valid data are generated. Missing or invalid data may reduce the precision of estimates or introduce bias, thus lowering the confidence level of the conclusions. Since completeness is expressed as a percentage, it does not take into account the completeness of such factors as critical sample locations or critical analytical parameters.

Sampling completeness will be assessed by evaluation of the total number of samples proposed in the FSP versus the actual number of samples collected and analyzed. The importance of any lost or suspect data will be evaluated in terms of the sample location, analytical parameter, nature of the problem, decision to be made, and the consequence of an erroneous decision. Critical locations or parameters for which data are determined to be inadequate may be resampled.

4.2.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. For this project, comparability will be achieved through the use of standard techniques to collect and analyze representative

samples and through the use of appropriate units to report analytical results. Standard field sampling and measurement methods are detailed in the SOPs (COE 1993 and Appendix A). Consistent, proper calibration of all equipment throughout the field exercises, as described in this QAPjP, will also assist in achieving the desired comparability of measurements. Field documentation and QA audits will be used to establish that protocols for sampling and measurement follow appropriate SOPs. Comparability of data depends on the accuracy and precision of the analytical analysis and proper qualification of data.

Results of field audits will be used to infer accuracy, precision, completeness, and representativeness associated with sampling activities. The field audit will be conducted early in the project to assist in the identification of potential out-of-control situations. The areas to be observed during the audit include (1) sample documentation and management, (2) field measurements and calibration, and (3) sampling protocols. Section 11 of this QAPjP discusses audits in more detail.

5 SAMPLING

The sampling strategy will be designed to provide samples that are as representative as practicable of the matrix or medium being investigated. Extreme care will be taken to avoid contamination of samples during collection activities. Procedures described in this section are designed to eliminate external contamination through the use of good sampling techniques. The SOPs for sampling activities are described by the COE (1993) and in Appendix A of this QAPjP.

5.1 GENERAL CONSIDERATIONS

5.1.1 Sample Labeling

At the time of sampling, each aqueous and solid sample will be assigned a unique sequential number that will be permanently affixed to the sample container. The sample label (Figure 5.1) will include the following information:

- Project name;
- Project number;
- Location (e.g., well number or surface water sampling number and other pertinent information about where the sample was taken);
- Date and time of sample collection;
- Analyses to be performed (include method name and number);
- Preservatives (for water samples);
- Sample number; and
- Number of containers for the sample.

INST:_____ SAMPLE:_____

ANALYTES: METALS VOC EXPLOSIVES
ORGANICS OTHER_____

FILTERED: [NO] [YES]

PRESERV.: NONE HNO_3 OTHER_____
DATE:__/__/__ SAMPLER:_____

FIGURE 5.1 Sample Bottle Label

All entries will be made with indelible ink. Labels will be covered with polyethylene tape to prevent the loss of the label during shipment. Procedures for sample labeling are discussed in SOP-001 (Sample Labels) (COE 1993).

All gaseous samples will also have a unique identification number. Sample numbers will be documented in the instrument logbook, the field logbook, and the instrument computer. In addition, the mass spectrometer control program for analysis of soil-gas samples will warn the operator if duplicate sample numbers are entered into the system.

5.1.2 Sample Containers

Before use, all sample containers will be cleaned in accordance with EPA and USATHAMA protocols. Certified precleaned sample containers may be obtained from the prime laboratory for groundwater and surface water samples. The sample containers to be used for aqueous and solid samples are described in Tables 5.1 and 5.2, respectively. All container requirements follow USATHAMA (1990) and CLP (EPA 1984c) specifications.

TABLE 5.1 Bottle Requirements, Preservation, and Holding Times for Aqueous Samples

Analyte	Bottle Requirements — Type[a] and Volume	Required Headspace	Preservative	Holding Time
VOCs	Type B (80 mL)	0%	Cool to 4°C	7 days
Semivolatile organic compounds	Type A, K, or H (2 L)	10%	Cool to 4°C	7 days to extraction (40 days after extraction)
Pesticides/PCBs	Type A, K, or H (2 L)	10%	Cool to 4°C	7 days to extraction (40 days after extraction)
Total metals	Type C, H, or L (1 L)	10%	HNO_3 to pH <2, cool to 4°C	6 months, except mercury (28 days)
Dissolved metals	Type C or L (1 L)	10%	HNO_3 to pH <2, cool to 4°C	6 months, except mercury (28 days)
Cyanide	Type C or L (1 L)	10%	NaOH to pH >12, cool to 4°C	14 days
CWA degradation products	One 1-L amber glass	10%	Cool to 4°C	None
Explosives	Two 1-L amber glass	10%	Cool to 4°C	7 days to extraction (40 days after extraction)
TOX	Type B (80 mL)	0%	H_2SO_4 to pH <2, cool to 4°C	7 days

TABLE 5.1 (Cont.)

Analyte	Bottle Requirements — Type[a] and Volume	Required Headspace	Preservative	Holding Time
TOC	250-mL plastic or glass	10%	HCl or H_2SO_4 to pH <2, cool to 4°C	28 days
Conductivity	250-mL amber glass	10%	Cool to 4°C	28 days
Major cations and anions	Type C (one 1-L), Type L (two 500-mL)		NA[b]	6 months
Radioactivity	Two 1-L polyethylene	10%	HCl or HNO_3 to pH <2, cool to 4°C	6 months

[a] Description of bottle types:

Type A Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm Teflon liner.

Type B Container: 40-mL glass vial, 24-mm neck finish.
Closure: White polypropylene or black phenolic, open-top, screw-cap, 15-cm opening, 24-400 size.
Septum: 24-mm disc of 0.005-in. Teflon bonded to 0.120-in. silicon for total thickness of 0.125 in.

Type C Container: 1-L high density polyethylene, cylinder-round bottle, 28-mm neck finish.
Closure: White polyethylene cap, white-ribbed, 28-410 size; F217 polyethylene liner.

Type H Container: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm Teflon liner.

Type K Container: 4-L amber glass, ring handle bottle/jug, 38-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm Teflon liner.

Type L Container: 500-mL high-density polyethylene, cylinder-round bottle, 28-mm neck finish.
Closure: White polypropylene cap, white-ribbed, 28-410 size; F217 polyethylene liner.

[b] NA = not applicable.

Sources: EPA (1987b); USATHAMA (1990).

TABLE 5.2 Bottle Requirements, Preservation, and Holding Times for Solid Samples

Analyte	Bottle Requirements — Type[a] and Volume	Preservative	Holding Time
CWAs	One 1-L amber glass	Cool to 4°C	None
TPHS	250-mL amber glass	Cool to 4°C	7 days to extraction (40 days after extraction)
Total PAHs	250-mL amber glass	Cool to 4°C	7 days to extraction (40 days after extraction)
Total metals	Type F or G (3 oz)	Cool to 4°C	6 months, except mercury (28 days)
Explosives	Two 1-L amber glass	Cool to 4°C	7 days to extraction (40 days after extraction)
PCBs	250-mL amber glass	Cool to 4°C	7 days to extraction (40 days after extraction)
Toxicity (MicroTox)	Glass[b]	NA[c]	NA
Soil microbes (MICKIT)	Glass[b]	NA	NA
Radioactivity	250-mL polyethylene or glass	Cool to 4°C	6 months

[a] Description of bottle types:

Type F: Container: 8-oz short, widemouthed, straight-sided, flint glass jar, 70-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm Teflon liner.

Type G: Container: 4-oz tall, widemouthed, straight-sided, flint glass jar, 48-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm Teflon liner.

[b] Containers to be supplied by the manufacturer.

[c] NA = not applicable.

Sources: EPA (1987b); USATHAMA (1990).

5.1.3 Sample Preservation

All samples will be transported to the laboratory in temperature-controlled coolers. Blue ice or wet ice will be used to maintain the internal cooler temperatures required for preservation of groundwater, surface water, soil, and sediment samples. Chemical preservatives will be required for selected samples in order to retard the degradation of constituents during transit and storage before laboratory analysis. These preservatives will be added to appropriate samples at the time of collection. The preservation requirements are listed in Tables 5.1 (aqueous) and 5.2 (solid). Step-by-step procedures for sample preservation are as follows:

1. Preservatives will be added to samples either with a pipette or directly to the sample if vials of preservatives are used.

2. The sample bottle will be capped and the bottle will be gently agitated (except for VOC samples) to homogenize the preservative throughout the sample.

3. The sample bottle cap will be reopened, a small amount of the sample will be transferred to a beaker, and the bottle will be closed.

4. Either pH paper or an electronic pH meter will be used to determine the pH of the sample. To avoid contamination of the sample and the equipment, the pH paper or meter will never be put directly into the sample bottle.

5. If the proper pH has been reached, the sample bottle will be left closed. If the proper pH has not been reached, the sample bottle will be reopened, more preservative added, the bottle shaken, and the pH test repeated. This process will continue until the proper pH has been reached.

6. Once the volume of acid or base to be added to a sample to achieve the proper pH has been established, the same volume will be used for all samples of equal type and volume.

7. For samples that require filtering, preservatives will always be added to the filtrate after the filtering.

5.2 FIELD SAMPLING PROCEDURES

5.2.1 Environmental Media Samples

Procedures for collection of samples of environmental media are described in the SOPs in COE (1993) and Appendix A of this QAPjP. The collection of samples will follow the

protocols outlined in the FSP (Volume 1 of the SAP) and in the following SOPs: SOP-005 (Decontamination), SOP-007 (Surface Water Sampling Procedures), SOP-013 (Collection of Monitoring Well Samples), SOP-020 (Active Soil Gas Sampling), SOP-021 (Sediment Sampling), SOP-025 (Soil Sampling), SOP-027 (Passive Soil Gas Survey), and SOP-S009 (EMFLUX Passive Soil Gas Surveys).

Information on procedures and field equipment for sampling soil, sediment, and water is provided in Section 5 of the FSP and in specific SOPs identified in Table 5.3 and described in COE (1993) and Appendix A.

5.2.2 Biological Samples

The procedures for collecting biological samples at J-Field will follow the protocols described in two COE reports (COE 1991, 1992).

5.2.3 Quality Control Samples

Stage I of the RI field work to be conducted at J-Field will include the collection of several types of QC samples. These samples will include duplicates, rinse blanks (from equipment decontamination), ambient field blanks, filter blanks, and trip blanks. This section describes the method and frequency of collection of field QC blanks.

Duplicate samples will be taken from areas that are known or suspected to be contaminated and will consist of one sample per week or 10% (1 per 10 samples) of all field samples. Fractions for the same analytical parameters will always be collected consecutively.

Rinse blanks will be collected when the sampling equipment is decontaminated and reused in the field or when a sample collection vessel (bailer or beaker) will be used. These blanks will be collected at a frequency of 10% (1 per 10 samples). The equipment used in sampling will be rinsed with water used to prepare blanks (high-pressure liquid chromatography [HPLC] grade for organic compounds, deionized water for inorganic compounds), and the water running off the equipment during decontamination will be collected in sample containers.

Field blanks will also be collected at a frequency of 10% (1 per 10 samples). A cleaned sample container will be filled with blank water (as described above) in the field and chemically analyzed to determine if detectable concentrations of contaminants are entering the sample bottles from the ambient air.

Filter blanks will be collected at a frequency of one filter per lot for samples collected for metal analysis. Deionized water will be run through the filter with the same filtering apparatus used to filter groundwater samples. The water will be collected in the appropriate sample bottles following filtering.

TABLE 5.3 Sources for Detailed Information on Collection Procedures for Soil, Sediment, and Water

Media	FSP Section	SOPs[a]
Soil gas	3.3.2	SOP-020, 026, 027, S005
Surface soil	3.3.3	SOP-025
Subsurface soil	3.3.4	SOP-025
Sediment	3.3.5	SOP-021
Surface water	3.3.5	SOP-007
Offshore water	3.3.5	SOP-007
Marsh water	3.3.5	SOP-007
Groundwater	3.4.3	SOP-013

[a] SOP titles are listed in Table A.1. SOPs developed by the U.S. Army Corps of Engineers are described in COE (1993); those developed by ANL (designated with an "S" for supplemental) are presented in Appendix A.

A blank for active soil gas will be sampled by use of a vacuum box to pass laboratory air through a shield point enclosed in a mixture of sand and bentonite and polyethylene tubing. This blank will verify that there is no contamination contribution by the components creating the sampling point or by venting of any potential sources into the air. At the end of each eight-hour analytical sequence, a calibration check will be run with benzene vapor to verify instrument sensitivity.

Trip blanks will be provided and will consist of HPLC-grade water. One trip blank per shipment will accompany the aqueous samples for VOC analysis to the laboratory.

5.3 FIELD EQUIPMENT DECONTAMINATION

Procedures for setting up decontamination stations will be presented in the HASP. Procedures for equipment decontamination and for logging of decontamination activities are summarized in SOP-005 (Decontamination) (COE 1993). Information on equipment decontamination is also provided in Section 5.2 of the FSP.

6 SAMPLE CUSTODY

6.1 CHAIN OF CUSTODY

Throughout the J-Field RI process, the possession and holding of samples will be regulated and maintained through established COC procedures, including use of appropriate COC documentation and affixing of proper custody seals to each container. A person will be considered to have custody of a sample if that sample is (1) actually in the person's possession, (2) within sight after being in the person's possession, and/or (3) in the person's possession at one time but moved to a locked area to prevent tampering.

Chain of custody will begin when sample containers are prepared and a sample is collected. Sample containers, sampling equipment, coolers, chemical preservatives, and COC documentation will be supplied by the contract laboratory. Typical sample requirements, including containers, preservation, holding time, and sample volume requirements for aqueous and solid environmental media are detailed in SOPs presented in COE (1993) and Appendix A. An example COC form is provided in SOP-002 and in Figure 6.1. Before on-site screening for CWAs, custody of samples will be maintained by the sampling team under the jurisdiction and direction of APG. The samples will be stored in an APG-designated storage facility in an OSL only after receiving approval from the APG project manager.

6.2 TRANSPORT OF SAMPLES

The following information on the transport of samples applies to all samples collected for shipment to the off-site prime and referee laboratories. Therefore, these requirements are applicable to the groundwater and surface-water samples collected in RI Stage I and all samples collected in Stages II and III, as described in the FSP.

6.2.1 Packaging and Handling

When applicable, all samples and sample extracts will be packaged and transported in a manner that will protect the integrity of the sample and ensure against any detrimental effects from possible leakage. Packaging procedures will vary, depending on the suspected contaminant concentrations and the U.S. Department of Transportation (DOT) hazard class.

Custody seals will be placed on each shipping container, and clear plastic tape will cover the seals to ensure they are not accidentally broken during shipment. The containers will be packaged in the following manner:

- Pack sample container in a resealable plastic bag (Ziploc or equivalent),
- Place the resealable plastic bag in a large plastic (garbage) bag sealed with a twist tie,

CHAIN-OF-CUSTODY RECORD

PROJ. NO.	PROJECT NAME					No. of Containers							REMARKS
SAMPLER: (Signature)													
Sta. No.	Date	Time	Comp.	Grab	Station Location								

Relinquished by:(Signature)	Date/Time	Received by:(Signature)	Relinquished by:(Signature)	Date/Time	Received by:(Signature)
Relinquished by:(Signature)	Date/Time	Received by:(Signature)	Relinquished by:(Signature)	Date/Time	Received by:(Signature)
Relinquished by:(Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks:	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

FIGURE 6.1 Typical Chain-of-Custody Form

- Secure the package with reinforced strapping tape,
- Affix a custody seal, and
- Affix a packing list pouch with a COC record listing the contents of the package.

6.2.2 Suspect Radioactive Materials

6.2.2.1 Radiation Screening

Before being prepared for shipment, a sample will be screened by qualified field personnel to determine if it should be transported as a radioactive shipment and, if so, how it should be packaged and shipped to the laboratory. Field radiation screening will consist of the following steps:

- Soil will be surveyed in screening and homogenization pans for gamma radiation before the soil is placed in containers.
- Handheld monitoring instruments, such as a Geiger-Müller counter, will be used to survey each sample. These instruments will be calibrated before use in the field. The survey will be conducted by qualified and trained personnel.
- A contact gamma survey will be performed on the outside of the sample container. In the case of a nonuniform sample (e.g., a soil sample), readings will be taken on all sides of the container. A contact reading will also be taken on the bottom of the sample bottle.
- All results will be recorded in a radiation screening log book.

6.2.2.2 Packaging of Radioactive Samples

For shipping purposes, a sample is considered radioactive if it contains a specific activity greater than 2×10^3 pCi/g (solids). Radioactive samples will be shielded to protect the health and safety of the public and the workers who will handle the sample shipments. Samples will be packaged in steel-belted coolers and checked by personnel on-site to ensure that readings are less than 0.5 mrem/yr at contact.

6.2.3 Shipping and Delivery Procedures

Samples will be processed in accordance with all sampling, handling, packaging, and shipping methods established by the EPA (1984b), as well as those required under DOT hazard classification regulations.

6.3 FIELD CUSTODY

6.3.1 Responsibilities

ANL field and on-site laboratory personnel will be responsible for the custody of samples from the time they are collected until they are transferred to the sample shipper or the prime or referee laboratory. A minimum number of persons will handle the samples to reduce the number of transfers. Basic COC procedures for the project are described in Section 6.1.

6.3.2 Procedures

Each sample collected will be identified with a unique ANL sample number by a self-sticking, prenumbered label affixed onto the sample container. The sample number and all sampling information, including a list of preservatives that have been added, will be recorded in the field logbook. The sample will be placed on ice in a thermal chest. The chest will remain within sight of the sampler or will be locked in the field vehicle for temporary storage and transport to the sample staging area.

At the sample staging area, each sample container will be rinsed with organic-free water as necessary to remove any exterior debris. Pertinent information will be recorded on a COC form (Figure 6.1) to account for each sample. At least 5-10% blind replicate samples will be sent to the referee laboratory, along with blanks and other QA samples (see Section 10). To ensure that the laboratory does not know the identity of these QA samples, information on the COC records accompanying the samples will not describe the sample; however, all other information on the record will be included. The sample will be described only on the ANL copy of the COC form. The ANL copy of the record will become a permanent record in the project files.

The sampler will sign and record the date and time in the appropriate block of the COC form before relinquishing custody. The sample shipper will also sign and record the date and time in the appropriate block of the form to accept custody. This procedure will be followed each time samples are transferred.

The sample shipper will ensure that the samples are properly packaged for dispatch to the appropriate laboratory. The shipper will sign and record the date and time in the appropriate block of the COC record. The laboratory copies of the COC records will be placed in plastic bags and taped to the inside cover of the appropriate shipping container. Samples will be shipped by available common carrier or hand carried. The sample shipping records will be retained as part of the permanent custody documentation.

6.4 STORAGE DURING CWA SCREENING

During CWA screening, samples will be stored in an APG-designated storage facility or, upon approval from the APG project manager, in the OSL.

6.5 LABORATORY CUSTODY

6.5.1 Laboratory Receipt

When the prime laboratory or the referee laboratory receives the samples, the laboratory sample coordinator will sign the COC forms and record the date and time. The coordinator will then complete all the appropriate laboratory tracking sheets and logs and report any problems to the ANL sample shipper. The laboratory coordinator will be responsible for the custody of samples after they are received at the laboratory and will be responsible for disposal of (1) wastes after sample analysis and (2) any leftover sample material.

6.5.2 Sample Holding Time

Verified time of sample receipt (VTSR) by the prime and/or referee laboratory will be used as the holding time reference day 0 for purposes of establishing holding times for analytical consideration (EPA 1991a). VTSR will be used for monitoring holding times for both the CLP and AEC methods. VTSR is the current holding time standard as defined in the current CLP statement of work (EPA 1991a); however, AEC methods define day 0 as the time of sample collection (USATHAMA 1990).

All parties must be cognizant that soil samples collected from below 6 in. of soil depth and all soil and sediment samples collected from pushout and burning pit areas of J-Field will be screened for CWAs prior to shipment and/or pickup by the contractor, in accordance with the requirements of APG. Occasionally, situations may necessitate delays in sample shipment or delivery from the date of collection at the site because of delays in release of samples from CWA screening. Under these special circumstances, holding times for CWA degradation products, as defined in the AEC methodologies, are likely to be lapsed before sample receipt by the contractor laboratory. Because such delays in sample shipment will be beyond the control of field sampling or contractor laboratory personnel, VTSR will be used to define sample analysis holding time.

6.5.3 Laboratory/ANL Coordination

The prime and referee laboratories will have established detailed SOPs for sample handling, storage, and disbursement for analysis. These procedures will include examples of the sample receiving and tracking information sheet, sample logging sheets, laboratory assignment sheet, and other such documentation. Results of analyses will be recorded on

appropriate forms, and the information will be given to the laboratory project manager. Unused portions of samples will be returned to ANL after the completion of the analysis or will be retained in the laboratory with the mutual understanding that waste disposal instructions are awaited from ANL.

6.6 WASTE DISPOSAL

6.6.1 Sample Wastes Generated during Laboratory Investigations

Unused portions of solid and liquid samples will be stored in approved storage areas after laboratory investigations. The contractor will be responsible for ensuring that the appropriate waste-acceptance criteria are fulfilled for proper disposal of on-site-generated waste, including both treated and unused portions of samples following laboratory analysis. ANL project personnel will coordinate with APG personnel and/or the APG waste-disposal contractor to ensure that the custody, storage, and disposal of wastes are properly conducted. Any contaminated waste, soil, or debris generated during on-site activities (including on-site laboratory operations) will be placed in appropriate containers and removed by the APG waste-disposal contractor.

6.6.2 Storage and Disposal of Drilling and Sampling Wastes Generated during Field Investigations

Solid wastes generated during field sampling activities (such as contaminated protective clothing) will be stored on-site in 55-gal drums. The wastes will be tested for residual CWAs and other contamination and routed for disposal during remediation activities. Soil and sediment samples that are not sent for laboratory analysis will be composited in a secure drum and sampled before disposal during remediation activities. Used sample collection bottles will be rinsed and disposed of along with contaminated clothing and other solid waste. Rinse water will be collected and disposed of as decontamination rinse water. Liquid wastes from well drilling, well purging, and equipment decontamination will be segregated and stored in tanks or 55-gal drums for testing and ultimate disposal. Decontamination wastewaters containing methanol rinse waste will be segregated from other rinse waters. The final disposal route for this waste may differ from other liquid wastes.

Solid wastes containing UXO or CWAs collected during site surveys will be handled and disposed of by authorized technicians according to APG regulations.

7 CALIBRATION PROCEDURES

This section summarizes the calibration procedures and frequency of calibration for principal instruments and instrument groups that will be used in RI activities at J-Field. The procedures given here are derived in part from ICF-Kaiser Engineers (1992a). Instruments other than those discussed in the following sections will be calibrated in accordance with the procedures given in the manufacturers' manuals or in accordance with EPA-approved Superfund field operations methods (EPA 1987b).

7.1 FIELD INSTRUMENTS

7.1.1 Photoionization Detectors and Organic Vapor Analyzers

Photoionization detectors (including HNu PI-101, HNu HW-101, and MICROTIP HL-200 models) and organic vapor analyzers (including the FOXBORO 128 model) will be calibrated upon arrival at the site and daily while in the field. Measurements of background VOCs will be documented and zeroed out, the calibration gas will be added, the reading will be recorded, and the instrument will be adjusted for proper calibration. The final reading will also be documented. Calibration protocols and measurements will be documented in a bound logbook that accompanies each instrument, as well as in the field logbook.

7.1.2 Conductivity and pH Meters

Conductivity and pH meters (including models HYDAC, ACCUMET [pH only], and STICKS) will be calibrated upon arrival at and departure from the site and daily while in the field. The meters will be calibrated more frequently if the temperature changes by 5°C or more. The procedure for daily calibration of pH and conductivity meters will include an initial measurement before calibration, a measurement after calibration, and a measurement at the end of the day. All measurements will be documented at the end of the field parameter form logbook or on separate calibration log forms.

7.2 CALIBRATION STANDARDS

Equipment will be calibrated with the appropriate standards specified below. The analytical accuracy of these standards is traceable to standard analytical reference materials (SARMs) from the National Institute of Standards and Technology (NIST). The standards are as follows:

- *Conductivity Solution:* 1,000 µmho/cm (±0.50%) at 25.00°C, 0.053% potassium chloride, 0.0002% iodine, water (CAS 7732-18-5).

- *pH Buffers:*
  - 4.00 ±0.01 at 25°C, color-coded red; potassium hydrogen phthalate (CAS 877-24-7), formaldehyde (CAS 50-00-0), water (CAS 7732-18-5).
  - 7.00 ±0.01 at 25°C, color-coded yellow; sodium phosphate, dibasic (CAS 7558-79-4), potassium phosphate, monobasic (CAS 7778-77-0), water (CAS 7732-18-5).
  - 10.00 ±0.02 at 25°C, color-coded blue; potassium borate, tetra (CAS 1332-77-0), potassium carbonate (CAS 584-08-7), potassium hydroxide (CAS 1310-58-3), sodium (di)ethylenediamine tetraacetate (CAS 6381-92-6), water (CAS 7732-18-5).
- *Photoionization Detector Standards:* Isobutylene (I-C_4H_8) 100 ppm ±5%, balance: air.
- *Organic Vapor Analyzer Standards:* Methane (CH_4) 95 ppm ±5%, balance: air.

7.3 LABORATORY INSTRUMENTS

Initial and daily calibrations of laboratory instruments and of soil-gas equipment will be conducted as stipulated in the QA procedures described in the prime and referee laboratories' QA/QC manuals. At a minimum, before samples are analyzed, chemical calibration of each target analyte must be performed to ensure that analytical instrumentation is functioning within the established sensitivity range. Protocols defining the procedures and QC measurements for instrument calibration shall be established in accordance with criteria specified by AEC and CLP QA programs (USATHAMA 1990; EPA 1984c).

7.3.1 Initial Calibration of Laboratory Instrumentation

Initial calibrations for the methods to be used in this project are performed routinely by the laboratory. Additional initial calibrations are not required unless the instrument fails the daily calibration procedure.

7.3.2 Daily Calibration of Laboratory Instrumentation

Before an analysis is performed, each instrument will be calibrated to ensure that its response has not changed from the previous calibration. Analysis should be performed on the highest concentration standard. A response within two standard deviations of the mean response for the same concentration as determined from precertification, certification, and prior initial/daily calibrations will be deemed acceptable. Should the response fail that

criterion, the daily standard must be reanalyzed. Failure of this reanalysis will necessitate that the instrument undergo initial calibration as specified in the AEC and CLP QA programs (USATHAMA 1990; EPA 1984c).

7.3.3 Calibration of Soil-Gas Equipment

The active-soil-gas analysis instrument will be recalibrated every eight hours of operation, at a minimum. Before each analytical sequence, an instrument blank (carrier gas) will be analyzed to demonstrate that there is no contamination contribution from the analysis process.

The Curie-point pyrolizer mass spectrometer (Cp/MS) and the thermal desorption gas chromatography/mass spectrometry (GC/MS) used for passive soil-gas analyses will be calibrated to perfluorotributylamine to establish the correct mass assignment and mass resolution before each analytical sequence.

7.4 LABORATORY STANDARDS VALIDATION

All calibration solutions and standards used for this project will be prepared and maintained under the normal laboratory standards tracking system. This system ensures preparation, checking, documentation, storage, and disposal standards according to specified procedures and schedules appropriate for each analyte of interest.

7.5 CALIBRATION AND MAINTENANCE FREQUENCY SCHEDULE

Calibration and maintenance must be accomplished at the manufacturer's recommended frequency, unless prior experience dictates a more frequent schedule. Should a schedule not be provided by the manufacturer, the calibration group servicing the equipment must provide a written calibration and maintenance frequency. A list of critical spare parts for field equipment is provided in the equipment operations manual and/or the SOPs and equipment manual, which will be located on-site during all field activities.

7.5.1 Field Equipment Calibration, Maintenance Frequency, and Calibration Standards

For purposes of preventive maintenance, field equipment in storage for use in the J-Field RI sampling activities will be calibrated by the RI field team leader according to the following schedule:

- Photoionization Detectors (PIDs)

  - HNu PI-101: Every 30 days while in storage,
  - HNu HW-101: Every 30 days while in storage, and
  - MICROTIP HL-200: Every 30 days while in storage.

- Organic Vapor Analyzers (OVAs)

  - FOXBORO 128: Every 30 days while in storage.

Conductivity, temperature, and pH meters are calibrated only in the field. The particular standards to which the PIDs and OVAs are calibrated by the RI field team leader are as follows:

- PID Calibration Standards

  - Benzene (C_6H_6) 1010 ppm +/- 1%, balance: air,
  - Benzene (C_6H_6) 100 ppm +/- 1%, balance: air,
  - Benzene (C_6H_6) 10 ppm +/- 1%, balance: air, and
  - Isobutylene (I-C_4H_8) 100 ppm +/- 2%, balance: air.

- OVA Calibration Standards

  - Methane (CH_4) 5 ppm +/- 5%, balance: air,
  - Methane (CH_4) 95 ppm +/- 5%, balance: air, and
  - Methane (CH_4) 950 ppm +/- 2%, balance: air.

Analytical accuracy of all calibration gases is traceable to SARMs from the NIST. Field calibration of soil-gas equipment is discussed in Section 7.3.3.

7.5.2 Laboratory Calibration and Maintenance Frequency Schedule

A list of critical spare parts for laboratory equipment can be found in the contract laboratories' Quality Assurance Program Plans (QAPPs) and/or SOPs.

8 ANALYTICAL PROCEDURES

8.1 INTRODUCTION

This section discusses analytical procedures proposed for use in testing J-Field environmental media in the field and in the on-site and off-site analytical laboratories. When necessary and feasible, SOPs have been developed and are presented in Appendix A of the QAPjP and the *Work Plan for CERCLA Remedial Investigation / Feasibility Study* (COE 1993). However, in many cases, procedures from standard reference sources will be employed, including those developed by the EPA under the CLP (EPA 1979, 1980b, 1983, 1984c, 1985, 1986a,b, 1988b,c, 1989a, 1991a,b) and USATHAMA (1987, 1990). Such procedures and methods are identified in the text and tables, and complete reference citations are given in Section 17.

Commercial products and trade names are mentioned in this QAPjP when deemed necessary for illustrative purposes. However, such references made by the authors of this QAPjP, by ANL, or by any government agency are not intended to constitute preferential treatment, endorsement, or recommendation for use.

The technical applicability and the effectiveness with which data acquisition is accomplished within the scope of investigation and QA protocols were considered in instrument use and prescribed procedures. Table 8.1 contains a summary listing of analytes for on-site screening and off-site analysis of soil and sediment. In addition to the environmental solid media samples, including soil and sediment samples, the off-site testing and analysis will also consist of analysis of groundwater, surface water, surface soil, subsurface soils (soil borings), sediment, and deep sediment samples. Proposed organizations responsible for conducting on-site and off-site testing and analysis based on logistics of operation and APG requirements are summarized in Table 8.2.

8.2 ANALYTICAL PROCEDURES FOR ON-SITE ANALYSES

The RI team will be constrained by the fact that the possible presence of UXO, CWAs, and (in some cases) radioactive materials will prevent off-site shipment of soil and sediment samples except in accordance with rigorous and expensive escort and handling procedures. Additionally, constraints on the off-site shipment of soil and sediment samples potentially contaminated with CWAs dictate that on-site analyses be performed to the extent possible. As a result, a number of organic, inorganic, and radiological constituents potentially present in the environmental media being investigated will be analyzed on-site in the OSL during the RI. The OSL will provide a qualitative, as well as a semiquantitative, estimation of groups of analytes present, including their chemical category or chemical class.

TABLE 8.1 Summary Listing of Analytes for On-Site and Off-Site Analyses of Soil and Sediments

Parameters/Category	Analytical Activity: Stage I, On-Site Analysis	Analytical Activity: Stages II and III, Off-Site Analysis
Metals	x[a]	x
PCBs	x	x
Total PAHs	x	–[b]
TPHs	x	–
Explosives/munition compounds	x	–
Radiological screening/gamma spectroscopy	x	–
Headspace analysis (VOCs)	x	–
VOCs	x	x[c]
SVOCs[d]	NA	x
Active soil gas	x	–
Passive soil gas (sampling only)	O[e]	–
Toxic chemicals — bioluminescence (MicroTox)	x	NA
Microbial assessment for occurrence (MICKIT III)	x	NA

[a] "x" denotes parameters or categories of parameters being considered for analysis.

[b] "–" denotes parameters or categories of parameters not being considered for analysis.

[c] NA = not applicable.

[d] SVOCs = semivolatile organic compounds.

[e] "O" denotes analysis of on-site samples in collection tubes in off-site prime laboratory.

The OSL will be set up by ANL during RI activities at J-Field with the assistance and collaboration of the CRDEC at APG. The main objective of the OSL will be to assist in sample analyses and to test samples of soil, dust, and air for radiological contamination during Stage I of the RI. The analytical methods to be used in on-site screening are summarized in Table 8.3. The results of the Stage I analyses will be used to select samples for detailed analysis at the off-site prime and referee laboratories during Stages II and III. The off-site laboratories will subject samples to comprehensive testing within established and strict QA/QC protocols and analytical procedures described in this QAPjP and the QA/QC manuals of the prime and referee laboratories. The laboratories will provide QA/QC documentation with the data reports.

TABLE 8.2 Summary of Proposed Testing Facilities for Specific Parameters and Categories

Parameters/Category	Responsible Facility[a]	
	On-Site Analysis	Off-Site Analysis
Metals	OSL	PL/RL
PCBs	OSL	PL/RL
Total PAHs	CRDEC	NA
TPHs	CRDEC	NA
Explosives/munition compounds	APG	NA
Radiological screening/gamma spectroscopy	OSL	NA
Headspace analysis (VOCs)	OSL	NA
VOCs	OSL	PL/RL
SVOCs[b]	NA	PL/RL
Active soil gas	OSL	NA
Passive soil gas (sampling only)	NA	PL
Toxic chemicals — bioluminescence (MicroTox)	CRDEC	NA
Microbial assessment for occurrence (MICKIT III)	OSL	NA

[a] Acronyms: OSL = on-site laboratory; PL = prime laboratory/contractor; RL = referee laboratory; CRDEC = Chemical Research Development and Engineering Center; NA = not applicable; APG = Aberdeen Proving Ground.

[b] SVOCs = semivolatile organic compounds.

During Stage I, a variety of analytical parameters will be screened on-site with available state-of-the-art instrumentation and methods. The Stage I analytical suite of parameters and tests includes the following:

- Metals;
- PCBs;
- Total PAHs;
- TPHs;
- Nitroaromatics — explosives;
- VOCs and some SVOCs, including phthalates and others;
- Toxic chemicals (MicroTox); and
- Various bacterial types (MICKIT).

The assessments for some of these parameters are summarized in the following subsections.

TABLE 8.3 Summary of Stage I Analytical Methods

Parameter	Technique/Analytical Method	SOP[a]/Reference
Metals	Energy-dispersive XRF (EDXRF) technique	SOP-S001
PCBs, Total PAHs, and TPHs	Semiquantitative estimation immunoassay field screening method for rapid PCB analysis	Van Emon and Mumma 1990, EPA 1991b, SOP-S002
Radiological screening	Portable radiation meters, soil and air sampling for gross alpha and gross beta detection	SOP-S004
	Gamma emitters — Geiger-Müller instruments, survey for gamma emission and gamma spectroscopy	
	Beta emitters — Ludium Model 2200	
Headspace analysis	Photoionization detector(s)	SOPs-011, 024
	Flame ionization detector/field gas chromatography	SOP-023
	EPA Method 3810, Head Space Technique	EPA 1986a
Active soil gas	Gas chromatography	SOPs-020, 026
Passive soil gas	Gas chromatography	SOP-027, S005
Toxic chemicals	Bioluminescence with MicroTox	-[b]
Microbial assessment	Bacterial culture/count with MICKIT III	SOP-S003
Soil and/or sediment physical parameters	USCS[c] classification	ASTM[d] Method D-2487
	Percent moisture	ASTM Method D-2216
	Liquid limit	ASTM Method D-4318
	Plastic limit	ASTM Method D-4318
	Plasticity index	ASTM Method D-4318
	Organic content	ASTM Method D-2974
	Grain size distribution	ASTM Method D-422

[a] SOPs developed by the U.S. Army Corps of Engineers are described in COE (1993); those developed by ANL (designated with an "S," e.g., SOP-S001) are presented in Appendix A.

[b] MicroTox analyses to be performed at an off-site laboratory.

[c] USCS = Unified Soil Classification System.

[d] ASTM = American Society for Testing and Materials.

8.2.1 Metals

Selected metals in soil will be analyzed with state-of-the-art elemental analyzers based on the energy-dispersive XRF (EDXRF) technique (SOP-S001). A modern instrument based on this technique can detect a majority of the metals included in EPA's CLP TAL (EPA 1989b). For example, the SPECTRACE 9000,[1] an instrument based on XRF technology, can detect 16 of the 23 TAL metals, including antimony, arsenic, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, potassium, selenium, and zinc.

8.2.2 Polychlorinated Biphenyls, Total Polyaromatic Hydrocarbons, and Total Petroleum Hydrocarbons

A state-of-the-art immunoassay field screening method described in SOP-S002 will be used to detect the potential presence of PCBs (including Aroclors), total PAHs, and TPHs in on-site soil samples. This methodology, which provides a semiquantitative estimation for these compounds, is discussed in more detail by Van Emon and Mumma (1990) and the EPA (1991b).

8.2.3 Munition Compounds and Chemical Warfare Agents

CWA screening will be conducted during field activities and by the CRDEC in the CRDEC Laboratory with methods developed at APG by Bossle et al. (1988, 1989, 1990). A field technique based on colorimetry developed by USATHAMA (USATHAMA 1984) will be used in the OSL to analyze for nitroaromatics.

8.2.4 Radiological Assessment

8.2.4.1 Screening for Gamma Emitters

Qualified personnel will conduct field radiation screening for gamma emissions to determine the presence of the radionuclides radium-226 and cesium-137. These radionuclides will be monitored because of historical information on materials potentially present at J-Field. Qualified personnel will also assist if a sample to be shipped to the prime or referee laboratory for further testing must be packaged and transported as a special radioactive shipment. Field radiation screening will consist of the following steps:

- Soil will be surveyed in screening and homogenization pans for gamma radiation before the soil is placed in containers.

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[1] Manufactured by Spectrace Instruments, Inc., Skillman, New Jersey.

- Handheld monitoring equipment, such as Geiger-Müller instruments, will be used to survey each sample. These instruments will be calibrated before they are used in the field. The survey will be conducted only by qualified and trained personnel.

- A contact gamma survey will be performed on the outside of the sample container if it is to be shipped to the prime or referee laboratory for further analysis. In the case of a nonuniform sample (e.g., soil samples), readings will be taken on all sides of the container. A contact reading will also be taken on the bottom of the sample bottle.

- All results will be recorded in a radiation screening log book.

- For shipping purposes, a soil or sediment sample is considered radioactive if it contains a specific activity greater than 2×10^3 pCi/g. Samples will be shielded to protect the health and safety of the public and personnel who will handle the sample containers, including transport workers. Samples will be packaged in steel-belted coolers and checked by personnel on-site to ensure readings are less than 0.5 mrem/yr at contact.

A conceptual SOP has been developed for use in field sampling and screening for radiological parameters (SOP-S004 in Appendix A). Rapid turnaround analysis can be accomplished with personal-computer-based multichannel analyzers (MCAs) and sodium iodide (NaI) or germanium photon or bismuth-germinate oxygen detectors in an on-site or off-site laboratory setting. A Canberra MCA with a Ludium 44-10 NaI detector (or equivalent instrument) is acceptable. Dried soil samples in fixed geometries (Petri dishes) can be analyzed in approximately 20 to 30 minutes with detection limits of approximately 5 pCi/g for radionuclides such as cesium-137.

8.2.4.2 Screening for Beta Emitters/Gross Beta

In measuring beta emitters such as strontium-90, soil samples will be dried, homogenized, placed in a Petri dish, and counted for finite time periods (5 minutes to 24 hours). An appropriate beta detector and scaler will be used for soils or other dried solids.

8.2.5 Soil-Gas Chemical Analysis

Active and passive soil gas will be sampled as outlined in SOPs-020, 026, and 027 (COE 1993) and in SOP-S005 (Appendix A). Soil-gas samples will be analyzed by noncertified USATHAMA, EPA CLP, and ASTM methods.

8.2.5.1 Active Soil-Gas Sampling

The instrument to be used in the analysis of the active soil-gas samples is a self-contained, field-portable gas chromatograph equipped with a combination argon ionization/electron capture detector. At APG, the detector will be operated in the argon ionization detector (GC/AID) mode, which is sensitive to compounds with an ionization potential at or below 11.6 eV. The instrument is controlled by a detachable, portable computer that records and integrates peaks, identifies peaks from calibration standards and libraries, and displays and records each trace, along with operating parameters and analysis results.

The GC/AID is calibrated to benzene before analysis of the samples. The sample is screened by a PID to determine the general concentration level of the VOCs and the possible presence of hazardous compounds. In the event that the PID indicates high levels of VOCs, dilutions will be made to ensure that specific compounds do not exceed the range of calibration of the GC/AID. Retention times of observed peaks are compared to a library file of compounds with known retention times. Compounds are identified and quantified on the basis of this library comparison. Compounds that are not identified are designated as unknowns and are quantitated on the basis of the response of benzene itself. Retention time requirements for benzene will be met, and instrument sensitivity will be demonstrated for all on-site analyses of active soil-gas samples.

8.2.5.2 Passive Soil-Gas Sampling

Passive soil-gas samples will be analyzed by either a Cp/MS or by thermal desorption GC/MS. The compounds that are typically detected by Cp/MS and thermal desorption GC/MS are listed in Table 8.4.

For analysis by Cp/MS, the ferromagnetic wire containing the bonded charcoal is thermally desorbed directly into an electron ionization quadrapole mass spectrometer. The desorbed soil-gas constituents are ionized and fragmented by electron impact. The resulting positively charged mass fragments in a range of 30 to 240 atomic mass units are separated by the quadrapole. The resulting signal corresponds to the intensity of a specific mass ion. The identification of the compound is determined by the presence of primary and secondary mass ions at ratios indicative of the target compound. The ion of quantification is chosen (normally the base mass ion) such that background ions or ions associated with other compounds do not contribute to the signal. The result for Cp/MS is reported as total ion intensity (termed flux) per compound identified.

The analysis of samples by thermal desorption GC/MS involves thermal desorption of the ferromagnetic wire containing the charcoal, followed by cyrofocusing of the desorbed soil gas, separation of the soil-gas constituents by gas chromatography (GC), and analysis of the separated constituents by the MS. This variation of Cp/MS is advantageous in that each

TABLE 8.4 List of Passive Soil-Gas Analytes

Aromatics (Benzene-Based)

All aromatic hydrocarbons from C_6 (benzene) to C_{12} (C_6 alkyl benzene) including specifically identified:

Benzene	Xylenes	Ethyl benzene	Propyl benzenes
Toluene	Ethyl methyl benzene	Trimethyl benzenes	

Alkanes (Aliphatic/Paraffins)

All alkane hydrocarbons from C_4 (butane) to C_{15} (pentadecanes), plus C_2 (ethane), including alkanes with various alkyl groups attached. All cycloalkanes with various alkyl groups attached, including specifically:

Ethane	Tridecanes	Cyclodecanes	Ethyl methyl cyclohexane
Butanes	Octadecanes	Octyl cyclopropane	Ethyl methyl ethyl cyclohexane
Pentanes	Cyclopropane	Methyl cyclopentane	
Hexanes	Cyclobutanes	Methyl propyl cyclopentane	Methyl octa decane
Heptanes	Cyclopentanes	Methyl hexane	Dimethyl heptane
Octanes	Cyclohexanes	Trimethyl hexane	Dimethyl heptane
Nonanes	Cyclo octanes	Trimethyl cyclohexane	Dimethyl octane
Undecanes	Cyclononanes		Ethyl methyl octane
Dodecanes			

Volatile Halogenated Compounds

Vinyl chloride	Trichloroethanes	Dichloropropene	Trichlorotrifluoromethane
Chloromethane	Tetrachloroethane	Trichloropropene	Bromoform
Methylene chloride	Dichloropropanes	Chlorobenzene	Dibromoethane
Chloroform	Dichloroethenes	Chlorotoluene	Bromodichloromethane
Carbon tetrachloride	Trichloroethene	Dichlorodifluoromethane	Dibromochloromethane
Chloroethane	Tetrachloroethene	Trichlorofluoromethane	Bromodichloropropane
Dichloroethanes			

Semivolatile Organic Compounds

Hexachloroethane	Hexachlorobenzene	C_2-C_4 naphthalenes	Nitrobenzene
Hexachlorocyclohexane	Dibromochloropropane	Chlorophenols	Nitrotoluene
Hexachlorobutadiene	Phenol	Chloronaphthalenes	Dinitrotoluene
Hexachloropentadiene	Methyl phenol	Chlorobenzotrifluoride	Anthracene
Dichlorobenzenes	C_2-C_4 phenol	Dichlorobenzotrifluoride	Phenanthrene
Trichlorobenzene	Naphthalene	Trichlorobenzotrifluoride	Acenaphthalene
Tetrachlorobenzene	Methyl naphthalenes		

Sulfur Compounds

Hydrogen sulfide	Sulfur dioxide	Carbon disulfide	Carbonyl sulfide

Alkenes (Olefins)

All alkenes from C_2 (ethylene) to C_{15} (pentadecene), including alkenes with various alkyl and other hydrocarbon groups attached. Also, C_4 to C_{15} cycloalkenes, including those with various alkyl groups and other hydrocarbons attached including specifically:

Ethylene	Heptenes	Cyclopentene	Cyclodecene
Propylene	Octenes	Cyclohexene	Methyl pentene
Butenes	Nonenes	Cycloheptene	Methyl cyclohexene
Pentenes	Decenes	Cyclo-octene	
Hexenes	Cyclobutene	Cyclononene	

TABLE 8.4 (Cont.)

Dienes	*Alkynes*	*Styrenes*	
Dienes from C_6-C_{16}	Alkynes from C_6-C_{16}	Styrene Methyl styrene C_2-C_6 styrenes	
Other Detectable Compounds			
Ethanol	Dimethyl butanol	Butanone	Tridecanone
Methoxyethanol	Hexanol	Methyl butanone	Aldehyde
Propanol	Nonanol	Hexanone	Benzaldehyde
Butanol	MEK	Methyl hexanone	Acetaldehyde
Mixtures			
PETREX has detected and can characterize fresh and aged hydrocarbon mixtures, including:			
Gasolines (leaded/unleaded)	Aviation gasoline	Lubricants (light oils to grease)	Coolants
Diesel fuels	White gasoline	Cutting oils	Sea oils
Jet fuels (JP4/JP5)	Hydraulic fluids		Creosote

detected compound will produce a unique mass spectrum. This analysis will enable verification of the mass ion assignments for the detected soil-gas constituents by comparison with existing mass spectrum databases. In addition, if an unknown soil-gas constituent is detected, the compound can be determined by a library search, and an ion quantitation can be established for data reduction of the Cp/MS results. The thermal desorption GC/MS can specifically be used to determine the presence of the relatively volatile constituents of explosive degradation products, such as nitrobenzene and dinitrotoluene, as well as degradation products of chemical agents, such as 1,4-dithiane and bis(2-chloroethyl)sulfide. Analysis by thermal desorption GC/MS will be conducted on samples from areas of high soil-gas concentration, at a frequency of 1 in 5 for all other samples.

8.2.5.3 Passive Soil-Gas Surveys Using EMFLUX Soil-Gas Analysis System

Passive soil gas surveys are conducted using the EMFLUX soil-gas analysis system described in SOP S005 (Appendix A). The organic compounds that are detected by thermal desorption GC/MS for the EMFLUX passive soil-gas survey samples are listed in Table 8.5.

EMFLUX cartridges are thermally desorbed and analyzed, in accordance with EPA-approved techniques, with a modified EPA Method 8240 (EPA 1986a) to analyze all samples with GC/MS equipment. Each cartridge is placed in a Tekmar Autosampler chamber, where it is desorbed at 270°C for 11 minutes at 40 mL/min helium flow through a sparging vessel containing 5 mL of water with internal standards and surrogates into a three-component trap

TABLE 8.5 List of Passive EMFLUX Soil-Gas Survey Analytes of Interest

Acetone	1,2-Dichloropropane
Benzene	*cis*-1,3-Dichloropropene
Bromodichloromethane	*trans*-1,3-Dichloropropene
Bromoform	Ethylbenzene
Bromomethane	1-Hexanone
2-Butanone	4-Methyl-2-pentanone
Carbon disulfide	Methylene chloride
Carbon tetrachloride	Styrene
Chlorobenzene	1,1,2,2-Tetrachloroethane
Chloroethane	Tetrachloroethylene
Chloroform	Toluene
Chloromethane	1,1,1-Trichloroethane
Dibromochloromethane	1,1,2-Trichloroethane
1,1-Dichloroethane	Trichloroethylene
1,2-Dichloroethane	Vinyl acetate
1,1-Dichloroethylene	Vinyl chloride
1,2-Dichloroethylene (total)	Xylenes (total)

on a Tekmar Liquid Sample Concentrator. The three components in the secondary trap are Tenax, silica gel, and coconut charcoal. The secondary trap is thermally desorbed at 220°C into a Restek 502.2 capillary column, in accordance with the EPA CLP Statement of Work (SOW) (EPA 1991a). Following the secondary thermal desorption, the GC/MS is scanned between 35 and 260 atomic mass units at 2 seconds per scan. The GC/MS equipment is calibrated in accordance with the EPA CLP method for water analyses (EPA 1991a). The internal standard method is used to determine the amount of analytes found. The organic compounds found are measured against 5 mL of aqueous standards tested previously.

8.2.6 Headspace Analysis for Volatile Organic Compounds

A portable HNu meter (PI-101) with a photoionization detector and/or a portable OVA (Century OVA 128) with a flame ionization detector/gas chromatography capability will be used to screen areas suspected of being contaminated with VOCs. Soil samples will also be collected in sealed glass containers and allowed to equilibrate at 90°C in the OSL as described in EPA Method 3810, SOW 846 (EPA 1986a). A sample of the headspace gas is withdrawn with a gas-tight syringe for screening analysis under conditions specified by EPA Method 8240 (EPA 1986a) or by one of the GC or GC/MS determinative methods for VOCs in a variety of solid matrices regardless of water content (EPA Methods 8010, 8015, 8020, 8030, or 8240 [EPA 1986a]).

A PID will also be used to measure the total ionizable concentration of organic vapors of samples collected by the methods described above. The instrument manufacturers' specifications for analysis of volatile organic compounds are presented in SOPs-011 and 024 (COE 1993). Quality assurance of measurements will be addressed by calibrating the

instrument, standardizing the measurement procedure, and analyzing appropriate QA samples, including periodic blanks, duplicates, and spiked samples. Instruments will be calibrated frequently with a certified calibrant gas, such as propane, to ensure accuracy and comparability. Precision will be evaluated to replicate analyses of a fixed percentage of samples. Standard data forms will be used to record data and ensure completeness.

8.2.7 Microbial Assessment

A state-of-the-art procedure will be used to conduct field determination for toxic chemicals in soils on the basis of bioluminescence. This method involves the use of the MicroTox test kit.

A state-of-the-art method will also be used to detect the potential presence of microorganisms in soil based on their oxygen requirements. The field scanning method, which permits microbial counting, will be used. This method, which uses the MICKIT, is discussed in SOP-S003 (Appendix A). This SOP also lists the detectable microorganisms.

8.2.8 Physical Analyses of Soil Samples

A subset of the environmental solid samples, including soil and sediment, will be subjected to physical analysis. The physical data will be used to interpret the movement of contaminants in the vadose zone and aquifers in the J-Field area and to verify boring logs. Percent moisture will be determined by ASTM Method D-2216. This method involves the determination of the percent water mass in a known mass of undried soil by weighing the soil before and after drying in an oven controlled at 110°C. The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles.

Grain-size distribution will be determined by ASTM Method D-442. This method covers the quantitative determination of the distribution of particle sizes in soil. A No. 200 sieve is used to separate particles larger than 75 μm from the soil, while the distribution of particles smaller than 75 μm is determined by a sedimentation process that uses a hydrometer to secure the necessary data.

The organic content of soils will be determined by ASTM Method D-2974. This method involves the ignition of an oven-dried soil sample in a muffle furnace. The weight of the sample is taken before and after ignition, and the organic mass is the difference of the two masses. The organic content (a percentage) is expressed as this difference divided by the weight of the sample before ignition.

Classification of soils will be based on laboratory determination of particle-size characteristics, liquid limit, and plasticity index by use of ASTM Method D-2487. The system is based on the Unified Soil Classification System (USCS).

Personnel performing physical analyses of soil samples are independent from laboratory performance evaluations and do not participate in the conduct of performance evaluation audits, including those under direction of the EPA.

8.3 ANALYTICAL PROCEDURES OF PRIME AND REFEREE LABORATORIES

Facilities for detailed chemical analysis will be available at the prime laboratory (or a contractor facility) and at a separate referee laboratory. The prime laboratory/contractor facility will use established methods of analysis (as discussed below) under strict QC measures.

8.3.1 Sample Holding Times — Program Requirements

In accordance with the QA and analytical program requirements, the prime laboratory will process the J-Field samples as soon as possible after receipt. To fulfill project requirements, analyses will be complete within the sample/extract holding time. Upon completion of analysis, results will be reported by phone to the ANL ECC. The prime laboratory and referee laboratory will thereafter submit a written report on the results. All samples will be returned to ANL/APG at the ANL project manager's direction upon completion of analysis. The samples may be forwarded to the APG waste disposal contractor with waste material.

8.3.2 Laboratory Analytical Procedures

CLP methodologies will be used to analyze TCL volatile organic compounds, TCL semivolatile organic compounds, TAL inorganic compounds, pesticides, and PCBs. USATHAMA methodologies, including those developed by the CRDEC at APG (Bossle et al. 1988, 1989, 1990), will be used for chemical analysis of explosives, CWA degradation products, and water chemistry analytes. These analytes are listed in Tables 8.6 through 8.11. The analytes involved in the passive and active soil-gas survey are listed in Tables 8.4 and 8.12, respectively. Table 8.13 lists physical parameters that will be determined or indirectly calculated for the soil samples. The following subsections briefly describe laboratory analytical methods that will be used for RI analyses of J-Field samples.

8.3.2.1 Inorganic Chemical Analyses for Aqueous and Solid Media

EPA TAL inorganic parameters will be analyzed by one of the following CLP methodologies (Table 8.14): inductively coupled plasma spectroscopy (ICP), graphite furnace atomic absorption spectroscopy (GFAA), or cold vapor atomic absorption (CVAA). Water chemistry analytes will be analyzed via autoanalyzer or ion chromatography (IC). Sample preparation, including digestion of metals, will be in accordance with CLP procedures.

TABLE 8.6 List of TCL Volatile Organic Compounds

Acetone	1,2-Dichloropropane
Benzene	*cis*-1,3-Dichloropropene
Bromodichloromethane	*trans*-1,3-Dichloropropene
Bromoform	Ethylbenzene
Bromomethane	2-Hexanone
2-Butanone	Methylene chloride
Carbon disulfide	4-Methyl-2-pentanone
Carbon tetrachloride	Styrene
Chlorobenzene	1,1,2,2-Tetrachloroethane
Chloroethane	Tetrachloroethylene
Chloroform	Toluene
Chloromethane	1,1,1-Trichloroethane
Dibromochloromethane	1,1,2-Trichloroethane
1,1-Dichloroethane	Trichloroethylene
1,2-Dichloroethane	Vinyl chloride
1,1-Dichloroethylene	Xylenes (total)
1,2-Dichloroethylene (total)	

The ICP method involves the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by optical spectrometry. Samples are nebulized, and the aerosol that is produced is transported to the plasma torch, where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored and controlled by a computer system. A background correction technique is used to compensate for variable background contribution to the determination of trace elements.

Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used will be free of spectral interference and will reflect the same change in background intensity as it occurs at the analyte wavelength measured.

Background correction will not be required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Interferences will also be recognized and appropriate corrections will be made. The CLP method number for analysis of ICP metals is Method 200.7 CLP-M (Modified).

To obtain reporting limits lower than those provided by the ICP method, arsenic, lead, and selenium will also be analyzed with GFAA, which involves the digestion of a representative sample with nitric acid and hydrogen peroxide. The digestate is subsequently analyzed by GFAA with the optimum instrumental conditions for the analytes of interest. The corresponding CLP method numbers from ILM02.0 are Method 239.2 CLP-M (lead); Method 270.2 CLP-M (selenium); and Method 206.2 CLP-M (arsenic).

TABLE 8.7 List of TCL Semivolatile Organic Compounds

Acenaphthene	Di-*n*-octylphthalate
Acenaphthylene	4,6-Dinitrophenol
Anthracene	2,4-Dinitrotoluene
Benz[*a*]anthracene	2,4-Dinitrotoluene
Benzo[*b*]fluoranthene	2,6-Dinitrotoluene
Benzo[*k*]fluoranthene	Fluoranthene
Benzo[*g,h,i*]perylene	Fluorene
Benzo[*a*]pyrene	Hexachlorobenzene
4-Bromophenyl phenyl ether	Hexachlorobutadiene
Butylbenzylphthalate	Hexachlorocyclopentadiene
bis(2-Chloroethoxyl)methane	Hexachloroethane
bis(2-Chloroisopropyl)ether	Indeno[*1,2,3-c,d*]pyrene
bis(2-Chloroisopropyl)ether	Isophorone
bis(2-Ethylhexyl)phthalate	2-Methylnaphthalene
Carbazole	2-Methylphenol
4-Chloroaniline	4-Methylphenol
4-Chloro-3-methylphenol	Naphthalene
2-Chloronaphthalene	2-Nitroaniline
2-Chlorophenol	3-Nitroaniline
4-Chlorophenyl phenyl ether	4-Nitroaniline
Chrysene	Nitroaniline
Dibenz[*a,h*]anthracene	2-Nitrophenol
Dibenzofuran	4-Nitrophenol
1,2-Dichlorobenzene	*N*-Nitroso-di-*n*-propylamine
1,3-Dichlorobenzene	*N*-Nitrosodiphenylamine
1,4-Dichlorobenzene	Pentachlorophenol
3,3'-Dichlorobenzidine	Phenanthrene
2,4-Dichlorophenol	Phenol
Diethylphthalate	Pyrene
2,4-Dimethylphenol	1,2,4-Trichlorobenzene
Dimethylphthalate	2,4,5-Trichlorophenol
Di-*n*-butylphthalate	2,4,6-Trichlorophenol

TABLE 8.8 List of TAL Inorganic Compounds

Aluminum	Iron
Antimony	Lead
Arsenic	Magnesium
Barium	Manganese
Beryllium	Mercury
Cadmium	Nickel
Calcium	Potassium
Chromium	Thallium
Cobalt	Vanadium
Copper	Zinc
Cyanide	

TABLE 8.9 List of TCL PCBs (Aroclors) and Pesticides

Aroclor-1016	4,4′-DDD
Aroclor-1221	4,4′-DDE
Aroclor-1232	4,4′-DDT
Aroclor-1242	Dieldrin
Aroclor-1248	Endosulfan I
Aroclor-1254	Endosulfan II
Aroclor-1260	Endosulfan sulfate
Aldrin	Endrin
alpha-BHC	Endrin aldehyde
beta-BHC	Endrin ketone
delta-BHC	Heptachlor
gamma-BHX (Lindane)	Heptachlor epoxide
alpha-Chlordane	Methoxychlor
gamma-Chlordane	Toxaphene

TABLE 8.10 List of Explosives Analytes

Cyclotrimethylene trinitramine (RDX)
Cyclotetramethylene tetranitramine (HMX)
1,3-Dinitrobenzene
2,4-Dinitrobenzene
2,6-Dinitrobenzene
n-Methyl-*n*-2,4,6-tetranitrobenzoaniline (TETRYL)
Nitroglycerin
Pentaerythritol tetranitrate (PETN)
1,3,5-Trinitrobenzene
2,4,6-Trinitrotoluene

To obtain a reporting limit lower than that provided by the ICP method, mercury will also be analyzed with CVAA. The CLP method numbers for mercury with CVAA are Method 245.2 CLP-M for aqueous samples and Method 245.5 CLP-M for solid samples. The CVAA analysis is based on the absorption of radiation at 253.7 nm by mercury vapor. A sample aliquot is initially digested with nitric acid to free any combined mercury. Solid samples undergo oxidation with potassium permanganate and potassium persulfate after the acid wash. The mercury is reduced to its elemental state and aerated from the solution into a closed system. The mercury vapor is passed through a cell positioned in the path of a mercury light source, and the measured absorbance is proportional to the concentration of mercury in the sample.

Cyanide will be analyzed in aqueous and solid samples using Methods 335.2 CLP-M and 335.3 CLP-M, respectively. The cyanide is released as hydrocyanic acid from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing

TABLE 8.11 List of Chemical Warfare Agent Degradation Products[a]

Benzathiazole
1,4-Dithiane
1,4-Oxathiane
p-Chlorophenylmethyl sulfone
p-Chlorophenylmethyl sulfide
p-Chlorophenylmethyl sulfoxide
Dimethyl disulfide
Diisopropyl methylphosphonate
Dimethyl methylphosphonate
Methylphosphonic acid
Isopropylmethylphosphonic acid
Thiodiglycol

[a] This table lists CWA degradation products detectable by available analytical methods. These products will be tested in Stage I. Additional compounds and their quantitation limits and analytical methods are provided in Table 8.18. (Many more CWA degradation products, as well as stabilizers that may be present and detectable, will be listed in data reports, depending on analytical method and level of detection.)

TABLE 8.12 List of Active Soil-Gas Analytes

Acetone	Methyl ethyl ketone
Benzene	Styrene
Carbon disulfide	1,1,2,2-Tetrachloroethane
Carbon tetrachloride	Toluene
Chloroform	1,1,2-Trichloroethane
1,1-Dichloroethylene	Trichloroethylene
trans-1,2-Dichloroethylene	*m*-Xylene
Ethylbenzene	*o*-Xylene
Isothane	*p*-Xylene
Methylene chloride	

sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically. In the colorimetric measurement, the cyanide is converted to cyanogen chloride by reaction with chloramine-T at a pH <8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridinebarbituric acid reagent. The absorbance is read at 620 nm for pyridine-pyrazolone or 578 nm for pyridinebarbituric acid. To obtain colors of comparable intensity, the sample and the standards will contain the same salt content. The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver-sensitive indicator.

TABLE 8.13 List of Physical Testing Parameters for Soil and Sediment Samples

USCS classification
Percent moisture
Liquid limit
Plastic limit
Plasticity index
Organic content
Grain size distribution

Total phosphorus will be analyzed by an autoanalyzer (USATHAMA Method TF27) in aqueous and solid samples. The sample is heated in the presence of H_2SO_4 and $(NH_4)_2S_2O_3$ for 30 minutes. The residue is cooled, diluted to 50 mL, and placed on the autoanalyzer for phosphorus determination. All forms of phosphate are converted to orthophosphate and determined colorimetrically. Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of orthophosphate to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the orthophosphate concentration.

8.3.2.2 Organic Chemical Analyses for Aqueous and Solid Media

All organic constituents will be analyzed in accordance with CLP methodologies (Tables 8.15 through 8.17) involving gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), gas chromatography with electron-capture detection (GC-ECD), gas chromatography with flame-photometric detection (GC-FPD), high-pressure liquid chromatography (HPLC), ion chromatography (IC), high-resolution capillary column gas chromatography/low-resolution mass spectrometry (HRGC/LRMS), Curie-point pyrolizer mass spectrometer (Cp/MS), and thermal desorption GC/MS.

The method for analyzing VOCs and base-neutral acid extractables (BNAs) (Method OLM01.0) involves purging of environmental sample and volatile organic-free water containing surrogates and internal standards with helium gas (following extraction). The purging chamber is heated to a predefined temperature and the vapor is transferred to a sorbent tube, which effectively traps the volatile organic compounds. The constituents are

TABLE 8.14 Target Analyte List, Analytical Methods, and Detection Limits: Inorganic Chemicals[a]

Analyte	Detection Limits[b]			Analytical Method[c]		
	Soil/Sediment (mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Aluminum	40	200	50[d]	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 202.2 EPA CLP-M
Antimony	12 (1)	5[e]	60	Method 200.7 EPA CLP-M	Method 204.2 EPA CLP-M	Method 200.7 EPA CLP-M
Arsenic	2	10	10	Method 206.2 EPA CLP-M	Method 206.2 EPA CLP-M	Method 206.2 EPA CLP-M
Barium	40	200	200	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Beryllium	1	1[e]	5	Method 210.2 EPA CLP-M	Method 210.2 EPA CLP-M	Method 200.7 EPA CLP-M
Cadmium	1	5	0.5[d]	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 213.2 EPA CLP-M
Calcium	1,000	5,000	5,000	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Chromium	2	10	10	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Cobalt	10	50	50	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Copper	5	25	1[d]	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 220.2 EPA CLP-M
Cyanide (total)	0.7[f]	10	1[d]	EPA CLP Spectrophotometric-Automated	EPA CLP Spectrophotometric-Automated	EPA CLP Spectrophotometric-Automated

TABLE 8.14 (Cont.)

Analyte	Detection Limits[b]			Analytical Method[c]		
	Soil/Sediment (mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Iron	20	100	100	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Lead	0.6	3	1[d]	Method 239.2 EPA CLP-M	Method 239.2 EPA CLP-M	Method 239.2 EPA CLP-M
Magnesium	1,000	5,000	5,000	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Manganese	3	15[d]	15	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Mercury	0.1 (0.2)[g]	0.2	0.01[d]	Method 245.2 EPA CLP-M	Method 245.2 EPA CLP-M	Method 245.2 EPA CLP-M
Nickel	8	40	5[d]	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 249.2 EPA CLP-M
Potassium	1,000	5,000	5,000	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Selenium	1	5	5	Method 270.2 EPA CLP-M	Method 270.2 EPA CLP-M	Method 270.2 EPA CLP-M
Silver	2	10	0.1[d]	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 272.2 EPA CLP-M
Sodium	1,000	5,000	5,000	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Thallium	2	1[e]	10	Method 279.2 EPA CLP-M	Method 279.2 EPA CLP-M	Method 279.2 EPA CLP-M

TABLE 8.14 (Cont.)

Analyte	Detection Limits[b] Soil/Sediment (mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Analytical Method[c] Soil/Sediment	Groundwater	Surface Water
Vanadium	10	50	50	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M
Zinc	4	20	20	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M	Method 200.7 EPA CLP-M

[a] Recommended detection limits are equal to the EPA's contract-required detection limits (CRDLs), unless otherwise noted.

[b] When a recommended risk-based value is not achievable using standard analytical methods, an alternate value is presented in parentheses.

[c] EPA (1989b). All referenced methods are modified in accordance with the CLP SOW.

[d] Recommended alternate quantitation limit based on potential aquatic toxicity.

[e] Recommended alternate quantitation limit based on potential human toxicity.

[f] The quantitation limit for cyanide in soil and sediment was determined by the following assumptions: semiautomated spectrophotometric method, cyanide CRDL, 5-g sample, 250-mL final volume of extraction solution, 70% solids, and 100% scrubbing efficiency.

[g] The detection limit for mercury in soil is the minimum value given for the range of the Method 245.5 CLP-M.

TABLE 8.15 Target Compound List, Analytical Methods, and Quantitation Limits: Volatile Organic Compounds[a]

Analyte	Quantitation Limits[b]			Analytical Method[c,d]		
	Soil/Sediment (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Acetone	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benzene	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Bromodichloromethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Bromoform	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Bromomethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Butanone	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Carbon disulfide	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Carbon tetrachloride	10	10	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Chlorobenzene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Chloroethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Chloroform	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.15 (Cont.)

Analyte	Quantitation Limits[b]			Analytical Method[c,d]		
	Soil/Sediment (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Chloromethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Dibromochloromethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,1-Dichloroethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,2-Dichloroethane	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
1,1-Dichloroethylene	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
1,2-Dichloroethylene (total)	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,2-Dichloropropane	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
cis-1,3-Dichloropropene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
trans-1,3-Dichloropropene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Ethylbenzene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Hexanone	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.15 (Cont.)

Analyte	Quantitation Limits[b] Soil/Sediment (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Analytical Method[c,d] Soil/Sediment	Groundwater	Surface Water
Methylene chloride	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
4-Methyl-2-pentanone	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Styrene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,1,2,2-Tetrachloroethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Tetrachloroethylene	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Toluene	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,1,1-Trichloroethane	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,1,2-Trichloroethane	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Trichloroethylene	10	5[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Vinyl chloride	10	2[e]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Xylenes (total)	10	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

See footnotes on next page.

TABLE 8.15 (Cont.)

[a] Recommended quantitation limits are equal to the EPA's contract-required quantitation limits (CRQLs), unless otherwise noted. The CRQL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For the CLP methodologies, the CRQL is determined by the lowest initial calibration standard that is run before the analytical sequence.

[b] Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment are calculated on a dry-weight basis and, therefore, will be higher.

[c] Source of analytical method SOW (OLM01.5) is EPA (1991b).

[d] Source of EPA Method 600 4-88 039 is EPA (1988b) (Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry). Note: Several hardware configurations exist for this method. The referenced values are for the following configurations: wide-bore capillary column, jet separator, and quadrapole mass spectrometer.

[e] Recommended alternate quantitation limit based on potential human toxicity.

TABLE 8.16 Target Compound List, Analytical Methods, and Quantitation Limits: Semivolatile Organic Compounds[a]

Analyte	Quantitation Limits[b]			Analytical Method[d]		
	Soil/Sediment[c] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Acenaphthene	330/150[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Acenaphthylene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Anthracene	330/50[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benz[*a*]anthracene	330/200[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benzo[*b*]fluoranthene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benzo[*k*]fluoranthene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benzo[*g,h,i*]perylene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Benzo[*a*]pyrene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Bromophenyl phenyl ether	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Butylbenzylphthalate	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Carbazole	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Chloroaniline	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.16 (Cont.)

Analyte	Quantitation Limits[b]			Analytical Method[d]		
	Soil/Sediment[c] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
bis(2-Chloroethyl)ether	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
bis(2-Chloroethoxy)methane	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Chloro-3-methylphenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Chloronaphthalene	330	10	5[e,f]	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Chlorophenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Chlorophenyl phenyl ether	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Chrysene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,2'-oxybis-(1-Chloropropane)[g]	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Dibenz[*a,h*]anthracene	330/50[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Dibenzofuran	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,2-Dichlorobenzene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,3-Dichlorobenzene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,4-Dichlorobenzene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.16 (Cont.)

Analyte	Quantitation Limits[b]			Analytical Method[d]		
	Soil/Sediment[c] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
3,3'-Dichlorobenzidine	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,4-Dichlorophenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Diethylphthalate	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,4-Dimethylphenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Dimethylphthalate	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4,6-Dinitro-2-methylphenol	800	25	25	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,4-Dinitrophenol	800	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,4-Dinitrotoluene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,6-Dinitrotoluene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Di-*n*-octylphthalate	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
bis(2-Ethylhexyl)phthalate	330	4[h]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA CLP SOW (OLM01.5)
Fluoranthene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Fluorene	330/30[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.16 (Cont.)

Analyte	Quantitation Limits[b]			Analytical Method[d]		
	Soil/Sediment[c] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Hexachlorobenzene	330	1[e,h]	5[f]	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039	EPA 600 4-88 039
Hexachlorobutadiene	330	10	5[f]	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039[i]
Hexachlorocyclopentadiene	330	10	5[f]	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039[i]
Hexachloroethane	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Indeno[*1,2,3-c,d*]pyrene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Isophorone	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Methylnaphthalene	330/50[e,f]	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Methylphenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Methylphenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Naphthalene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Nitroaniline	800	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
3-Nitroaniline	800	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Nitroaniline	800	25	25	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

TABLE 8.16 (Cont.)

Analyte	Quantitation Limits[b]			Analytical Method[d]		
	Soil/Sediment[c] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Nitrobenzene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2-Nitrophenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
4-Nitrophenol	800	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
N-Nitroso-di-*n*-propylamine	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
N-Nitrosodiphenylamine	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Pentachlorophenol	800	1[e,h]	5[f]	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039[i]	EPA 600 4-88 039[i]
Phenanthrene	330/200[e,f]	10	5[f]	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039[i]
Phenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
Pyrene	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
1,2,4-Trichlorobenzene	330	5[h]	10	EPA CLP SOW (OLM01.5)	EPA 600 4-88 039[i]	EPA CLP SOW (OLM01.5)
2,4,5-Trichlorophenol	800	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)
2,4,6-Trichlorophenol	330	10	10	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)	EPA CLP SOW (OLM01.5)

See footnotes on next page.

TABLE 8.16 (Cont.)

[a] Recommended quantitation limits are equal to the EPA's contract-required quantitation limits (CRQLs), unless otherwise noted. The CRQL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For the CLP methodologies, the CRQL is determined by the lowest initial calibration standard that is run before the analytical sequence.

[b] Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment are calculated on a dry-weight basis and, therefore, will be higher.

[c] If two values are listed, the first is the recommended quantitation limit of soil and the second is the recommended quantitation limit for sediment.

[d] Sources of analytical methods, except as noted: EPA CLP, SOW (OLM01.5) = EPA (1991b) (note that for semivolatile organic analysis of sediment samples, standing liquid should be discarded prior to extraction); EPA 600 4-88 039 = EPA (1988b) (Method 525, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry; note that several hardware configurations exist for this method and that the referenced values are for the ion trap mass-spectrometer configuration).

[e] Although this limit cannot be achieved using the referenced methodology, the compound can be detected and confirmed at less than the quantitation limit. This value would be confirmed but considered estimated and available for use in risk assessment.

[f] Recommended alternate quantitation limit based on potential aquatic toxicity.

[g] Previously known by the name bis(2-chloroisopropyl)ether.

[h] Recommended alternate quantitation limit based on potential human toxicity.

[i] EPA (1988b) (Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry; note that several hardware configurations exist for this method and that the referenced values are for the following configurations: wide-bore capillary column, jet separator, and quadrapole mass spectrometer).

TABLE 8.17 Target Compound List, Analytical Methods, and Quantitation Limits: Pesticides/PCBs/Aroclors[a]

Analyte	Quantitation Limits[b,c]			Analytical Method[e]		
	Soil/Sediment[d] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Soil/Sediment	Groundwater	Surface Water
Aldrin	1.7	0.05	0.05	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
alpha-BHC	1.7	0.05	0.05	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
beta-BHC	1.7	0.05	0.05	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
delta-BHC	1.7	0.05	0.05	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
gamma-BHC (lindane)	1.7	0.05	0.05	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
alpha-Chlordane	1.7/0.5[f] (1.7)	0.05	0.001[f] (0.0015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
gamma-Chlordane	1.7/0.5[f] (1.7)	0.05	0.001[f] (0.0015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
4,4-DDD	3.3/2[f] (3.3)	0.1	0.001[f] (0.0025)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
4,4-DDE	3.3/2[f] (3.3)	0.1	0.001[f] (0.01)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
4,4-DDT	3.3/1[f] (3.3)	0.1	0.001[f] (0.06)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Dieldrin	3.3/0.02[f] (3.3)	0.1	0.001[f] (0.02)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Endosulfan I	1.7	0.05	0.001[f] (0.015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Endosulfan II	3.3	0.1	0.001[f] (0.024)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057

TABLE 8.17 (Cont.)

Analyte	Quantitation Limits[b,c] Soil/Sediment[d] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Analytical Method[e] Soil/Sediment	Groundwater	Surface Water
Endosulfan sulfate	3.3	0.1	0.001[f] (0.015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Endrin	3.3/0.02[f] (3.3)	0.1	0.001[f] (0.015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Endrin aldehyde	3.3	0.1	0.001[f] (0.025)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Endrin ketone	3.3	0.1	0.001[f] (0.01)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
Heptachlor	1.7	0.05	0.001[f] (0.01)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Heptachlor epoxide	1.7	0.05	0.001[f] (0.015)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Methoxychlor	17	0.5	0.01[f] (0.05)[g]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057
Aroclor-1016	33	1.0	0.01[f] (0.08)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]
Aroclor-1221	67	2.0	0.01[f] (0.08)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]
Aroclor-1232	33	1.0	0.01[f] (2.0)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)
Aroclor-1242	33	1.0	0.01[f] (0.48)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]
Aroclor-1248	33	1.0	0.01[f] (0.31)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]
Aroclor-1254	33	1.0	0.01[f] (0.10)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]

TABLE 8.17 (Cont.)

Analyte	Quantitation Limits[b,c] Soil/Sediment[d] (µg/g or mg/kg)	Groundwater (µg/L)	Surface Water (µg/L)	Analytical Method[e] Soil/Sediment	Groundwater	Surface Water
Aroclor-1260	33	1.0	0.01[f] (0.19)[h]	CLP SOW (OLM01.5)	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]
Toxaphene	170	1[h]	0.0001[f] (1.0)[h]	CLP SOW (OLM01.5)	EPA-600/ 4-82-057[i]	EPA-600/ 4-82-057[i]

[a] Recommended quantitation limits are equal to the EPA's contract-required quantitation limits (CRQLs), unless otherwise noted. The CRQL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. For the CLP methodologies, the CRQL is determined by the lowest initial calibration standard that is run before the analytical sequence.

[b] Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment are calculated on a dry-weight basis and, therefore, will be higher.

[c] When a recommended risk-based value is not achievable using standard analytical methods, an alternate value is presented in parentheses. The value within the parentheses is the quantitation limit or detection limit that is attainable with the referenced methodology.

[d] If two values are listed, the first is the recommended quantitation limit for soil and the second is the recommended quantitation limit for sediment.

[e] Sources of analytical methods, except as noted: CLP SOW (OLM01.5) = EPA (1991b) (note that for pesticide/PCB organic analysis of sediment samples, standing liquid should be discarded prior to extraction); EPA-600/4-82-057 = EPA (1988b) (Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector).

[f] Recommended alternate quantitation limit based on potential aquatic toxicity.

[g] The referenced value is an estimated detection limit (EDL), defined either as the method detection limit (MDL) or as a level of compound in a sample yielding a peak in the final extract with a signal-to-noise ratio of approximately 5, whichever is higher. The MDL is defined as the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero (40 CFR Part 136, Appendix B). Note that if a quantitated value is greater than the EDL or MDL values but less than the CRQL, the reported concentration would be considered estimated but available for use in risk assessment.

[h] The referenced value is an MDL. Note that if a quantitated value is greater than the EDL or MDL values but less than the CRQL, the reported concentration would be considered estimated but available for use in risk assessment.

[i] EPA (1988b) (Method 505, Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl Products in Water by Microextraction and Gas Chromatography; note that several hardware configurations exist for this method and that the referenced values are for the following configurations: wide bore capillary column, jet separator, and quadrapole mass spectrometer).

then backflushed onto a packed gas chromatographic column that is temperature programmed to separate the organic constituents. The volatile compounds are then detected with a mass spectrometer operating in the electron impact and full scan mode.

Occasionally, short turnaround times are required for results of analysis of samples from select J-Field AOCs. In such situations, elimination of the gel permeation chromatography (GPC) cleanup will result in expedited data delivery for the CLP analysis. GPC is both time- and labor-intensive and can cause significant delays. In the interest of timeliness, sample extracts will be prepared for analysis without the GPC cleanup. When sample extracts do not appear to be viscous or highly pigmented, no significant impacts on sample analysis are anticipated. While GPC is required under CLP methodology, and this is a deviation from the published method under special situations, absence of GPC cleanup will not affect data useability. Therefore, under such situations, performing GPC cleanup may not be required before analysis of environmental media such as soils for CLP semivolatile compounds, pesticides, and PCBs.

Explosives will be analyzed by HPLC with USATHAMA Methods UW14 (aqueous) and LW12 (solid) and/or as proposed by Bossle et al. (1988, 1989, 1990) (Tables 8.18 and 8.19). The methods involve solid-phase extraction of 500 mL of an environmental aqueous sample or 1 g of environmental solid sample with acetonitrile. The target analytes are separated by HPLC column with isocratic elution and are detected by ultraviolet (UV) absorbance at 230 nm.

GC-ECD will be used in the determination of chlorinated pesticides in water. USATHAMA Method UH14 will be used. A measured volume of the sample (800 mL) is adjusted to a pH of 13.0 with 6 N sodium hydroxide and shaken periodically for one hour to hydrolyze derivatives. Extraneous organic material is removed by a solvent wash. The sample is acidified, and the chlorinated compounds are extracted with methylene chloride by mechanical shaking in a separatory funnel. The acids are converted to their methyl esters with diazomethane as the derivating agent. Excess derivating reagent is removed, and the esters are determined by GC-ECD. HPLC will be used according to USATHAMA Methods LW03 and LH11 to analyze pesticides in solid samples.

Dimethylmethylphosphonate (DMMP) and diisopropylmethylphosphonate (DIMP) will be analyzed in aqueous and solid samples with GC-FPD by USATHAMA Methods T8 and TT9, respectively. A measured volume of sample or extract is directly injected onto the gas chromatographic column. Chromatographic conditions are described that permit the separation and measurement of DMMP and DIMP in "standard" or environmental aqueous or solid samples. Qualitative identification is performed via retention times, and quantitative analysis is performed via standard curves.

Isopropylmethylphosphonic acid and methylphosphonic acid will be analyzed by USATHAMA Methods UT02 (aqueous samples) and AAA9 (solid samples) with gradient ion chromatography. The results will be calculated against a calibration curve of the analytes prepared with deionization water. Samples containing particulate matter will be filtered before analysis.

TABLE 8.18 Quantitation Limits and Analytical Methods for Principal Degradation Products of Selected CWAs[a]

CWA	Associated Degradation Product	Quantitation Limits[b]		Analytical Methods[c]	
		Soil/Sediment (µg/g or mg/kg)	Water (µg/kg)	Soil/Sediment	Water
Mustard (HD)	Thiodiglycol	5,000	500	HPLC-EC/UV	GC-FPD
Mustard	1,4-Dithiane	2,500	5	GC-FPD	GC-FPD
Lewisite (L)	Chlorovinylarsine oxide	500	100	HPLC-EC/UV	HPLC-EV/UV
Lewisite	2-Chlorovinylarsonic acid	–[d]	1,000[e]	–	IC-UV[f]
Lewisite	2-Chlorovinylarsonous acid	1,000[e]	1,000[e]	HPLC-EC/UV[g]	HPLC-EC/UV[g]
Sarin (GB)	Isopropylmethylphosphonic acid (IMPA)	4,000[e]	200[e]	MPIC-EC/UV[h]	MPIC-EC/UV[h]
Sarin	Methylphosphonic acid (MPA)	–	200[e]	–	MPIC-EC/UV[h]
Sarin	Dimethylmethylphosphonate (DMMP)[i]	500	100	GC-FPD	GC-FPD
Sarin	Diisopropylmethylphosphonate (DIMP)[i]	500	100	GC-FPD	GC-FPD
VX	Methylphosphonic acid (MPA)	–	200[e]	–	MPIC-EC/UV[h]
VX	Ethylmethylphosphonic acid (EMPA)	4,000[e]	200[e]	MPIC-EC/UV[h]	MPIC-EC/UV[h]
Soman (GD)	Pinacolylmethylphosphonic acid	4,000[e]	200[e]	MPIC-EC/UV[h]	MPIC-EC/UV[h]
Soman	Methylphosphonic acid (MPA)	–	200[e]	–	MPIC-EC/UV[h]
Chloroacetophenone (CN)	Acetophenone	660[j]	210[k]	GC/MS[l]	GC/MS[l]
CS	Malononitrile	100[m]	100[m]	GC/MS[n]	GC/MS[n]
Adamsite (DM)	10,10'-oxybis(5,10-Dihydrophenarsine)	–	1,000	–	HPLC-EC/UV[l]

TABLE 8.18 (Cont.)

CWA	Associated Degradation Product	Quantitation Limits[b]		Analytical Methods[c]	
		Soil/Sediment (µg/g or mg/kg)	Water (µg/kg)	Soil/Sediment	Water
BZ	Benzilic acid	–	1,000	–	HPLC-EC/UV[n]
N,N'-Dichlorobis (2,4,6-trichlorophenyl)urea (CC2)[o]	bis(2,4,6-Trichlorophenyl)urea (TCPU)[p]	–	–	GC-FPD[q]	GC-FPD[q]
N,N'-Dichlorobis (2,4,6-trichlorophenyl)urea (CC2)[o]	2,4,6-Trichloroaniline[p]	–	–	GC-FPD[q]	GC-FPD[q]

[a] This table provides a comprehensive list of CWA degradation products for which quantitation limits and analytical methods are available. Only those compounds listed in Table 8.11 will be tested.

[b] Unless otherwise noted, the referenced values are equivalent to USATHAMA target reporting limits and associated with standard USATHAMA analytical protocols. CWA degradation products detectable by available analytical methods are listed. Many more CWA degradation products, as well as stabilizers that may be present and detectable, will be listed in data reports, depending on analytical method and level of detection.

[c] HPLC-EC/UV = high-pressure liquid chromatography with a pulsed electrochemical detector and confirmation by ultraviolet detector; GC-FPD = gas chromatography with flame photometric detection; IC-UV = ion chromatography with an ultraviolet detector; MPIC-EC/UV = mobile phase ion chromatography with combination ultraviolet and pulsed electrochemical detector; GC/MS = gas chromatography/mass spectrometry.

[d] Quantitation limits or analytical methods have not yet been identified.

[e] This value represents a demonstrated MDL.

[f] Bossle et al. (1990).

[g] Bossle et al. (1989).

[h] Bossle et al. (1988).

[i] DIMP is a by-product formed during the manufacture of sarin. DMMP may be a production by-product or a degradation product of sarin.

[j] Although the compound is listed under SW-846, Method 8270, a practical quantitation limit (PQL) has not been determined for soil. The referenced value is the PQL that is attainable for semivolatile compounds in soil by Method 8270A.

TABLE 8.18 (Cont.)

[k] This value is the PQL, which is comparable to the CLP CRQL.

[l] EPA (1986a) (Method 8270A; note that this method has not been promulgated and is still open to public comment).

[m] Although this analyte is on the 8240A TCL, the specific quantitation limits have not been determined. The PQL is indicative of compounds that have poor purging efficiencies, as discussed in Method 8204A.

[n] Kuronen (1986).

[o] This compound is not a CWA per se, but it is related to CWAs in that it is a clothing-impregnating compound used to protect against CWA exposure.

[p] TCPU and 2,4,6-trichloroaniline are intermediates in the production of CC2.

[q] These compounds are amenable to analysis by GC-FPD. The papers using this instrumentation have been requested and will be reviewed to establish method quantitation limits.

TABLE 8.19 Quantitation Limits and Analytical Methods for Selected Explosives and Related Compounds

Chemical	Quantitation Limits[a]		Analytical Method[b]	
	Soil/Sediment (µg/g or mg/kg)	Water (µg/L)	Soil/Sediment	Groundwater
Nitrobenzene	0.26	0.5	HPLC-UV	HPLC-UV
1,3-Dinitrobenzene	0.25	0.5	HPLC-UV	HPLC-UV
1,3,5-Trinitrobenzene	0.25	1	HPLC-UV	HPLC-UV
2,4,6-Trinitrotoluene (TNT)	0.25	1	HPLC-UV	HPLC-UV
2,4-Dinitrotoluene	0.25	0.007	HPLC-UV	HPLC-UV[c]
2,6-Dinitrotoluene	0.26	0.006	HPLC-UV	HPLC-UV[c]
Pentaerythritol tetranitrate (PETN)	0.50	2	HPLC-UV	HPLC-UV
Nitroglycerin	0.50	1.5	HPLC-UV	HPLC-UV
Cyclotetramethylene tetranitramine (HMX)	2.2	1	HPLC-UV	HPLC-UV
Cyclotrimethylene trinitramine (RDX)	1.0	1	HPLC-UV	HPLC-UV
n-Methyl-*n*-2,4,6-tetranitroaniline (TETRYL)	0.65	5	HPLC-UV	HPLC-UV
Picric acid	2.9	-[d]	HPLC-UV	-[d]
Tetrazene	1.3	7.3	HPLC-UV	HPLC-UV
2,4,6-Trinitrobenzaldehyde	1	1	HPLC-UV	HPLC-UV

[a] Values listed are equivalent to USATHAMA target reporting limits.

[b] Referenced methods obtained from AEC (formerly USATHAMA) Technical Support Division, APG, Maryland. All methods are USATHAMA standardized methods for military-unique compounds.

[c] USATHAMA-certified low-level method.

[d] Analytical method has not yet been identified.

Organopesticides and PCBs/Aroclors will be analyzed with GC-ECD (Table 8.17). The USATHAMA methods to be employed are UH02 for aqueous samples and LN05, LH16, and LN10 for solid samples.

Organosulfur compounds will be analyzed in aqueous and solid samples by GC via USATHAMA Methods UL04 (aqueous) and LL03 (solid). The methods employ extraction of the water matrix with methylene chloride, solvent concentration via standard Kuderna-Danish techniques, and analysis by GC-FPD in the sulfur mode.

Thiodiglycol will be analyzed in aqueous and solid samples via HPLC by USATHAMA Methods UW22 and LW18, respectively. The environmental aqueous and solid samples are extracted as follows:

- Aqueous samples — A measured volume of the sample is concentrated by boiling and is passed through an Amberlight XAD-7 resin column and further concentrated by another boiling step. The extract is buffered and brought to volume with water.

- Solid samples — A measured weight of the sample is extracted with alkaline methanol on a wrist-action shaker. A portion of the methanol is filtered and removed by evaporation under a nitrogen stream. The extract is acidified and buffered and brought to volume with water.

Liquid chromatographic conditions achieved by these methods permit the separation and measurement of the thiodiglycol in the extract from the environmental aqueous and solid samples. Analytes are identified on the basis of retention times, and quantitative analysis is performed with a standard curve of area counts.

8.3.2.3 Air Monitoring Analytical Methods

Methods and techniques for analysis of VOCs, SVOCs, and CWA degradation products in vapor collection filters and for analysis of inorganic compounds (TAL metals and cyanide), SVOCs, and CWA degradation products in particulate filters are currently being researched.

8.3.2.4 CLP Contract-Required Quantitation and Detection Limits

The CLP has established quantitation limits for organic analyses, as set forth in the SOW, equivalent to the concentration of the lowest calibration standard analyzed for each analyte. The quantitation limit differs from the detection limit in that the amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to simply be detected above the background noise. The contract-required detection limit (CRDL) is the minimum level of detection acceptable under the contract SOW. The CRDLs for inorganic compounds are presented in Table 8.20. The contract-required quantitation limit (CRQL) is the minimum level of detection acceptable under the method and contract SOW. The CRQLs for VOCs, SVOCs, and pesticides/PCBs are presented in Tables 8.21 through 8.23. The specific quantitation and detection limits, provided in Tables 8.14 through 8.19 for inorganic chemicals, VOCs, SVOCs, pesticides/PCBs, and selected CWAs and explosives, are highly matrix-dependent and thus may not always be achievable.

8.3.2.5 USATHAMA-Certified and Upper Reporting Limits

The lowest concentration that is reported for any analyte has been established in the USATHAMA (now AEC) program from a statistical analysis of spikes and blanks. That concentration, termed the certified reporting limit (CRL), is the lowest value that can be reported within a 90% confidence limit. The upper reporting limit (URL) for the certified range was developed during the method certification. Tables 8.24 and 8.25 present the USATHAMA reporting limits for aqueous and solid samples of explosives, CWA degradation products, and total phosphorus.

TABLE 8.20 CLP Contract-Required Detection Limits for TAL Inorganic Compounds

Analyte	USATHAMA Acronym	CRDLs[a] Solid (µg/g or mg/kg)	CRDLs[a] Aqueous (µg/L)
Aluminum	AL	40	200
Antimony	SB	12	60
Arsenic	AS	2	10
Barium	BA	40	200
Beryllium	BE	1	5
Cadmium	CD	1	5
Calcium	CA	1,000	5,000
Chromium	CR	2	10
Cobalt	CO	10	50
Copper	CU	5	25
Cyanide	CN	2	10
Iron	FE	20	100
Lead	PB	0.6	3
Magnesium	MG	1,000	5,000
Manganese	MN	3	15
Mercury	HG	0.1	0.2
Nickel	NI	8	40
Potassium	K	1,000	5,000
Selenium	SE	1	5
Silver	AG	2	10
Sodium	NA	1,000	5,000
Thallium	TL	2	10
Vanadium	V	10	50
Zinc	ZN	4	20

[a] The CRDLs originated from the CLP SOW (ILM 02.0) and can be obtained using ICP, except for GFAA for AS, SE, TL, and PB; CVAA for HG; and spectroscopy for CN.

TABLE 8.21 CLP Contract-Required Quantitation Limits for TCL Volatile Organic Compounds

Analyte	USATHAMA Acronym	CRQLs Solid (µg/kg)[a]	CRQLs Aqueous (µg/L)
Methylene chloride	CH2CL2	10.0	10.0
1,1-Dichloroethane	11DCLE	10.0	10.0
trans-1,2-Dichloroethylene	12DCE	10.0	10.0
1,1-Dichloroethane	11DCE	10.0	10.0
Chloroform	CHCL3	10.0	10.0
1,2-Dichloroethane	12DCLE	10.0	10.0
1,1,1-Trichloroethane	111TCE	10.0	10.0
Carbon tetrachloride	CCL4	10.0	10.0
Trichloroethylene	TRCLE	10.0	10.0
Benzene	C6H6	10.0	10.0
1,1,2-Trichloroethane	112TCE	10.0	10.0
Tetrachloroethylene	TCLEE	10.0	10.0
Toluene	MEC6H5	10.0	10.0
Chlorobenzene	CLC6H5	10.0	10.0
Ethylbenzene	ETC6H5	10.0	10.0
1,2-Dichloropropane	12DCLP	10.0	10.0
cis-1,3-Dichloropropylene	C13DCP	10.0	10.0
Vinyl chloride	C2H3CL	10.0	10.0
Chloroethane	C2H5CL	10.0	10.0
Chloromethane	CH3CL	10.0	10.0
Bromoform	CHBR3	10.0	10.0
Dibromochloromethane	DBRCLM	10.0	10.0
trans-1,3-Dichloropropene	T13DCP	10.0	10.0
1,1,2,2-Tetrachloroethane	TCLEA	10.0	10.0
Bromodichloromethane	BRDCLM	10.0	10.0
Bromomethane	CH3BR	10.0	10.0
Acetone	ACET	10.0	10.0
Carbon disulfide	CS2	10.0	10.0
2-Butanone	MEK	10.0	10.0
4-Methyl-2-pentanone	MIBK	10.0	10.0
Styrene	STYR	10.0	10.0
Xylene	XYLEN	10.0	10.0

[a] Quantitation limits listed for solids are based on wet weight. The quantitation limits calculated by the laboratory (calculated on a dry-weight basis as required by contract) will be higher.

TABLE 8.22 CLP Contract-Required Quantitation Limits for TCL Semivolatile Organic Compounds

Analyte	USATHAMA Acronym	CRQLs Solid[a] (µg/kg)	CRQLs Aqueous (µg/L)
Phenol	PHENOL	330	10
bis(2-Chloroethyl)ether	B2CLEE	330	10
2-Chlorophenol	2CLP	330	10
1,3-Dichlorobenzene	13DCLB	330	10
1,4-Dichlorobenzene	14DCLB	330	10
1,2-Dichlorobenzene	12DCLB	330	10
2-Methylphenol	2MP	330	10
bis(2-Chloroisopropyl)ether	B2CIPE	330	10
4-Methylphenol	4MP	330	10
N-Nitroso-di-*n*-propylamine	NNDNPA	330	10
Hexachloroethane	CL6ET	330	10
Nitrobenzene	NB	330	10
Isophorone	ISOPHR	330	10
2-Nitrophenol	2NP	330	10
2,4-Dimethylphenol	24DMPN	330	10
bis(2-Chloroethoxy)methane	B2CEXM	330	10
2,4-Dichlorophenol	24DCLP	330	10
1,2,4-Trichlorobenzene	124TCB	330	10
Naphthalene	NAP	330	10
4-Chloroaniline	4CANIL	330	10
Hexachlorobutadiene	HCBD	330	10
4-Chloro-3-methylphenol	4CL3C	330	10
2-Methylnaphthalene	2MNAP	330	10
Hexachlorocyclopentadiene	CL6CP	330	10
2,4,6-Trichlorophenol	246TCP	330	10
2,4,5-Trichlorophenol	245TCP	800	25
2-Chloronaphthalene	2CNAP	330	10
2-Nitroaniline	2ANIL	800	25
Dimethylphthalate	DMP	330	10
Acenaphthylene	ANAPYL	330	10
2,6-Dinitrotoluene	26DNT	330	10
3-Nitroaniline	3NANIL	800	25
Acenaphthene	ANAPNE	330	10
2,4-Dinitrophenol	24DNP	800	25
4-Nitrophenol	4NP	800	25
Dibenzofuran	FURANS	330	10
2,4-Dinitrotoluene	24DNT	330	10
Diethylphthalate	DEP	330	10
4-Chlorophenyl phenyl ether	4CLPPE	330	10
Fluorene	FLRENE	330	10
4-Nitroaniline	4NANIL	800	10
4,6-Dinitro-2-methylphenol	46DN2C	800	10
N-Nitrosodiphenylamine	NNDPA	330	10
4-Bromophenyl phenyl ether	4BRPPE	330	10

TABLE 8.22 (Cont.)

Analyte	USATHAMA Acronym	CRQLs Solid[a] (µg/kg)	CRQLs Aqueous (µg/L)
Hexachlorobenzene	CL6BZ	330	10
Pentachlorophenol	PCP	800	10
Phenanthrene	PHANTR330	330	10
Anthracene	ANTRC	330	10
Di-*n*-butylphthalate	DNBP	330	10
Fluoranthene	FANT	330	10
Pyrene	PYR	330	10
Butylbenzylphthalate	BBZP	330	10
3,3'-Dichlorobenzidine	33DCBD	330	10
Benz[*a*]anthracene	BAANTR	330	10
Chrysene	CHRY	330	10
bis(2-Ethylhexyl)phthalate	B2EHP	330	10
Di-*n*-octylphthalate	DNOP	330	10
Benzo[*b*]fluoranthene	BBFAN	330	10
Benzo[*k*]fluoranthene	BKFANT	330	10
Benzo[*a*]pyrene	BAPYR	330	10
Indeno[*1,2,3-c,d*]pyrene	ICDPR	330	10
Dibenz[*a,h*]anthracene	DBAHA	330	10
Benzo[*g,h,i*]perylene	BGHIPY	330	10

[a] Quantitation limits for solids are based on wet weight. The quantitation limits calculated by the laboratory (calculated on a dry-weight basis as required by the contract) will be higher.

8.3.3 Reference Materials

Reference standards are needed to generate certification data, calibrate instruments, spike analytical surrogates or standards, and prepare QC samples. These solutions must be of known concentration and purity to meet the validation criteria of analytical procedures. Standards will be either standard analytical reference materials (SARMs) or interim reference materials (IRMs). SARMs that are developed and distributed by the Central QA Laboratory of the NIST will be the preferred standards. IRMs are not as rigorously characterized as SARMs.

Reference materials for metal analyses may be stored at room temperature in a locked storage area. Materials for organic analyses must be stored in a locked refrigerator at or below 4°C.

Table 8.26 presents a tentative list of J-Field environmental samples, by media and AOC, that will be subject to analyses in an off-site laboratory.

TABLE 8.23 CLP Contract-Required Quantitation Limits for Pesticides/Aroclors

Analyte	USATHAMA Acronym	CRQLs Solid[a] (µg/kg)	CRQLs Aqueous (µg/L)
Aroclor-1016	PCB016	33.0	1.0
Aroclor-1221	PCB021	33.0	2.0
Aroclor-1232	PCB232	33.0	1.0
Aroclor-1242	PCB242	33.0	1.0
Aroclor-1248	PCB248	33.0	1.0
Aroclor-1254	PCB254	33.0	1.0
Aroclor-1260	PCB260	33.0	1.0
alpha-BHC	ABHC	1.7	0.05
beta-BHC	BBHC	1.7	0.05
delta-BHC	DBHC	1.7	0.05
gamma-BHC (Lindane)	LIN	1.7	0.05
Heptachlor	HPLC	1.7	0.05
Aldrin	ALDRN	1.7	0.05
Heptachlor epoxide	HPCLE	1.7	0.05
Endosulfan I	AENSLF	1.7	0.05
Dieldrin	DLDRN	3.3	0.10
4,4´-DDE	PPDDE	3.3	0.10
Endrin	ENDRN	3.3	0.10
Endosulfan II	BENSLF	3.3	0.10
4,4´-DDD	PPDDD	3.3	0.10
Endosulfan sulfate	ESFSO4	3.3	0.10
4,4´-DDT	PDDDT	3.3	0.10
Endrin ketone	ENDRNK	3.3	0.10
Methoxychlor	MEXCLR	17.0	0.50
Endrin aldehyde	ENDRNA	3.3	0.10
alpha-Chlordane	ACLDAN	33.0	0.05
gamma-Chlordane	GCLDAN	67.0	0.05
Toxaphene	TXPHEN	33.0	5.0

[a] Quantitation limits for solids are based on wet weight. The quantitation limits calculated by the laboratory (calculated on a dry-weight basis as required by the contract) will be higher.

TABLE 8.24 USATHAMA Reporting Limits for Explosives

Analyte	USATHAMA Acronym	Solid (µg/g)		Aqueous (µg/L)	
		Certified	Upper	Certified	Upper
Nitrobenzene	NB	2.41	27.4	1.07	54.9
1,3-Dinitrobenzene	13DNB	0.496	24.8	0.519	40.1
1,3,5-Trinitrobenzene	135TNB	0.448	24.4	0.626	42.1
2,4,6-Trinitrotoluene	246TNT	0.456	22.8	0.588	40.2
2,4-Dinitrotoluene	24DNT	0.424	21.2	0.612	40.2
2,6-Dinitrotoluene	26DNT	0.524	26.2	1.15	52.4
Nitroglycerin	NG	4.00	200	NA[a]	NA
Pentaerythritol	PETN	4.00	80	NA	NA
Cyclotetramethylene tetranitrate	HMX	0.666	33.3	1.65	28.9
Cyclotrimethylene trinitramine	RDX	0.587	21.9	2.11	43.9
n-Methyl-*n*-2,4,6-tetranitroaniline	TETRYL	0.731	20.2	0.556	44.5

[a] NA = not applicable; the analyte is not certified in the aqueous analysis of explosives.

TABLE 8.25 USATHAMA Reporting Limits for CWA Degradation Products and Total Phosphorus

Analyte	USATHAMA Acronym	Solid (µg/g)		Aqueous (µg/L)	
		Certified	Upper	Certified	Upper
1,4-Dithiane	DITH	1.47	11.3	1.11	22.2
1,4-Oxathiane	OXAT	0.856	17.1	1.98	39.5
p-Chlorophenylmethyl sulfone	CPMSO2	2.37	47.4	4.72	106
p-Chlorophenylmethyl sulfide	CPMS	1.08	21.6	1.26	25.3
p-Chlorophenylmethyl sulfoxide	CPMSO	2.25	45.0	4.23	106
Benzathiazole	BTZ	1.08	13.2	2.11	42.2
Dimethyldisulfide	DMDS	0.692	13.8	1.14	22.8
Diisopropyl methylphosphonate	DIMP	0.114	4.57	10.5	210
Dimethyl methylphosphonate	DMMP	0.133	4.18	15.2	305
Methylphosphonic acid	MPA	2.0	10	128	9,000
Isopropylmethylphosphonic acid	IMPA	2.1	40	100	9,000
Thiodiglycol	TDGCL	3.94	102	187	4,880
Total phosphorus	TPO4	7.49	100	13.3	500

TABLE 8.26 Numbers of Stage I Samples Required, by Media and AOC

AOC	Number of Samples by Media					
	Ground-water[a]	Surface water[a]	Surface Soil	Soil Boring	Surface Sediment	Sediment Boring[b]
TBP	21	9	53(+)[c]	80[d]	11	35
WPP	14	5	36(+)	32[e]	5	0
RCP	6	7	9	12[f]	0	5
PB	1	2	6	6[g]	0	0
SBDG	1	5	0	0	2	0
SBT	1	0	4	0	0	0
RPDG	2	7	6	0	3	0
RPTS	1	3	8(+)	0	3	0
Other	1	1	0	0	1	0
Opportunity available[h]	0	5	0	0	0	0
Total	48	44	122(+)	130	25	40

[a] Groundwater and surface water samples will be analyzed for the CLPAS.

[b] Number assumes five depth intervals sampled per sediment boring.

[c] The "+" refers to an additional but unknown number of samples to be collected.

[d] Number assumes six depth intervals sampled per boring in the Toxic, VX, and Mustard Pits, and four per boring for the eight other locations.

[e] Number assumes four depth intervals sampled per boring.

[f] Number assumes three depth intervals sampled per boring.

[g] If boring is drilled, it is assumed that six depth intervals will be sampled.

[h] Surface water that accumulates in the TBP main pits, WPP principal pits, and/or the RCP may be sampled on an opportunity-available basis.

8.3.4 Radiochemical Testing

A variety of samples, including soil, sediment, and air, will be sampled to test for strontium-90 and cesium-137. The sampling and analysis procedures to be followed include SOP-S004 (Appendix A), as well as EPA radiochemical analytical methods (EPA 1979, 1980b).

9 DATA VALIDATION, REDUCTION, AND REPORTING

Data will initially be collected, converted to standard reporting units (e.g., µg/g or mg/kg for solid media and µg/L for aqueous media), and recorded in standard formats by the project analysts. These analysts will then use a variety of methods and procedures to conduct preliminary data analyses. Because many analytical instruments that will be used are microprocessor controlled, some of the requisite analyses can be performed directly in the instrument's operating or output mode. When the instruments can be linked to stand-alone computers or microprocessors, it is often possible to write (or modify) data analysis programs that produce customized data formats. Data requiring manual recording, integration, and/or analysis can be converted to a more appropriate format before subsequent analyses. Through all stages of processing, the data will be double-checked for translation or transcription errors and initialed by both the recorder and the verifier. The QA officer or other designated individual not directly involved in the analysis will review the data for acceptability.

9.1 VALIDATION

Data validation is the process whereby an evaluation is made to determine the limitations, if any, of the data when applied to the endpoint use (e.g., regulatory, toxicological). The criteria for the data depend on the referenced sampling and analytical methodologies and include the associated QA/QC requirements. The guidelines to be used for validation of J-Field data are given in *Laboratory Data Validation; Functional Guidelines for Evaluating Organic Analyses* (EPA 1985) and *Laboratory Data Validation; Functional Guidelines for Evaluating Inorganic Analyses* (EPA 1988c), with modifications stipulated by EPA Region III.

The data packages to be used will be independently reviewed to ensure compliance with specified analytical, QA, and data reduction procedures; data-reporting requirements; and required accuracy, precision, and completeness measures. The following items may be reviewed to validate the data:

- Sample holding times;
- Documentation that the analytical results are in control and within the certified (linear) range of the analysis;
- Qualitative and quantitative data used in determining the presence and concentration of the target compounds;
- Calibration data associated with specific methods and instruments;
- Routine instrument checks (calibration, control samples, etc.);
- Documentation on traceability of instrument standards, samples, and data;

- Documentation on analytical methodology and QC methodology;
- The potential presence of interferences in analytical methods (check of reference blanks and spike recoveries);
- Documentation of routine maintenance activity to ensure analytical reliability; and
- Documentation of sample preservation and transport.

All data generated will be assessed for accuracy, precision, and completeness. Data assessment techniques will include routine QC checks and system audits.

Precision will be assessed from measurements of replicates of the same measurement at different times. Control charts will be maintained to provide a timely assessment of precision for measurement functions. Accuracy will be assessed from measurements of samples spiked with known concentrations of reference materials. The assessment for accuracy will be independent of the routine calibration process (reference materials will be obtained from independent sources and will be prepared independently).

9.2 REDUCTION

Data reduction frequently includes computation of analytical results from raw instrument data and summary statistics, including standard errors, confidence intervals, test of hypothesis relative to the parameters, and model validation. Procedures that the laboratory will use address the reliability of computations and the overall accuracy of the data reduction. The numerical transformation algorithms used for data reduction will be verified against a known problem set to ensure that the reduction methods are correct.

The equations and the typical calculation sequence that should be followed to reduce the data to the acceptable format is instrument- and method-specific. When standard methods are modified, data reduction techniques will be described in a report accompanying the data.

Auxiliary data produced for internal records and not reported as part of the analytical data will include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standard records, maintenance records, calibration records, and associated quality control. These sources will document data reduction and will be available for inspection during audits and for use in determining the validity of the data.

Outliers will be identified by the AEC or CLP control chart programs, and the rationale used for data acceptance or rejection will be described and documented.

9.3 REPORTING

All quantitative laboratory chemical data (EPA analytical levels III, IV, and V) and associated laboratory and field blank data will be reported in the Installation Restoration Data Management System (IRDMS), as described in the *USATHAMA QA Program* (USATHAMA 1990). The AEC will make available the IRDMS software PC tool, or any other software that will assist in the entering, editing, and error-checking of data. Microcomputer hardware and software requirements for the OSL and the off-site contractor laboratories are detailed in Section 6 (Records Management) of the FSP (Volume 1 of the SAP).

The analyst will quantify each analyte in the method blank and spiked QC sample each day of analysis. Method blank data will generally be reported as "less than" the CRDL for each analyte. Values detected above the CRDL will be reported as determined, with entry into the AEC data management system in terms of concentration. Values below the CRDL will be quantified and flagged as estimated values. Additional sample lots will not be processed until the results of the previous lot have been calculated and plotted on control charts as required and the entire analytical method is shown to be under control. All data will be entered into the IRDMS with correct method numbers and appropriate data qualifiers. The data reported in the IRDMS for each analyte include sample date, test method number, Boolean,[1] concentration, units, and sample depth (where applicable).

Geotechnical and map data will also be reported in the IRDMS. Procedures for data input are given in *USATHAMA QA Program* (USATHAMA 1990) and *Geotechnical Requirements for Drilling, Monitor Wells, Data Acquisition, and Reports* (USATHAMA 1987).

Login/logout procedures for the IRDMS are given in SOP-006 (Use of the IRDMS Network) (COE 1993). All qualitative data generated in the field will be entered into a separate database created and maintained by ANL.

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[1] "Boolean" refers to information that must accompany data entered into the IRDMS database that characterizes the data in terms of "greater than," "less than," "none detected," or "no measurement taken."

10 QUALITY CONTROL CHECKS

10.1 INTRODUCTION

During environmental media sampling and analysis, QC checks will be conducted as stipulated in a set of protocols established for the QC program. The QC program will involve verification of field and laboratory activities through a set of controls consisting of blank samples. The number and types of blanks prepared will depend on the environmental media being sampled, the proposed analytical method, and the parameter being analyzed. Quality control procedures will be established for field sampling and for laboratory analysis.

During field sampling, QC will include the generation of: (1) field blanks and equipment rinsate blanks to detect false contamination resulting from improper sampling procedures; (2) background samples, including soil samples from areas known to be uncontaminated and laboratory air blanks during soil-gas sampling; (3) water blanks consisting of deionized water and organic-free water that is used in equipment cleaning and decontamination procedures; and (4) trip blanks consisting of organic-free water supplied by prime and referee laboratories and used routinely for sample container preparation and laboratory analytical equipment decontamination. Proposed QC samples to be collected during RI field sampling activities are listed in Table 10.1. Field sampling procedures for QC samples are discussed in Section 5.2.3.

The analytical laboratories will follow the established QC program described in their QA/QC program manuals. Table 10.2 summarizes the proposed QC samples to be collected during laboratory investigations. The laboratory QC program will include, at a minimum, (1) reagent and/or method blanks to determine if any contamination is being caused by extraction procedures or general laboratory practices, (2) surrogate blanks to confirm recoveries of surrogate compounds and spiked samples for spiked compound recoveries, and (3) duplicate samples to establish RPD.

10.2 DOCUMENT CONTROL

Document control procedures will be practiced as described in the prime laboratory QA/QC manual. Field operations procedures will be archived as described in SOP-015 (Document Control System) presented in COE (1993) and in Section 6 of the FSP.

10.3 BLANKS

Data quality requirements mandate that a percentage of samples shipped to the prime and referee laboratories must consist of samples known to contain no measurable concentrations of the parameters for which the sample is being analyzed. These samples will

TABLE 10.1 Summary of Proposed Quality Control Samples To Be Collected during Field Sampling Activities

Sample Description	Purpose	Frequency Percent Range (%, except as noted) Minimum	Maximum
Equipment rinsate blank	Detect false contamination during field sampling	10	20
Background soil/sediment	Determine soil/sediment concentration in uncontaminated zone	10	20
Background laboratory air	Determine air concentration in uncontaminated zone	10	20
Deionized water	Verify background metals concentration	10	20
Filter blank (per lot)	Detect false contamination during filtration for dissolved or total metals	1[a]	1[a]
Organic-free water	Verify background organic compounds concentration	10	20
Trip blank (per shipment)	Verify container preparation and volatile organic compounds contamination during transit	1[b]	1[b]
Matrix spike	Confirmatory	10	20
Matrix spike duplicate	Confirmatory	10	
Duplicate	Evidentiary ANL internal QC	10	20

[a] Not a percent, one filter per lot.

[b] Not a percent, one container per shipment.

TABLE 10.2 Summary of Proposed Quality Control Samples To Be Generated during Laboratory Investigations

Sample Description	Purpose	Frequency Percent Range (%) Minimum	Maximum
Reagent blank/method blank	Verify extraction procedure and/or laboratory practices	10	20
Method blank	Verify method validity	10	20
Surrogate blank	Surrogate compounds	10	10
Spiked blank	Laboratory control for percent recovery	10	10
Duplicate/split samples	Relative percent difference	10	10

be used as QC checks on container preparation procedures and the laboratory methodology. Specific percentages of blanks for each category are listed in Tables 10.1 and 10.2 for field and laboratory activities, respectively.

10.3.1 Field Blanks

Sampling devices will be cleaned between sampling points during sampling of environmental media to ensure that the samples are not contaminated by the equipment. Rinsates (equipment blanks) of deionized water (for samples collected for metals analysis) and of organic-free water (for samples collected for organic constituents) will be collected and composited to determine whether the sampling equipment is completely free of contamination. Each rinsate will be analyzed by methods proposed for parameters being tested. The resulting data will indicate if any contamination is resulting from improper sampling procedures or inadequate equipment decontamination.

In addition to the rinsate (equipment blank), at least one field blank will be collected from every 10 samples, that is, from 10% of the samples collected. Field blanks of soils or air will consist of soil or air collected from clean areas of the site, background soils, or air collected upwind from the sampling site. Field blanks generated during water sampling will contain deionized and/or organic-free water used for decontamination and may contain water collected from high-gradient locations away from any known source of contamination. Table 10.1 lists the proposed QC samples for field operations.

10.3.2 Laboratory Blanks

Laboratory QC samples will include reagent/method blanks, spike samples, split samples, internal standards, and QC samples for surrogate recoveries. The proposed QC samples, including percentage of blanks of each category proposed during J-Field RI laboratory analyses, are summarized in Table 10.2.

10.4 LABORATORY QUALITY CONTROL CHECKS

Laboratory QC checks will include application of internal QC methods, such as analysis of spike samples, split samples, internal standards, QC samples, calibration standards, and calibration devices (Table 10.2). Quality control checks include demonstration of daily standards, system performance checks, multiple internal standards for sample analysis, and method blanks for control of system contamination. The frequency, control limits, corrective actions, and purpose of quality control checks for the prime laboratory are largely implicit in the methods used. The prime laboratory and referee laboratory will submit results of their most recent EPA-sponsored performance evaluation samples.

10.5 CONTROL CHARTS

Control charts will be used to monitor the trends and variations in the accuracy and precision of analyses. The control chart shall contain the following information:

- Title, analyte, method number, and laboratory name;
- Spike concentration;
- Analysis date and/or code;
- Percent recovery (X charts) or range (R charts) along the ordinate;
- Upper and lower control limits; and
- Upper and lower warning limits.

10.6 OUT-OF-CONTROL CONDITIONS

All out-of-control conditions for all project aspects will be investigated, and appropriate corrective actions will be promptly instituted. Areas in which operator error is normally associated with out-of-control conditions include (1) failure to achieve calibration, (2) record-keeping omissions, (3) improper sample storage and preservation, and (4) poor analytical protocols. The detection of out-of-control conditions always warrants some type of corrective action. Section 15 of this plan provides protocols for documenting corrective action.

11 PERFORMANCE AND SYSTEM AUDITS

The ANL project QAO or designee will conduct at least one performance and system audit during field sampling and laboratory testing activities. The field sampling system and the on-site, prime, and referee laboratories will all be audited. The audits are intended to ensure that the proper procedures are used for field sampling and laboratory analysis and to assess data precision, accuracy, and completeness. Audit protocols will be based on the QC procedures outlined in SOPs for each of the field operations or laboratory procedures. In addition to these procedures, EPA and USATHAMA protocols and procedures will be followed in the audits (EPA 1980b, 1984a; USATHAMA 1990). Depending on the results of the audit, the project QAO will issue a nonconformance notice, formulate and recommend a corrective action acceptance report, prepare an audit report, and coordinate with contract laboratory QAOs.

11.1 FIELD SYSTEM AUDITS

The ANL project QAO or designee will conduct at least one performance and system audit during field sampling. A system audit checklist for field operations is shown in Appendix B.

11.2 LABORATORY SYSTEM AUDITS

The ANL project QAO or designee will conduct at least one system audit of on-site laboratory analyses and of prime and referee laboratory analyses of environmental samples. A laboratory system audit checklist contains system-, performance-, and method-specific components.

11.3 PERFORMANCE AUDITS

The ANL project QAO or designee will conduct at least one performance audit during on-site, prime, and referee laboratory analyses of environmental samples. In addition, the contract and referee laboratories participate in external performance and system audits sponsored by state and federal regulatory agencies. The QAO of each laboratory will supply reports of such audits.

Any modifications of field or laboratory procedures that result from system and performance audits must be approved by the project manager and the QAO and must be documented in writing. Copies of all system and performance audit reports will be maintained in the project files.

12 PREVENTIVE MAINTENANCE

12.1 GENERAL

For the J-Field RI activities, preventive maintenance will have three principal objectives: ensure accuracy of measurement systems, minimize downtime, and maintain adequate critical spare parts, backup systems, and equipment. Preventive maintenance procedures outlined in individual SOPs will be followed.

12.2 CALIBRATION AND MAINTENANCE SCHEDULES

Calibration and maintenance schedules will be established in accordance with manufacturer recommendations in instrument operation manuals and with SOPs, presented in COE (1993) and Appendix A.

12.2.1 Field Equipment

Field equipment calibration, maintenance frequency, and calibration standards are described in SOPs in COE (1993) and Appendix A. Analytical accuracy of all calibration gases is traceable to SARMs from the NIST.

12.2.2 Laboratory Equipment

Laboratory equipment calibration, maintenance frequency, and calibration standards, including lists of spare parts for laboratory equipment, are specified in the prime and referee laboratories' QAPPs and/or SOPs. It is the understanding that the laboratories' analytical accuracy of all calibration gases is traceable to SARMs from NIST.

13 RECORD KEEPING

13.1 GENERAL

Bound logbooks will be used for all record keeping, both in the field and in the laboratory. Requirements for recording of sampling and field investigation data are presented in SOP-003 (Field Logbook) and SOP-016 (Surface Water, Groundwater, and Soil/Sediment Logbooks) (COE 1993). The bound books will provide a chronological sequence of data insertion. All logbooks will contain a unique document control number. If ANL-controlled logbooks are used, the document control number will be on all pages. Non-ANL controlled logbooks will be bound, and the document control number need only be on the document cover. At a minimum, all pages with information recorded on them will be numbered (blank pages may also be numbered if desired).

To facilitate data validation, a person making an entry in a logbook must sign and date the entry. All entries must be recorded in indelible ink. Corrections to entries will be made by drawing a line through the incorrect entry, recording the correct information, and initialing and dating the corrected entry. If computerized information is used, a hard copy that has been permanently affixed to the logbook will be acceptable as an original record of sampling and laboratory logging.

Logbooks containing information specific to the project will be archived in ANL's Document Control Center. Should the need arise to provide the AEC with information from ANL-controlled logbooks, copies of all relevant logbook pages will be submitted.

13.2 SAMPLING RECORDS

Logbooks for sampling and field investigation activities must meet the requirements specified in SOP-003 (Field Logbook) and SOP-016 (Surface Water, Groundwater, and Soils/Sediment Logbooks) (COE 1993). The books must be bound and entries recorded with waterproof ink. The logbook must contain sufficient information to distinguish samples from each other. The following information should be included for each sample collected: (1) AEC project, (2) sequential field sample number, (3) matrix sampled, (4) sample depth, (5) sampling date and time, (6) specific sampling location, (7) method of sampling, (8) preservation techniques, (9) filtration method, (10) analytes of interest, (11) volume of water removed during well development, (12) sampling observations, (13) results of field measurements, (14) printed names and signatures of samplers, (15) date of shipment, (16) number of shipping containers, and (17) samples sent and name of carrier.

Overnight delivery air bills will also be retained as part of the COC documentation. The white (original) and yellow copies will be given to the laboratory sample custodian when samples are submitted to the laboratory. The pink copy will be retained by the field operations leader. The white copy (original) will be transmitted to the project data coordinator (PDC) after the laboratory has received the samples. The PDC will retain the

original with the project files and submit the information as a deliverable to the AEC at the completion of the project.

Field parameter forms bound into permanent logbooks will be used to record field information required for input into the IRDMS. Copies of the applicable forms and procedures are contained in SOP-016 (COE 1993).

13.3 LABORATORY RECORDS

13.3.1 Laboratory Logging

Once samples have been received by a laboratory, they will be logged into a bound laboratory notebook. The following information should be included for each sample: (1) field sample number, (2) laboratory receipt date, (3) condition in which sample arrived, (4) analysis requested, and (5) sample identification number.

13.3.2 AEC Sample Identification

The system used for reporting of data to the IRDMS requires that each aliquot of a sample be assigned a six-character identification number. The number consists of two three-letter character designations. The first three characters define the analytical lot (the size of which is determined by the number of samples that can be processed in a 24-hour period). The last three characters pertain to the sequential order in which the instrumental analysis will be performed within the lot. Different lot designations are used for each analytical method. Multianalyte methods have the same lot designation for each analyte in a single sample aliquot. If the prime laboratory uses an internal numbering system, the correlation to the ANL-assigned sample identification number must be provided in the laboratory logbook.

Requirements for reporting data to the IRDMS are outlined in SOP-006 (Use of the IRDMS Network) listed in COE (1993). The AEC sample identification numbering system is discussed in Section 4 of the FSP.

13.3.3 Analytical Records

13.3.3.1 Reference Materials

The following information on all reference materials used for analytical purposes must be recorded in bound logbooks: (1) date of receipt, (2) source, (3) purity, (4) composition, (5) storage conditions, and (6) expiration date.

13.3.3.2 Sample Handling

All personnel involved in performing any aspect of the analytical protocol must maintain a record of those activities in a bound logbook. Although this logbook must be specific to the operation, it need not be operator-specific. The logbook should be signed and dated daily and must contain the following information: (1) samples handled, (2) standards used, (3) QC samples prepared, (4) procedures used, and (5) resulting calculations.

13.3.3.3 Instrument Operation

Each instrument must have a dedicated logbook. Information in the logbook must reflect routine and emergency maintenance activities, tuning, absolute and chemical curve calibration, and all analytical activities conducted with the instrument. A new page must be started each day during equipment operation, and the following information must be included on each page: (1) date, operator, and project name; (2) description of any instrument maintenance or modification; (3) tuning and calibration activities; (4) instrument settings; (5) instrument operating condition; and (6) samples analyzed.

The use of automated data acquisition systems will require that a reference to the data file be recorded for each standard or sample. Hard copy data output from integrators and chromatograms should have the following information clearly evident on the printout: (1) analysis date and time, (2) test name and sample number, (3) reference to the calibration curve used for quantitation, (4) logbook reference to recorded analytical activities, and (5) identification of chromatographic peaks.

14 DATA ASSESSMENT PROCEDURES

Precision, accuracy, and completeness are data quality parameters that allow a reviewer to evaluate the consistency of the measurement data. Analytical precision and accuracy will be calculated and reported by the analytical laboratory for every data set. ANL personnel will calculate field precision during the data validation process because samples to assess field precision are submitted blind to the laboratory. Analytical completeness will also be calculated by ANL once all data measurements have been completed and validated.

14.1 DATA ACCURACY

Data accuracy is a measure of the closeness of analytical measurements to the true value. It is commonly expressed as percent recovery or percent bias. Percent recovery often is used because laboratories assess accuracy through the use of spiked samples. Percent recovery is defined as:

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100 \quad , \tag{14.1}$$

where

SSR = spiked sample result,

SR = sample result, and

SA = spike added.

Percent bias is a standardized average error that measures the under- or overestimation of the true value. It is the average error divided by the actual or spike concentration and converted to a percentage. Percent bias can also be expressed as:

$$\%\ Bias = \%\ Recovery - 100 \quad . \tag{14.2}$$

14.2 DATA PRECISION

Data precision is a measure of the size of the closeness of agreement among individual measurements. It examines the spread of the reported values about their mean. The spread of reported values refers to how different the individual reported values are from the average reported value. One method of determining data precision is by calculating the standard deviation or sample variance (the square of the standard deviation).

Laboratories commonly determine precision from replicate (duplicate) samples. In this case, precision is expressed as the RPD or relative standard deviation (RSD) (also called coefficient of variation). The RPD is calculated as follows:

$$RPD = [(XA - XB)/XM] \times 100 , \quad (14.3)$$

where

XA and XB are duplicate analyses, and

XM is the mean value of duplicate analyses XA and XB.

The RSD is calculated as follows:

$$RSD = [2(|XA - XB|)/(XA + XB)] \times (100/\sqrt{2}) . \quad (14.4)$$

14.3 MEASURE OF DATA COMPLETENESS

Completeness is a measure of the relative number of analytical data points that are judged to be valid (i.e., that meet all the acceptance criteria for accuracy, precision, and any other criterion required by the specific analytical methods used). The percent completeness for analytical data is expressed as follows:

$$\% \ Completeness = \frac{V}{T} \times 100 , \quad (14.5)$$

where

V = valid data, and

T = total analyses.

15 CORRECTIVE ACTION

This section outlines corrective action protocols that will be instituted if problems are detected in the sample analysis process. If problems develop in the prime or referee laboratory, the ANL project QAO will be notified. The QAO will then determine the necessary corrective action on the basis of technical judgment and knowledge of established procedures or on the basis of predetermined limits under the J-Field RI Work Plan. Depending on the LOC and the need for APG's involvement, the ANL project QAO shall consult with the APG project QAO regarding the need for action and resolution of the problem. Data may be rejected as a result of either data validation procedures or QC checks. If so, the sample matrix will be resampled and reanalyzed, if possible. If corrective action is necessary, the QAO will use the following protocol to ensure that the nonconforming activity meets specified QA/QC requirements:

- Nonconformance reports will be filed for all activities not performed in accordance with the established requirements.

- The project QAO, in consultation with the project manager, will review the activity to identify the source of the problem and develop a plan to correct the nonconforming items. Corrective action for field sampling and testing problems will be developed with the assistance of the RI field team leader, and corrective action for laboratory problems will be developed with the assistance of the laboratory project manager.

- Any work dependent on the nonconforming activity will be halted until the problem is corrected. The project manager will have the ultimate responsibility for ensuring that corrective actions are fully implemented.

- The project QAO will be notified when corrective actions are completed. At that time, a follow-up audit will be conducted, and a written report of corrective action acceptance will be filed with the program managers.

16 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The QAO will prepare QA reports on an as-needed basis for distribution to the program managers and project manager. These documents will report on assessments of measurement data accuracy, precision, and completeness; results of performance and system audits; and detection of any significant QA problems and recommended solutions.

The following documents and deliverables will be submitted as deemed necessary to the DSHE in support of the RI work conducted at J-Field:

- Audit reports,
- QA/QC reports during field and laboratory activities and data review,
- IRDMS submissions,
- Logbooks,
- QA section of the project final report, and
- Project final report.

The AEC will be responsible for the final storage and security of all data files at APG. Any changes proposed to this QAPjP before the close of the project will be submitted to the J-Field project manager at DSHE, John Wrobel.

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U.S. Environmental Protection Agency, 1984b, *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule,* 40 CFR Part 136, Part VIII, Washington, D.C., Oct. 26.

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18 LIST OF PREPARERS

This QAPjP was prepared for the U.S. Army, Directorate of Safety, Health, and Environment, Aberdeen Proving Ground, by the Environmental Assessment Division of ANL. The following ANL staff members have contributed to the preparation of this report.

Name	Education/Experience	Contribution
Louis Martino	M.S., environmental toxicology; 15 years experience in environmental assessment.	J-Field project manager
Surya Prasad	Ph.D., soil science/soil chemistry; 17 years experience in agricultural and environmental assessment.	Lead author
Terri Patton	M.S., geology; 6 years experience in radiochemical analysis; 5 years experience in environmental assessment.	Author

APPENDIX A:

STANDARD OPERATING PROCEDURES FOR THE J-FIELD REMEDIAL INVESTIGATION

APPENDIX A:

STANDARD OPERATING PROCEDURES FOR THE J-FIELD REMEDIAL INVESTIGATION

This appendix presents the supplemental SOPs developed by ANL for J-Field RI activities. Other SOPs used by ANL were developed by the COE Waterways Experiment Station and are presented in COE (1993). These SOPs are updated periodically by COE and are accessible through the Internet. A list of all SOPs used in the RI work is provided in Table A.1.

TABLE A.1 Identification Numbers and Titles of Relevant Standard Operating Procedures for J-Field[a]

SOP No.	Title
U.S. Army Corps of Engineers SOPs[b]	
001	Sample Labels
002	Chain-of-Custody Form
003	Field Logbook
004	Sample Packing and Shipping
005	Decontamination
006	Use of the IRDMS Network
007	Surface Water Sampling Procedures
008	pH Measurement
009	Temperature Measurements
010	Water Level and Well-Depth Measurements
011	Photoionization Detector (HNu Model P1-101)
012	Specific Conductance Measurements
013	Collection of Monitoring Well Samples
014	Collection of Production Well Samples
015	Document Control System
016	Surface Water, Groundwater, and Soil/Sediment Field Logbooks
017	Ground Penetrating Radar Survey
018	Electromagnetic Induction (Terrain Conductivity) Surveys
019	Monitoring Well Installation
020	Active Soil Gas Sampling
021	Sediment Sampling
022	Benthic Tissue Sampling
023	Organic Vapor Analyzer (Foxboro 128 GC)
024	Photoionization Detector (Microtip HL-200)
025	Soil Sampling
026	Active Soil Gas Analysis
027	Passive Soil Gas Survey
028	Well and Boring Abandonment
029	Extraction Wells
030	Radioactivity Surveys

TABLE A.1 (Cont.)

SOP No.	Title
031	Sample Container Cleaning
032	Piezometer Installation
033[c]	Slug Tests
034	"Orphan or Unclaimed" Wells
035	Agent Screening
036	Turbidity Measurements
037	Dissolved Oxygen Measurements
038	Redox Potential Measurements
039	Sample Preservation
040	Confined Space Entry
041	Sludge Sampling Procedures
042[c]	Disposal of Environmental Well Development/Purge Water
043[c]	Hydrolab Multiparameter Water Quality Monitoring Instrument
044[c]	Assessment of Existing Wells Using Downhole Camera
045[c]	Assessment of Tidal Effects on Groundwater
Argonne National Laboratory Supplemental SOPs[d]	
S001	Field Screening of Metals in Soil by X-Ray Fluorescence
S002	Immunoassay Field Screening Method for Rapid PCB, TPH, and PAH Analysis of Soil
S003	Microbial Assessment Using MICKIT
S004	Surface Soil Sampling for Radiological Testing
S005	EMFLUX Passive Soil-Gas Surveys

[a] The SOPs listed here are periodically updated.

[b] Source: COE (1993).

[c] Denotes SOP in draft.

[d] Developed by Argonne National Laboratory for J-Field RI.

REFERENCE

U.S. Army Corps of Engineers (COE), 1993, *Work Plan for CERCLA Remedial Investigation / Feasibility Study; Appendix J, Standard Operating Procedures*, Directorate of Safety, Health, and Environment, U.S. Army Aberdeen Proving Ground, Md., in cooperation with Waterways Experiment Station, Vicksburg, Miss., Dec.

STANDARD OPERATING PROCEDURE S001
FIELD SCREENING OF METALS IN SOIL
BY X-RAY FLUORESCENCE

1 PURPOSE

The purpose of this standard operating procedure is to present the instruments and protocols for screening selected metals in soil using state-of-the-art elemental analyzer(s) based on the energy-dispersive x-ray fluorescence (EDXRF) technique.

2 MATERIAL

X-ray fluorescence analyzer.

3 PROCEDURE

3.1 INSTRUMENT

The x-ray fluorescence (XRF) offers the advantage of little to no sample preparation and speed of analysis. The technology, in association with portable instrumentation, offers convenience for field use. The hand-held probe provides convenience for in situ analysis of soils and soil cores.

Information was obtained about Spectrace 9000, distributed by Spectrace Instruments, Inc., in Skillman, New Jersey. Standard operating procedures of this instrument will be followed per documentation furnished by the individual manufacturer and are retained in project file. This analyzer extends analysis sensitivity at the level of part per million.

3.2 DETECTABLE METALS

The metals that are detectable with the XRF analyzer include a majority of those listed under the EPA's CLP TAL (EPA 1985, 1988, 1989). At least 16 of the 23 TAL metals on EPA's CLP list can be detected with Spectrace 9000. The detectable metals include antimony, arsenic, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, potassium, selenium, and zinc.

3.3 LIMITS OF DETECTION

The limits of detection for each element with the above-described technique and instrument are shown in documentation furnished by the manufacturer and retained in the project file.

4 MAINTENANCE

See documentation furnished by the manufacturer.

5 PRECAUTIONS

None.

6 REFERENCES

U.S. Environmental Protection Agency, 1985, *U.S. EPA Contract Laboratory Program, Statement of Work (SOW) for Inorganic Analysis, Multi-Media, Multi-Concentration,* SOW NO. 785, Washington, D.C., July.

U.S. Environmental Protection Agency, 1988, *User's Guide to Contract Laboratory Program,* EPA/540/8-89/012, Office of Emergency and Remedial Response, Washington, D.C., December.

U.S. Environmental Protection Agency, 1989, *U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration,* SOW 788, revised June 1980, February 1989, and June 1989, Washington, D.C.

STANDARD OPERATING PROCEDURE S002 IMMUNOASSAY FIELD SCREENING METHOD FOR RAPID PCB, TPH, AND PAH ANALYSIS OF SOIL

1 PURPOSE

Selected PCBs/Aroclors, PAHs, and TPHs in soils, including surface and near-surface soils, will be screened with state-of-the-art immunoassay field screening methods for rapid field analysis. The method is based on immunochemistry for environmental monitoring (EPA 1991).

2 MATERIAL

Immunoassay field kit.

3 PROCEDURE

3.1 STANDARD OPERATING PROCEDURES

Standard operating procedures for each analyte and environmental medium '(including soils and water) based on immunochemistry are presented in copies of product information furnished by the manufacturers and are retained in the project file. For example, the soil test for PCBs consists of four steps: sample preparation, sample analysis, color development, and interpretation of results.

1. Sample preparation: A sample is collected and extracted with methanol. The methanol extract is filtered and diluted with a buffer.

2. Sample analysis: The prepared sample is added to a test tube that is coated with antibodies that specifically detect PCBs. After 10 minutes, several drops of enzyme-labeled analyte are also added to the tube. The contents are mixed and allowed to stand for 5 minutes.

3. Color development: A substrate and chromogen that react with the enzyme are added to each tube. The color intensity formed is inversely proportional to sample concentration.

4. Interpretation of results: The concentration of the sample is determined with a portable photometer by comparison to a standard.

3.2 DETECTABLE ANALYTES

The detectable PCBs include Aroclors 1242-1260 within a detection limit of 5 ppm and sensitivity within 5 ppm and Aroclors 1016-1221 within a sensitivity range of 10-20 ppm and detection limit of 5 ppm. The Aroclors included are those listed under the EPA's CLP TAL (EPA 1988, 1989, 1991). Literature on the immunoassay methods and commercially available field kits developed by EnSys, Research Triangle Park, North Carolina, and Millipore Corporation, Marlborough, Massachusetts, are retained in project files for use toward field application.

4 MAINTENANCE

See documentation furnished by manufacturer.

5 PRECAUTIONS

None.

6 REFERENCES

U.S. Environmental Protection Agency, 1988, *User's Guide to Contract Laboratory Program,* EPA/540/8-89/012, Office of Emergency and Remedial Response, Washington, D.C., December.

U.S. Environmental Protection Agency, 1989, *U.S. EPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration,* SOW No. 2/88, including revisions 9/88 and 4/89, Washington, D.C.

U.S. Environmental Protection Agency, 1991, *Immunochemistry for Environmental Monitoring, U.S. Environmental Protection Agency Fact Sheet,* Environmental Monitoring Systems Laboratory, Las Vegas, Nev.

STANDARD OPERATING PROCEDURE S003
MICROBIAL ASSESSMENT USING MICKIT™

1 PURPOSE

The purpose of this standard operating procedure is to delineate the steps of performing a microbial assessment on soil to determine what bacteria are present. The method detects microorganisms that can be correlated with toxic contaminants present in the soil. The MICKIT™ III is a complete kit that detects acid-producing bacteria, sulfur-reducing bacteria, and anaerobic and aerobic bacteria.

2 MATERIAL

The MICKIT™ bacterial detection system is developed by:

BioIndustrial Technologies, Inc.
Post Office Box 323
Grafton, New York 12082
(518) 279-9731

3 PROCEDURE

3.1 PREPARING THE SAMPLES

1. Determine medium to be sampled (e.g., soil or water) and begin the procedure as soon as possible. Take care that the area being sampled is disturbed as little as possible with tools and that the area is not touched by anybody's hands. If a water sample is to be taken, pour out the liquid that is in the sample collection bottle labeled "ADS," then collect the water sample in this bottle. Try to get at least 10 mL of sample. If solid samples are to be collected and then analyzed for anions and organic acids, use the sterile di water contained in the 50-mL centrifuge tube to prepare a separate slurry from the same sample site.

2. Before taking the sample, label each set of media bottles (with the colored caps) with numbers 1, 2, 3, 4, and 5 with the marking pen provided. *Label all bottle sets.*

3. With the sterile tongue depressor provided, scrape soil from the metal surface, approximately 1 $in.^2$ if available, as completely as possible into the sample collection bottle labeled ADS. For chemical analyses, try to divide the sample equally and prepare an additional slurry from the same sample site using the sterile di water in the 50-mL centrifuge tube. You may use the top edge of the ADS bottle or centrifuge tube to aid in scraping the material into the bottle itself. Do not touch the area being sampled, the end of the tongue depressor, or the inside of the bottle with your hands.

4. Shake the bottle and tube well to make a slurry of the material.

5. Flip off the tops of the media bottles (the flip-top bottles have a matching colored seal to facilitate keeping track of them once the top is popped). Wipe the rubber bottle tops with the alcohol spongette provided.

6. Withdraw 1 mL of the slurry from the ADS bottle with one of the sterile 3-mL syringes and needles provided.

7. Inject this 1 mL of the slurry into the green-capped bottle labeled #1.

8. Without removing the needle from the bottle, draw 1 to 2 mL of the media-sample mixture into the syringe and inject it back into the bottle several times. This rinses the syringe of remaining sample and simultaneously mixes the solution. Now withdraw 1 mL of this mixture and inject it into the green-capped bottle labeled #2 and mix as just described. Continue this procedure for each of the three remaining green-capped bottles in the set.

9. With a new sterile 3-mL syringe, repeat Steps 6 through 8 for each set of media bottles.

10. With the 5-mL syringes provided, move 5 mL of the slurry from the centrifuge tube.

11. Next, remove the needle from the syringe and attach the syringe to the threaded end of the blue filter unit. Attach the sterile needle provided in pin wrapper to the other end of the filter unit. Note: Be careful not to touch the needle with your hands. Leave the plastic needle cover on until it is used.

12. Inject as much of the sample as possible, preferably all 5 mL, through the filter and into the empty bottle with the white top. Note: In some cases, it may be difficult to push the sample through the filter. Do not force it through the filter because the filter may break. Slowly push it through the filter until the syringe is evacuated or the filter becomes completely clogged.

13. With the 1-mL syringe provided, remove 1 mL of slurry from the ADS bottle and inject it into the bottle with the blue top that contains a buffered fixative solution. Please do this step last because this bottle contains a preservative that may damage the other bottles.

14. Discard the tongue depressor, alcohol swab, syringes, needles, and filter. Needles must be destroyed before disposal by cutting the tip off or bending back the needles. According to federal law, syringes must be destroyed by breaking or shattering the barrel.

15. Incubate all bottles of media at room temperature, preferably in the dark box, for up to 28 days. Most bottles will react in a few days. The long incubation period is required only for samples with a very small number of bacteria in the inoculum; they need longer to grow and give the appropriate reaction.

3.2 READING THE BOTTLES

1. Green-capped bottles detect sulfate-reducing bacteria. These bottles will turn black in the presence of such organisms. Record the highest bottle number that becomes black.

2. Blue-capped bottles detect the presence of anaerobic or partially anaerobic bacteria. A positive reaction results in a cloudy appearance from bacterial growth. Record the highest bottle number in which the media is cloudy as opposed to the golden clear appearance of the sterile bottles before inoculation.

3. Red-capped bottles detect acid-producing organisms. The media will turn from red to yellow if such bacteria are present. Record the highest bottle number that turned yellow, 1 through 5.

4. Gold-capped bottles detect the presence of general aerobic bacteria and are read in the same manner as the blue-capped bottles. Record the highest number of these bottles with a cloudy appearance. Note: So far

serial, decimal dilutions of the sample in these media bottles are performed. This means that you can get an approximation of the numbers of the various types of bacteria using the chart below:

Highest Bottle to React	Approximate Count (in cells/mL of sample)
1	1-10
2	10-100
3	100-1,000
4	1,000-10,000
5	Greater than 10,000

5. The bottle with the blue top that contains the sample in a fixative solution is included.

6. The empty bottle into which you filtered a sample is also provided, in case you require further analysis of the sample for materials important in microbial corrosion, metals, sulfates, nitrates, chlorides, and organic acids.

4 MAINTENANCE

See documentation furnished by manufacturer.

5 PRECAUTIONS

None.

6 REFERENCE

BioIndustrial Technologies, Inc., 1992, *MICKIT™ III for the Detection of Problem-Causing Bacteria Involved in Microbiologically Influenced Corrosion*, Grafton, N.Y.

STANDARD OPERATING PROCEDURE S004
SURFACE SOIL SAMPLING FOR RADIOLOGICAL TESTING

1 PURPOSE

The purpose of this standard operating procedure is to detail procedures for surface soil sampling for radiological testing (ANL Procedure No: SAM-010 REV-00, dated March 13, 1992) that will be used by ANL personnel during the RI at J-Field, U.S. Army Aberdeen Proving Ground, Aberdeen, Maryland. The objective of the operation is to be able to obtain representative soil samples for radiological testing.

2 MATERIAL

1. Trowel
2. Ziploc bags (8 in. by 8 in.)
3. Plastic bags (large)
4. Tape measure
5. Electrical tape
6. Marinelli beakers (for 3 in. by 3 in. NaI) (optional)
7. Plastic gloves
8. Data book (or similar log)
9. Alcohol wipes or alcohol and paper towels

3 PROCEDURE

1. Identify suitable (or designated) area for sampling by map and/or surrounding fixed features.

2. Lay out a square meter area for sampling.

3. Remove the top 0.5 in. of soil from at least each corner and the center area of the square. Minimum sample size should be about 0.5 kg.

4. Place this soil in a large Ziploc bag or a 500-mL MarineIIi beaker, as directed.

5. Mark bag or beaker with sample number, location, and data. Group this information and other comments in logbook.

6. Place bag or beaker in another plastic bag and seal.

7. Clean trowel with alcohol wipes.

8. Repeat process as necessary.

9. Bag and label all potentially contaminated wastes.

10. Return sample to field mobile laboratory for gamma spectrometric analysis.

4 MAINTENANCE

Not applicable.

5 PRECAUTIONS

None.

6 REFERENCES

None.

STANDARD OPERATING PROCEDURE S005
EMFLUX® PASSIVE SOIL-GAS SURVEYS

1 SCOPE AND APPLICATION

This standard operating procedure describes the field procedure for collecting soil-gas samples using the EMFLUX® passive, nonintrusive, surface-based soil-gas detection system. This system measures the time-integrated gas emission flux rate at the soil surface and can detect soil-gas contaminants in the part per billion (ppb) range.

Questions regarding variations in this field procedure should be addressed to the technical staff at:

Quadrel Services, Inc.
1896 Urbana Pike No. 20
Clarksburg, Maryland 20871
(301) 874-5510

2 MATERIAL

1. EMFLUX® system components and plastic carrying case
2. Short-handled garden hoe, to clear vegetation as necessary
3. Gloves
4. Doughnut-shaped weights, for underwater sampling

3 PROCEDURE

Sampling will take place at predetermined sampling sites. The sites will be surveyed in using compass bearings from fixed landmarks at the site and measuring at specified distance intervals with a tape measure.

3.1 SAMPLER ASSEMBLY EMPLACEMENT

The following procedure describes how the soil-gas sampler should be emplaced at each survey point. Underwater sampling is similar to on-land sampling except as noted. For best results, a two-person team should emplace the sampler assembly. One team member, designated as team member 1 in the following text, must wear gloves and will have the exclusive responsibility for handling the system components, which must be protected from contamination. Team member 2 is responsible for handling all other equipment.

1. Team member 2 will clear a 4-in. diameter area of vegetation using the short-handled garden hoe, as necessary.

2. Team member 2 will then open the resealable bag containing the stainless steel stake and allow team member 1 to remove the stake with a gloved hand. Team member 2 will also open the air-tight vial containing the laboratory-prepared sampler cartridge and up-end the vial over the gloved hands of team member 1 so that the sampler will fall into his/her hands. Team member 2 will place the cap back on the vial.

3. Team member 1 will then affix the sampler cartridge to the stake and secure the sampler assembly to the specified point on the ground (to a depth of about 1/4 in.).

   NOTE: For underwater sampling, team member 1 will affix the sampler cartridge to the inside top of the sampler shell. The sampler cartridge will hang in place.

4. Team member 2 will immediately place the sampler shell, open-end down, over the sampler assembly and surround it with a collar of sand or local soil (to minimize effects of ambient airflow). Assembly of the sampler and emplacement of sampler shell should occur rapidly to minimize the sampler's exposure to ambient air.

   NOTE: For underwater sampling, team member 2 will place several doughnut shaped weights on top of the sampler shell (enough to keep the sampler shell in place).

5. Team member 2 will record the survey point location number, cartridge number, date and time of emplacement, and any other relevant information required by SOP-003 (Field Logbook). This information will also be recorded on the air-tight vial for each sample.

6. The team will leave the sampler in place for at least 72 hours.

3.2 SAMPLER ASSEMBLY RETRIEVAL

The following procedure describes how the soil-gas sampler should be retrieved from each survey point.

1. Team member 2 will remove the sampler shell so that team member 1 can recover the sampler assembly with a gloved hand. Team member 2 will hold and open the air-tight vial marked with the location number, cartridge number, and date and time of emplacement.

2. Team member 1 will disassemble the sampler assembly and immediately place the sampler cartridge in the air-tight vial. Team member 2 will reseal the vial.

   NOTE: For underwater sampling, team member 1 will remove the sampler cartridge from the sampler she and immediately place it in the air-tight vial.

3. Team member 1 will place the steel stake in the plastic carrying case. Team member 2 will place the sampler shell in the plastic carrying case. These components will be decontaminated for reuse at a later time.

   NOTE: For underwater sampling, the steel stake is not used. The only component to be placed in the plastic carrying case is the sampler shell (by team member 2).

4. Team member 2 will record the survey point location number, cartridge number, date and time of retrieval, and any other relevant information required by SOP-003 (Field Logbook). The date and time of retrieval will also be recorded on the air-tight vial for each sample.

5. The resealed sample vials will be packaged for shipment by Federal Express to Maryland Spectral Services for analysis. Other sampler components will be shipped to Quadrel Services, Inc., for decontamination.

4 MAINTENANCE

Not applicable.

5 PRECAUTIONS

Care must be taken to ensure that sample vials are not broken during sample emplacement, retrieval, and transport. Samples for which vials are found to be broken will be considered invalid.

6 REFERENCE

Quadrel Services, Inc., 1993, *Field Procedures for EMFLUX® Soil Gas Surveys*, Clarksburg, Md.

APPENDIX B:

SYSTEM AUDIT CHECKLIST FOR FIELD OPERATIONS

SYSTEM AUDIT CHECKLIST FOR FIELD OPERATIONS[1]

Project Name:_____

Project Number:_____ Date:_____

Project Location:_____

Field Team Leader:_____

Field Team Members:_____

_____

_____

Name of Auditor:_____

Signature of Auditor:_____

Yes_____ No_____ 1. Is a set of accountable field documents checked out to the site manager?

Comments:_____

_____

_____

_____

[1] This checklist is based on guidance presented in EPA reports QAMS-005/80 and OWRS QA-1 (EPA 1980, 1984).

Yes_____ No_____ 2. Is the transfer of field operations from the site manager to field participants documented in a logbook?

Comments:_____

_____

_____

_____

Yes_____ No_____ 3. Is there a written list of sampling locations and descriptions?

Comments:_____

_____

_____

_____

Yes_____ No_____ 4. Are samples collected as stated in the work plan or as directed by the site manager?

Comments:_____

_____

_____

_____

Yes_____ No_____ 5. Are samples collected in the type of containers specified in the project plan or as directed by the site manager?

Comments:_____

_____

_____

_____

Yes_____ No_____ 6. Are samples preserved as specified in the project plan or as directed by the site manager?

Comments:_____

_____

_____

_____

Yes_____ No_____ 7. Are the number, frequency, and type of samples collected as specified in the project plan or as directed by the site manager?

Comments:_____

_____

_____

_____

Yes_____ No_____ 8. Are the number, frequency, and type of measurements taken as specified in the project plan or as directed by the site manager?

Comments:_____

_____

_____

_____

Yes_____ No_____ 9. Are samples identified with sample labels?

Comments:_____

_____

_____

_____

Yes_____ No_____ 10. Are blank and duplicate samples properly identified?

Comments:_____

Yes_____ No_____ 11. Are samples and serial numbers for samples split with other organizations recorded in a logbook or on a chain-of-custody record?

Comments:_____

Yes_____ No_____ 12. Are samples listed on a chain-of-custody record?

Comments:_____

Yes_____ No_____ 13. Is chain-of-custody documented and maintained?

Comments:_____

Yes_____ No_____ 14. Are quality assurance checks performed as directed?

Comments:_____

Yes_____ No_____ 15. Are photographs documented in logbooks as required?

Comments:_____

_____

_____

_____

Yes_____ No_____ 16. Are all documents accounted for?

Comments:_____

_____

_____

_____

Yes_____ No_____ 17. Have any documents been voided?

Comments:_____

_____

_____

_____

Yes_____ No_____ 18. Have any documents been destroyed?

Comments:_____

_____

_____

_____

REFERENCES

U.S. Environmental Protection Agency, 1980, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*, QAMS-005/80, Washington, D.C., Dec.

U.S. Environmental Protection Agency, 1984, *Guidance for Preparation of Combined Work/Quality Assurance Project Plans for Environmental Monitoring*, OWRS QA-1, Washington, D.C., May.