Environmental Restoration Program

METHODS COMPENDIUM

Mound Plant
Miamisburg, Ohio

Department of Energy
Ohio Field Office
EG&G Mound Applied Technologies

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Introduction

INTRODUCTION

INTRODUCTION

In 1995, the DOE Mound Plant Environmental Restoration (ER) department, the Ohio Environmental Protection Agency (OEPA) and the United States Environmental Protection Agency (USEPA) developed a program to identify and evaluate potential release sites and, if required, remediate the contaminated sites. These potential release sites were identified on the basis of data collected during previous sampling and investigative programs. Because there is previous data which characterizes the contamination at these release sites, the sampling and analysis methods selected for further evaluating the release sites could, in many cases, be highly focused. For example, if a potential release site had been identified to have chromium contamination and more information was required, then DOE resources could be used to focus the sampling and analysis methods on collecting additional chromium data and not spent confirming the lack of other contaminants (e.g. volatile organics, semi-volatile organics, other metals, etc.)

Given this change in the focus of the DOE Mound Plant mission, this compendium was generated. The compendium was designed to act as a depository for sampling, analysis, and quality control methods implemented on the plant site. The initial compendium methods were extracted from the Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan (QAPP), April 1995, revision 4. Because the QAPP was approved for evaluating the nature and extent of contamination throughout the plant site and contains extensive target analyte lists, the QAPP methods were included in the compendium to provide a common basis in the event potential release sites are identified which lack adequate data to develop a focused target analyte list.

The compendium is divided into five sections: Analytical Methods, Field Methods, Quality Assurance Methods, Data Validation Methods, and Field Standard Operating Procedures. Methods within each section have been identified using a prefix and a sequential number. The prefix codes are shown below.

Type of Method	Prefix
Analytical Methods	A
Field Methods	F
Quality Assurance Methods	Q
Data Validation Methods	D
Field Standard Operating Procedures	S

At the beginning of the each section, there is a short introduction and a table of contents for the section. The methods extracted from the QAPP can be identified in three ways: by the table of contents at the beginning of each section, within the introduction to each of the sections, or on the title page of each of the methods where the source document is listed. As new methods are

added, the table of contents will be updated and distributed to the copy holders with the new methods. Each of the new methods will include a source document reference and document date. The source document and document date will be used to identify the first potential release site approved for the use of the method.

Section 1

ANALYTICAL METHODS

ANALYTICAL METHODS

Analytical methods describe the quality control requirements for methods of analysis performed at off-site laboratories. Analytical methods 1 to 18 were extracted from the Remedial Investigation/Feasibility Study Operable Unit 9, Site-wide Quality Assurance Project Plan (QAPP). The extracted methods retain as much of the original text from the QAPP as feasible. However, because many of the methods with similar quality control requirements were discussed within the same paragraph of the QAPP, some text was revised for inclusion in the individual methods. Additionally, the method modifications in Appendix B of the QAPP, and included in each of the CLP methods (A-001, A-003, A-004, and A-005) were revised to only address the changes applicable to the attached method. Each of the methods extracted from the QAPP lists the source document as QAPP and the document date as April 1995.

The methods extracted from the QAPP should be usable for characterizing the extent and degree of contamination of potential release sites which have:

- limited analytical data;
- inconclusive analytical data; or
- no previous sample data.
-

Where release site data are available, the analyte list for the QAPP approved methods should be appropriately reduced or new methods should be introduced to collect focused and usable analytical data. If the analyte list is reduced, the reduced analyte list should be noted in the appropriate sample plan. If a new method is added, then:

- the method should be added to this compendium;
- section 1.1 of the method should describe the use of the method; and
- the method should be identified in the sample plan.

When a new method is approved for use with a specific release site, then:

- the Source Document and Document Date on the title page of the method must be updated, and

- both the method and a revised table of contents for the section must distributed to all copy holders. The DOE prime contractor will be responsible for the distribution or assigning the distribution to a subcontractor.

ANALYTICAL METHODS — TABLE OF CONTENTS

MOUND

Environmental
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Method: A-001

Volatile Organic Analysis by CLP SOW OLM01.8

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Soil/sediment and surface water samples will be analyzed for VOCs by the CLP SOW using gas chromatography and mass spectometry as a means for compound identification. Capillary columns as specified in the method will be employed. A modification to the CLP SOW (Attachment A) has been prepared to account for six additional volatile organic compounds: acrylonitrile, acetonitrile, trichlorotrifluoroethane, iodomethane, hexane, and diethyl benzene.

1.2 References

EPA 1990a. "U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multi-Concentration." Document No. ILM1.0 including Revisions 1.1 through 1.8. Environmental Protection Agency, March, 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Volatile Organic Analysis - CLP SOW OLM01.8
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Volatile Organic Compounds	CLP SOW	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH≤2 Cool 4°C	14 days
Soil	Volatile Organic Compounds	CLP SOW	Glass bottle with Teflon-lined septum	120 mL (no headspace)	Cool 4°C	14 days

3. CALIBRATION

Gas Chromatograph/Mass Spectrometry (GC/MS) will be used for analysis of volatile organic compounds. Mass spectral abundance criteria must be met prior to sample analysis. Bromofluorobenzene (BFB) is used to verify instrument performance of the GC/MS system and must meet specific ion abundance criteria established in the CLP SOW. Meeting these criteria is demonstrated daily or every 12-hour time period, whichever is more frequent. The instrument performance is also verified whenever a corrective action to the GC/MS system is taken that affects the tuning (e.g., ion source cleaning or repair).

Initial calibration of the GC/MS system is accomplished with a minimum of five concentrations of target compounds. Relative Response Factors (RRFs) must be greater than or equal to 0.05. Relative standard deviations for the RRFs must be less than or equal to 30%. Initial calibration is

not valid if this criterion is not met. The relative retention times of each compound in each standard run must agree within 0.06 units.

The initial calibration is verified every 12-hour period with a continuing calibration standard containing all target volatile compounds and surrogate compounds. RRFs are compared to the average RRF from the initial calibration. The minimum RRF for the target compounds must be met. The percent difference between the initial RRFs and the continuing RRF must be less than or equal to 25 percent for the initial calibration to be valid. Prior to sample analysis, the GC/MS system is evaluated and corrective action taken if these criteria are not met.

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis CLP SOW OLM01.8 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOA, CLP SOW	Trip Blank	1 per shipping container to lab	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Sample bank blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Ambient blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (**water**) 1 every 10 or fewer field samples (**soil**)	≤ 25% RPD N/A	Evaluate data for usability. Evaluate variability.

Table 4.2 - Volatile Organic Analysis CLP SOW OLM01.8 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOA, CLP SOW	Method Blank	Once per 12-hour period	≤ 5 x CRQL of common lab contaminants ≤ CRQL others	Investigate source; reanalyze associated samples.
	Matrix Spike	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability.
	Laboratory control sample	Once per 12-hour period	See Table 4.3	Evaluate associated data for usability.
	System monitoring compounds	All lab and field samples	CLP SOW	See CLP SOW.
	Instrument performance check	Daily or each 12-hour period, whichever is more frequent	CLP SOW	Retune: Reanalyze associated samples
	Calibration	CLP SOW	±0.06 relative retention time units (sample and standard)	Recalibrate before sample analysis
	Retention time window	CLP SOW	CLP SOW	See CLP SOW.
	Qualitative verification	When a detection occurs in a sample	CLP SOW	See CLP SOW.
	Calibration check	With every calibration	CLP SOW	Recalibrate.
	Internal standard	Every standard and sample	CLP SOW	See CLP SOW.
	Continuing calibration check	Once each 12-hour period	CLP SOW	Identify source and correct. Recalibrate if source not found and corrected.

Table 4.3 - Volatile Organic Analysis CLP SOW OLM01.8 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
CLP SOW Volatile Organic Compounds	*Matrix Spike/LCS*						
	1,1-DCE	per CLP SOW	per CLP SOW	61-145	59-172	≤14	≤22
	Trichloroethene	per CLP SOW	per CLP SOW	71-120	62-137	≤14	≤24
	Benzene	per CLP SOW	per CLP SOW	76-127	66-142	≤11	≤21
	Toluene	per CLP SOW	per CLP SOW	76-125	59-139	≤13	≤21
	Chlorobenzene	per CLP SOW	per CLP SOW	75-130	60-133	≤13	≤21
	Surrogates						
	Toluene-d8	per CLP SOW	per CLP SOW	88-110	84-138	NA	NA
	4-Bromo-fluorobenzene	per CLP SOW	per CLP SOW	86-115	59-113	NA	NA
	1,2-Dichloroethane-d4	per CLP SOW	per CLP SOW	76-114	70-121	NA	NA

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Volatile Organic Analysis CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Chloromethane	10	10
Bromomethane	10	10
Vinyl Chloride	10	10
Chloroethane	10	10
Methylene chloride	5	5
Acetone	10	10
Carbon disulfide	5	5
1,1-Dichloroethene	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (total)	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
2-Butanone	10	10
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
Bromodichloromethane	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
Trichloroethene	5	5
Dibromochloromethane	5	5
1,1,2-Trichloroethane	5	5
Benzene	5	5
trans-1,3-Dichloropropene	5	5
Tribromomethane	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	5	5
Toluene	5	5
1,1,2,2-Tetrachloroethane	5	5
Chlorobenzene	5	5
Ethylbenzene	5	5
Styrene	5	5
Xylenes (total)	5	5
Additional Compounds:		
Acrylonitrile	100	100
Acetonitrile	100	100
Diethylbenzene	5	20
Trichlorotrifluoroethane	5	10
Hexane	10	10
Iodomethane	NA	10
Vinyl Acetate	10	10

ATTACHMENT TO METHOD A-001

Attachment to Method A-001

Modification to CLP Organic SOW OLM01.8
"Statement of Work for Organic Analysis,
Multi-media, Multi-concentration"

The purpose of this addendum is to outline modifications to the Contract Laboratory Program (CLP) "Statement of Work for Organic Analysis, Multi-media, Multi-concentration" which are project specific to the QAPP prepared by Roy F. Weston, Inc. for investigative activities at the Department of Energy/LANL Mound Plant, Miamisburg, Ohio.

This addendum extends the analysis to include acetonitrile, acrylonitrile, 1,2-diethylbenzene, hexane, iodomethane 1,1,2-trichloro-1,2,2-trifluoroethane, and vinyl acetate for volatiles.

Exhibit A - Summary of Requirements

No modifications to this section.

Exhibit B - Reporting and Deliverables Requirements

Section I: Contract Reports/Deliverables Distribution
No modifications to this section.

Section II: Report Descriptions and Order of Data Deliverables
No modifications to this section.

Section III: Form Instructions
No modifications to this section.

Section IV: Data Reporting Forms:
The following compounds must be added on Form I (Data Sheets).

CAS No.	Analyte
75-05-8	Acetonitrile
107-13-1	Acrylonitrile
76-13-1	1,2,2-Trichloro-1,2,2-trifluoroethane
74-88-4	Iodomethane
110-54-3	Hexane
135-01-3	1,2-Diethylbenzene
108-05-04	Vinyl acetate

Form VI VOA (Initial Calibration), and Form VII VOA (Continuing Calibration) must be modified to include these additional seven VOA compounds.

Exhibit C - Target Compound List (TCL) and Contract Required Quantitation Limits(CRQL)

The following should be added to the Target Compound List (TCL) and Contract required Quantitation Limits(CRQL, Page C-2 and Page C-4):

Analyte	CAS No.	CRQL			On Col. (ng)
		Low Water ug/L	Low Soil ug/kg	Med. Soil ug/kg	
Acetonitrile	75-05-8	100	100	6000	300
Acrylonitrile	107-13-1	100	100	6000	300
1,2,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	10	1200	50
Iodomethane	74-88-4	NA	10	1200	50
Hexane	110-54-3	10	10	1200	50
1,2-Diethylbenzene	135-01-3	5	20	1200	50
Vinyl acetate	108-05-04	10	10	1200	50

Form III

VOA-1 Water: Add: Acrylonitrile and acetonitrile QC Limits for Recovery 70-130% and RPD 15%.

VOA-2 Soil: Add: Acrylonitrile and acetonitrile QC Limits for Recovery 60-140% and RPD 25%.

Exhibit D - Analytical Methods for Volatiles:

Section I: Introduction:

1.1 Scope and Application: No modifications to this section

1.2 Problems: This section is modified to include:

- Acetonitrile may have poor purge efficiency
- Iodomethane can be easily degraded.

Section II: Sample Preparation and Storage

No modifications to this section.

Section III: Optional Screening

No modifications to this section.

Section IV: GC/MS Analysis of Volatiles:

1. Summary of Methods: No modifications to this section.
2. Interferences: No modifications to this section.
3. Apparatus and Materials: No modifications to this section.
4. Reagents: No modifications to this section.
5. Standards:

5.1 - 5.4 The above seven additional compounds must be added to the TCL of standards for preparation of stock standard solutions, secondary dilution standards, and working standards.

5.4.5 Add:

Acrylonitrile is be added to the matrix spike solution at a concentration of 250 ug/L.

5.5 Aqueous Calibration Standard Solutions

5.5.1 Prepare five aqueous initial calibration standard solutions containing all purgeable TCL and additional compounds and system monitoring compounds at 10, 20, 50, 100, 200 ug/L levels except acetonitrile and acrylonitrile which will be prepared at 50, 100, 250, 500, 1000 ug/L.

5.5.2 No modifications to this section

5.5.3 The 50 ug/L aqueous calibration standard solution for all TCL except acetonitrile and acrylonitrile which will be at 250 ug/L is the continuing calibration solution.

5.6 No modifications to this section.

6. Instrument Operating Conditions:

6.1 No modifications to this section.

6.2.1 Final hold time is changed to "Until all target compounds elute."

6.3 and 6.4 No modifications to this section.

7. Calibration:

7.1 - 7.4.5 No modifications to this section.

7.4.6 The additional compounds acetonitrile, acrylonitrile, 1,2-diethylbenzene, hexane, iodomethane, vinyl acetate, and 1,1,2-trichlorotrifluoroethane must be added to the list of compounds. The maximum %RSD of 20.5 and maximum percent difference of 25 is acceptable for all the additional compounds, except acetonitrile and acrylonitrile. Acetonitrile may have a maximum %RSD of 35 and acrylonitrile may have a maximum %RSD of 30. The maximum percent difference for acetonitrile and acrylonitrile is 30. However, these compounds must meet the minimum RRF criteria of 0.01.

These are advisory limits and final limits will be established after method validation.

7.4.7 - 7.4.8 No modifications to this section.

7.5 - 7.9 No modifications to this section.

8. Sample Analysis:

8.1.1 -8.1.15 No modifications to this section.

8.1.16 Add: The concentration of acrylonitrile, the additional matrix spike compound is 250 ug/L.

8.1.17 - 8.1.18 No modifications to this section.

8.2.1.1 - 8.2.1.7 No modifications to this section.

8.2.1.8 Add: The concentration of the additional matrix spike compound acrylonitrile would be 250g/kg.

8.2.1.9 - 8.2.1.10 No modifications to this section.

8.2.2.1 - 8.2.2.8 No modifications to this section.

8.2.2.9 Add: The resulting concentration of the additional matrix spike compound in the soil is 31,250 ug/kg.

9. Qualitative Analysis: No modifications to this section.

10. Quantitative Analysis: No modifications to this section.

Table 3 No modifications to this section.

Table 4 The following is added to Table 4:

Analyte	Primary Ion	Secondary Ions
Acetonitrile	41	40
Acrylonitrile	53	52,51
1,1,2-Trichlorotrifluoroethane	101	103, 151,153
Iodomethane	142	127
Hexane	57	86,43,41
1,2-Diethylbenzene	119	134, 115
Vinyl Acetate	43	86

Table 5 Add: The additional compounds acetonitrile, acrylonitrile, 1,2-diethylbenzene, hexane, iodomethane, vinyl acetate and 1,1,2-trichlorotrifluoroethane must be quantitated using the nearest eluting internal standard.

Table 6 No modifications to this section.

Table 7 Add:

	Water		Soil	
Compound	%Recovery	RPD	%Recovery	RPD
Acetonitrile	70-130	15	60-140	25
Acrylonitrile	70-130	15	60-140	25

Exhibit E - QA/QC Requirements

I. Overview:
No modifications to this section.

II. Quality Assurance Plan:
No modifications to this section.

III. Standard Operating Procedure:
No modifications to this section.

IV. QA/QC Requirements: Volatile QA/QC requirements

1. GC/MS Mass Calibration and Ion Abundance Patterns:
No modifications to this section.
2. GC/MS Initial Calibration:
Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.
3. Continuing Calibration:
Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.
4. Internal Standards Responses and Retention Times:
No modifications to this section.
5. Method Blank Analysis:
No modifications to this section.
6. System Monitoring Compound Recoveries:
No modifications to this section.
7. Matrix Spike and Matrix Spike Duplicate Analysis:
Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.
8. Dilution of Samples, MS and MSD
No modifications to this section.

V Analytical Standards Requirements
No modifications to this section.

VI Contract Compliance Screening
No modifications to this section.

VII Regional Data Review
No modifications to this section.

VIII Laboratory Evaluation Samples
No modifications to this section.

IX GC/MS Tape Audits
No modifications to this section.

X Data Package Audits
No modifications to this section.

XI On Site Laboratory Evaluations
No modifications to this section.

XII Quality Assurance and Data Management
No modifications to this section.

XIII Data Management
No modifications to this section.

Exhibit F - Chain of Custody, Document Control, and Standard Operating Procedures

No modifications to this section.

Exhibit G - Glossary of Terms

No modifications to this section.

Exhibit H - Data Dictionary and Format for Data Deliverables in Computer-Readable Format

No modifications to this section

MOUND

Environmental
Restoration
Program

Method: A-002

Volatiles Organic Analysis/ EPA Method 8021

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1. Description

Groundwater samples will be analyzed for halogenated and aromatic VOCs using gas chromatography with a Hall electrolytic conductivity detector and a photoionization detector. The methodology to be followed is EPA Method 8021 (EPA 1987). This method was chosen over the CLP SOW for groundwater samples in order to achieve lower detection limits. Because some of the additional VOCs may coelute with other compounds on the specified capillary column, a Gas Chromatography/Mass Spectrometry (GC/MS) confirmation or second column confirmation will be performed for any detection at the same retention times. If GC/MS confirmation is used, then the data must be reported per the CLP specification as described in Subsection 9.2.3 of the OU9 site-wide QAPP (DOE 1996).

1.2. References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Volatile Organic Analysis - EPA Method 8021
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Volatile Organic Compounds	SW5030/SW8021	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH<2 Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of volatile organic compounds in groundwater (Methods SW-8021). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column). A minimum of five external standards for volatile organic analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for volatile organic compounds (VOCs) are valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for volatile organic analysis is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time windows for VOCs are established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis - EPA Method 8021 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8021	Trip Blank	1 per shipping container to lab	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Sample bank blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Ambient blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (**water**)	≤ 35% RPD	Evaluate data for usability.

Table 4.2 - Volatile Organic Analysis - EPA Method 8021 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8021	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3 x SD of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Volatile Organic Analysis - EPA Method 8021 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS						
Volatile Organic Compounds, SW8021	Bromodichloromethane	*	*	42-172	NA	≤15	NA
	Bromoform	*	*	13-159	NA	≤15	NA
	Carbon tetrachloride	*	*	43-143	NA	≤15	NA
	Chloroform	*	*	49-133	NA	≤15	NA
	Dibromochloromethane	*	*	24-191	NA	≤15	NA
	1,4-Dichlorobenzene	*	*	42-143	NA	≤15	NA
	1,2-Dichloroethane	*	*	51-147	NA	≤15	NA
	1,1-Dichloroethene	*	*	28-167	NA	≤15	NA
	1,1,1-Trichloroethane	*	*	41-138	NA	≤15	NA
	Trichloroethene	*	*	35-146	NA	≤15	NA
	Vinyl Chloride	*	*	28-163	NA	≤15	NA
	Benzene	*	*	39-150	NA	≤15	NA
	Surrogates						
	Bromochloromethane	30	30	59-117	70-130	≤15	≤30
	Fluorobenzene	30	30	48-120	70-130	≤15	≤30
	1,4-dichlorobutane	30	30	60-140	60-140	≤15	≤15
	2-bromo-1-chloropropane	30	30	60-140	60-140	≤15	≤15

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1
Volatile Organic Analysis - EPA Method 8021
Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Vinyl chloride	1.0	NA
Trichlorofluoromethane	2.0	NA
1,1-dichloroethene	1.3	NA
Methylene chloride (dichloromethane)	5.0	NA
1,1-dichloroethane	0.7	NA
Trichloromethane (chloroform)	0.5	NA
1,1,1-trichloroethane	0.3	NA
Carbon tetrachloride	1.2	NA
1,2-dichloroethane	0.3	NA
Trans-1,2-dichloroethene	1.0	NA
Trichloroethene	1.2	NA
1,2-dichloropropane	0.4	NA
Bromodichloromethane	1.0	NA
Dibromomethane	2.0	NA
1,1,2-trichloroethane	0.2	NA
Tetrachloroethene	0.3	NA
Dibromochloromethane	0.9	NA
Chlorobenzene	2.5	NA
1,1,1,2-tetrachloroethane	1.0	NA
Bromoform	2.0	NA
1,1,2,2-tetrachloroethane	0.3	NA
1,2,3-trichloropropane	1.0	NA
Phenyl bromide (bromobenzene)	2.0	NA
Chlorotoluene	1.0	NA
1,3-dichlorobenzene	3.2	NA
1,4-dichlorobenzene	2.4	NA
1,2-dichlorobenzene	1.5	NA
Benzene	2.0	NA
Chlorobenzene	2.0	NA
1,2-Dichlorobenzene	4.0	NA
1,3-Dichlorobenzene	4.0	NA
1,4-Dichlorobenzene	3.0	NA
Ethylbenzene	2.0	NA
Toluene	2.0	NA
Xylene	2.0	NA
Additional Compounds:		
Cis-1,2-dichloroethene	1.0	NA
2-chloroethyl vinyl ether	1.3	NA
Cis-1,3-dichloropropene	3.4	NA
Additional Compounds:		
Trans-1,3-dichloropropene	3.4	NA
1-chlorohexane	1.0	NA

Table 5.1
Volatile Organic Analysis - EPA Method 8021
Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Bis(2-chloroisopropyl)ethyl	20	NA
Trichlorotrifluoroethane	2	NA
Diethylbenzene	1	NA
Vinyl acetate	3	NA
Carbon disulfide	5	NA
Acetone	20	NA
Methylethyl ketone (2-butanone)	10	NA
Methylisobutyl ketone (4-methyl-2-pentanone)	5	NA

MOUND

Environmental
Restoration
Program

Method: A-003

CLP Semi-Volatile Analysis/ CLP SOW OLM01.8

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Soil/sediment and water samples will be analyzed for semi-volatile organic compounds by the EPA CLP SOW Document Number OLOM01.8 (EPA, 1990a), using Gas Chromatography/Mass Spectrometry (GC/MS). A modification to the CLP SOW (Attachment A and B) has been prepared to specify criteria for three additional analytes: benzoic acid, 2-benzyl-4-chlorophenol, and benzyl alcohol.

1.2 References

U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multi-Concentration. Document No. OLM01.8.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Semi-Volatile Organic Analysis - CLP SOW OLM01.8
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Semi-Volatile Organic Compounds	CLP SOW	Amber glass bottle with Teflon-lined lid	Two 1000 mL bottles	Cool 4°C	7 days extraction/ 40 days analysis
Soil	Semi-Volatile Organic Compounds	CLP SOW	Amber glass bottle with Teflon-lined lid	100 grams	Cool 4°C	14 days extraction/ 40 days analysis

3. CALIBRATION

GC/MS will be used for analysis of semi-volatile organic compounds. Mass spectral abundance criteria must be met prior to sample analysis. Decafluorotriphenylphosphine (DFTPP) is used to verify instrument performance of the GC/MS system and must meet specific ion abundance criteria established in the CLP SOW. Meeting these criteria is demonstrated daily or every 12-hour time period, whichever is more frequent. The instrument performance is also verified whenever a corrective action to the GC/MS system is taken that affects the tuning (e.g., ion source cleaning or repair).

Initial calibration of the GC/MS system is accomplished with a minimum of five concentrations of target compounds. Only a four point calibration is required by the CLP SOW for eight of the target semi-volatile compounds that have higher CRQLs. Relative response factors (RRFs) must be greater than or equal to 0.05 Relative standard deviations for the RRFs must be less than or

equal to 30%. Initial calibration is not valid if this criterion is not met. The relative retention times of each compound in each standard run must agree within 0.06 units.

The initial calibration is verified every 12-hour period with a continuing calibration standard containing all target semi-volatile surrogate compounds. RRFs are compared to the average RRF from the initial calibration. The minimum RRF for the target compounds must be met. The percent difference between the initial RRFs and the continuing RRF must be less than or equal to 25 percent for the initial calibration to be valid. Prior to sample analysis, the GC/MS system is evaluated and corrective action taken if these criteria are not met.

4. QC CRITERIA

Table 4.1 - Semi-Volatile Organic Analysis - CLP SOW OLM01.8 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
SVOC, CLP SOW	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 × level in associated samples	Evaluate variability
	Field Duplicate	1 every 10 or fewer field samples (**water**)	≤ 55% RPD	Evaluate data for usability
		1 every 10 or fewer field samples (**soil**)	Not applicable	Evaluate variability

Table 4.2 - Semi-Volatile Organic Analysis - CLP SOW OLM01.8 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOA, CLP SOW	Method Blank	1 per 20 samples of a given matrix or whenever a batch of samples is prepared in a day, whichever is more frequent; see CLP SOW	≤ 5 × CRQL phthalate esters ≤ CRQL	Investigate source; re-extract and reanalyze associated samples
	Matrix spike	1 per 20 samples of a given matrix or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability
	Matrix spike duplicate	1 per 20 samples of a given matrix or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability
	Laboratory Control Sample	1 per 20 samples or a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent	See Table 4.3	Evaluate data for usability
	Surrogate spike	All lab and field samples	See Table 4.3	See CLP SOW
	Instrument performance check	Daily or each 12-hour period, whichever is more frequent	CLP SOW	Retune; reanalyze associated samples
	Calibration	CLP SOW	CLP SOW	Recalibrate before sample analyses
	Calibration check	With every calibration	CLP SOW	Recalibrate
	internal standard	Every standard and sample	CLP SOW	See CLP SOW
	Continuing calibration check	Once each 12-hour period	CLP SOW	Identify source and correct. Recalibrate if source not found and corrected
	Retention time window	CLP SOW	± 0.06 relative retention time units (sample and standard)	See CLP SOW

Table 4.3 - Volatile Organic Analysis CLP SOW OLM01.8 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS						
SVOC, CLP SOW	Phenol	per CLP SOW	per CLP SOW	12-110	26-90	≤42	≤35
	2-Chlorophenol	per CLP SOW	per CLP SOW	27-123	25-102	≤40	≤50
	1,4-Dichlorobenzene	per CLP SOW	per CLP SOW	36-97	28-104	≤28	≤27
	N-nitroso-di-n-propylamine	per CLP SOW	per CLP SOW	41-116	41-126	≤38	≤38
	1,2,4-Trichlorobenzene	per CLP SOW	per CLP SOW	39-98	38-107	≤28	≤23
	4-Chloro-3-methylphenol	per CLP SOW	per CLP SOW	23-97	26-103	≤42	≤33
	Acenaphthene	per CLP SOW	per CLP SOW	46-118	31-137	≤31	≤19
	4-Nitrophenol	per CLP SOW	per CLP SOW	10-80	11-114	≤50	≤50
	2,4-Dinitrotoluene	per CLP SOW	per CLP SOW	24-96	28-89	≤38	≤47
	Pentachlorophenol	per CLP SOW	per CLP SOW	9-103	17-109	≤50	≤47
	Pyrene	per CLP SOW	per CLP SOW	26-127	35-142	≤31	≤36
	Surrogates						
	Nitrobenzene-d5	per CLP SOW	per CLP SOW	35-114	23-120	NA	NA
	2-Fluorobiphenyl	per CLP SOW	per CLP SOW	43-116	30-115	NA	NA
	p-Terphenyl-d14	per CLP SOW	per CLP SOW	33-141	18-137	NA	NA
	Phenol-d5	per CLP SOW	per CLP SOW	10-110	24-113	NA	NA
	2-Fluorophenol	per CLP SOW	per CLP SOW	21-110	25-121	NA	NA
	2,4,6-Tribromophenol	per CLP SOW	per CLP SOW	10-123	19-122	NA	NA
	2-Chlorophenol-d4	per CLP SOW	per CLP SOW	33-110	20-130	NA	NA
	1,2-Dichlorobenzene-d4	per CLP SOW	per CLP SOW	16-110	20-130	NA	NA

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Semi-Volatile Organic Analysis - CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Phenol	10	330
bis(2-Chloroethyl)ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
2,2'-oxybis(1-Chloropropane)*	10	330
4-Methylphenol	10	330
N-nitroso-di-n-dipropylamine	10	330
Hexachloroethane	10	330

Table 5.1 - Semi-Volatile Organic Analysis - CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
bis(2-Chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	10	330
(para-chloro-meta-cresol)	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene[2]	NA	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	25	800
2-Chloronaphthalene	10	330
2-Nitroaniline	25	800
Dimethylphthalate	10	330
Acenaphthylene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	25	800
Acenaphthene	10	330
2,4-Dinitrophenol	25	800
4-Nitrophenol	25	800
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	25	800
4,6-Dinitro-2-methylphenol	25	800
N-nitrosodiphenylamine	10	300
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	25	800
Phenanthrene	10	330
Anthracene	10	330
Carbazole	10	330
Di-n-butylphthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	10	330
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330

Table 5.1 - Semi-Volatile Organic Analysis - CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Di-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330
Additional Compounds		
2-Benzyl-4-chlorophenol	10	330
Benzyl alcohol	10	330
Benzoic acid	50	1600

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

[2] Spike recoveries in water for hexachlorocyclopentadiene from method validation studies have demonstrated that the compound can't be adequately detected by this method

ATTACHMENT A FOR METHOD A-003

Attachment A for Method A-003

Modification to CLP Organic SOW OLM01.8
"Statement of Work for Organic Analysis,
Multi-media, Multi-concentration"

The purpose of this addendum is to outline modifications to the Contract Laboratory Program (CLP) "Statement of Work for Organic Analysis, Multi-media, Multi-concentration" which are project specific to the QAPP prepared by Roy F. Weston, Inc. for investigative activities at the Department of Energy/LANL Mound Plant, Miamisburg, Ohio.

This addendum extends the analysis to include 4-chloro-2-(phenylmethyl)phenol, benzoic acid, and benzyl alcohol for semi-volatiles.

Exhibit A - Summary of Requirements

No modifications to this section.

Exhibit B - Reporting and Deliverables Requirements

Section I: Contract Reports/Deliverables Distribution

No modifications to this section.

Section II: Report Descriptions and Order of Data Deliverables

No modifications to this section.

Section III: Form Instructions

No modifications to this section.

Section IV: Data Reporting Forms:

The following compounds must be added on Form I (Data Sheets).

CAS No.	Semi-Volatiles
120-32-1	4-Chloro-2-(phenylmethyl)phenol
100-51-6	Benzyl Alcohol
65-85-0	Benzoic Acid

Form VI SV-2 (Initial Calibration) and Form VII SV-2 (Continuing Calibration) must be modified to include these additional compounds: 4-Chloro-2-(phenylmethyl)phenol, benzyl alcohol, and benzoic acid..

Exhibit C - Target Compound List (TCL) and Contract Required Quantitation Limits(CRQL)

The following should be added to the Target Compound List (TCL) and Contract required Quantitation Limits(CRQL, Page C-2 and Page C-4):

Analyte	CAS No.	CRQL			On Col. (ng)
		Low Water ug/L	Low Soil ug/kg	Med. Soil ug/kg	
4-Chloro-2-(phenylmethyl)phenol	120-32-1	10	330	10000	20
Benzyl Alcohol	100-51-6	10	330	10000	20
Benzoic Acid	65-85-0	50	1600	50000	100

The following are required CRQLs for residential well samples for TCL semi-volatile organic compounds:

Compound	CRQL µg/L
Semi-Volatiles	
Phenol	5
bis-(2-Chloroethyl)ether	5
2-Chlorophenol	5
2-Methyphenol	5
2,2'-oxybis(1-Chloropropane)	5
4-Methylphenol	5
N-Nitroso-di-n-propylamine	5
Hexachloroethane	5
Nitrobenzene	5
Isophorone	5
2-Nitrophenol	5
2,4-Dimethylphenol	5
bis-(2-Chloroethoxy)methane	5
2,4-Dichlorophenol	5
1,2,4-Trichlorobenzene	5
Napthalene	5
4-Chloraniline	5
Hexachlorobutadiene	5
4-Chloro-3-methylphenol	5
2-Methylnaphthalene	5
Hexachlorocyclopentadiene	5
2,4,6-Trichlorophenol	5

Compound	CRQL µg/L
2,4,5-Trichlorophenol	5
2-Chloronapthalene	5
2-Nitroaniline	20
Dimethylphthalate	5
Acenaphthylene	5
2,6-Dinitrotoluene	5
3-Nitroaniline	20
Acenaphthene	5
2,4-Dinitrophenol	20
4-Nitrophenol	20
Dibenzofuran	5
2,4-Dinitrotoluene	5
Diethylphthalate	5
4-Chlorophenyl-phenylether	5
Fluorene	5
4-Nitroaniline	20
4,6-Dinitro-2-methylphenol	20
N-Nitrosodiphenylamine	5
4-Bromophenyl-phenylether	5
Hexachlorobenzene	5
Pentachlorophenol	20
Phenanthrene	5
Anthracene	5
Di-n-butylphthalate	5
Fluoranthene	5
Pyrene	5
Butylbenzylphthalate	5
3,3'-Dichlorobenzidine	5
Benzo(a)anthracene	5
Chrysene	5
bis-2-Ethyl(hexyl)phthalate	5
Di-n-octylphthalate	5
Benzo(b)fluoranthene	5
Benzo(k)fluoranthene	5
Benzo(a)pyrene	5
Indeno(1,2,3-cd)pyrene	5
Dibenzo(a,h)anthracene	5
Benzo(g,h,i)perylene	5
Benzyl Alcohol	10
Benzoic Acid	50

Exhibit D - Analytical Methods for Semi-Volatiles (SV)

Section I: Introduction

No modifications to this section.

Section II: Sample Preparation and Storage

No modifications to this section.

Section III: Screening of SV organic Extracts

No modifications to this section.

Section IV: GC/MS Analysis of SV

1. Summary of Method: No modifications to this section.

2. Apparatus and Materials: No modifications to this section.

3. Reagents:

3.1 Internal standards - No modifications to this section.

3.2 Calibration standards - 4-Chloro-2-(phenylmethyl)phenol must be added to the calibration standards prepared at 20, 50, 80, 120 and 160 total ng per 2 µL. Benzoic acid must be added to the calibration standard prepared at 50, 80, 120, 160 total ng per 2 µL.

3.3 DFTPP solution - No modifications to this section.

4. Instrument operating Conditions: No modifications to this section.

5. Calibration:

5.1 No modifications to this section.

5.2 and Table 2 Add: 4-Chloro-2-(phenylmethyl)phenol, benzoic acid, and benzyl alcohol must be calibrated using the closest eluting internal standard.

5.3 - 5.5 No modifications to this section.

Table 4 Add:

Parameter	Primary Ion	Secondary Ion (s)
4-Chloro-2-(phneylmethyl)phenol	218	183,165,140
Benzyl Alcohol	108	79,77
Benzoic Acid	122	105,77

5.6.1 No modifications to this section.

5.6.2 Add 4-Chloro-2-(phenylmethyl)phenol, benzoic acid, and benzyl alcohol to the list of compounds. The maximum %RSD must be ± 25 and maximum % Difference ± 30. However, this compound must meet the minimum RRF criteria of 0.01.

These are advisory limits and final limits will be established after method validation.

5.7 - 5.13 No modifications to this section.

6. Sample Analysis: No modifications to this section.

7. Qualitative Analysis: No modifications to this section.

8. Quantitation: No modifications to this section.

9. GC/MS Confirmation of Pesticides and Aroclors: No modifications to this section.

Exhibit E - QA/QC Requirements

I. Overview:

No modifications to this section.

II. Quality Assurance Plan:

No modifications to this section.

III. Standard Operating Procedure:

No modifications to this section.

IV. QA/QC Requirements: Semi-volatile QA/QC requirements

1. GC/MS Mass Calibration and Ion Abundance Patterns:

No modifications to this section.

2. GC/MS Initial Calibration:
Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.

3. Continuing Calibration:

Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.

4. Internal Standards Responses and Retention Times:

No modifications to this section.

5. Method Blank Analysis:

No modifications to this section.

6. System Monitoring Compound Recoveries:

No modifications to this section.

7. Matrix Spike and Matrix Spike Duplicate Analysis:

Reference to Exhibit D includes the modifications to Exhibit D presented in this addendum.

8. Dilution of Samples, MS and MSD

No modifications to this section.

VII Regional Data Review

No modifications to this section.

VIII Laboratory Evaluation Samples

No modifications to this section.

IX GC/MS Tape Audits

No modifications to this section.

X Data Package Audits

No modifications to this section.

XI On Site Laboratory Evaluations

No modifications to this section.

XII Quality Assurance and Data Management

No modifications to this section.

XIII Data Management

No modifications to this section.

Exhibit F - Chain of Custody, Document Control, and Standard Operating Procedures

No modifications to this section.

Exhibit G - Glossary of Terms

No modifications to this section.

Exhibit H - Data Dictionary and Format for Data Deliverables in Computer-Readable Format

No modifications to this section.

ATTACHMENT B FOR METHOD A-003

Attachment B for Method A-003

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-003.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-004

CLP Pesticide Analysis/CLP SOW OLM01.8

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Soil/sediment and water samples will be analyzed for pesticides/PCBs by the CLP SOW Document Number OLM01.8. This method uses gas chromatography for separating and identifying the pesticide/PCB compounds. The capillary columns specified in the method will be used. Attachment A to this method includes an additional preparatory step which must be followed.

1.2 References

EPA 1990a. "U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia, Multi-Concentration." Document No. ILM1.0 including Revisions 1.1 through 1.8. Environmental Protection Agency, March, 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Pesticides/PCB Analysis - CLP SOW OLM01.8
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Pesticides/ PCBs	CLP SOW	Amber glass bottle with Teflon-lined lid	Two 1000 mL bottles	Cool 4°C	7 days extraction/40 days analysis
Soil	Pesticides/ PCBs	CLP SOW	Glass bottle with Teflon-lined lid	100 grams	Cool 4°C	14 days extraction/40 days analysis

3. CALIBRATION

Gas chromatography will be used for analysis of pesticides/PCBs (CLP SOW for organic analysis). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column) or as required in the CLP SOW for pesticide/PCB analysis. A minimum of three external standards for pesticide/PCB analysis of different concentrations are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. A quadratic curve may also be used. Linearity requirements and allowed percentage breakdown of endrin and 4,4'-DDT for pesticide/PCB analysis are presented in the methods.

The CLP SOW for pesticide/PCB analysis requires that the retention times be established and the retention time windows be determined for the target compounds and surrogate compound. The procedures and acceptance criteria are established in the methods.

Performance evaluation mixtures and individual midpoint pesticide/PCB standard mixtures are also analyzed at specified intervals as defined in the CLP SOW. The calibration factor for each standard is established in the methods.

4. QC CRITERIA

Table 4.1 - Pesticides/PCBs Analysis - CLP SOW OLM01.8 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Pesticides/PCBs, CLP SOW	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water) 1 every 10 or fewer field samples (soil)	≤ 35% RPD NA	Evaluate data for usability. Evaluate variability.

Table 4.2 - Pesticides/PCBs Analysis - CLP SOW OLM01.8 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Pesticides/PCBs	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ CRQL; surrogate retention times per CLP SOW	Investigate source; Re-extract and re-analyze associated samples See CLP SOW.
	Sulfur cleanup blank	When portion of samples require sulfur cleanup	≤ CRQL; surrogate retention times per CLP SOW	Investigate source; re-extract and re-analyze associated samples. See CLP SOW.
	Instrument blank	CLP SOW	CLP SOW	See CLP SOW.
	Matrix spike	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix in a case or fewer; see CLP SOW	See Table 4.3	Evaluate data for usability.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Evaluate associated data for usability.
	Surrogate spike	All field and lab samples	See Table 4.3	Evaluate data for usability
	Calibration (initial and continuing)	CLP SOW	CLP SOW	Re-calibrate, see CLP SOW
	GC/MS confirmation	Any sample with a detection from the TCL list for pesticides/PCBs	CLP SOW	See CLP SOW
	Retention time sand Retention time window	CLP SOW	CLP SOW	See CLP SOW

Table 4.3 - Pesticides/PCBs Analysis - CLP SOW OLM01.8 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS						
Pesticides/ PCBs, CLP SOW	Lindane	per CLP SOW	per CLP SOW	56-123	46-127	≤15	≤50
	Heptachlor	per CLP SOW	per CLP SOW	40-131	35-130	≤20	≤31
	Aldrin	per CLP SOW	per CLP SOW	40-120	34-132	≤22	≤43
	Dieldrin	per CLP SOW	per CLP SOW	52-126	31-134	≤18	≤38
	Endrin	per CLP SOW	per CLP SOW	56-121	42-139	≤21	≤45
	4,4'-DDT	per CLP SOW	per CLP SOW	38-127	23-134	≤27	≤50
	Surrogates						
	Tetrachloro-m-xylene	per CLP SOW	per CLP SOW	60-150	60-150	NA	NA
	Decachlorobiphenyl	per CLP SOW	per CLP SOW	60-150	60-150	NA	NA

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Pesticides/PCBs Analysis - CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
α-BHC	0.05	1.7
β-BHC	0.05	1.7
δ-BHC	0.05	1.7
γ-BHC (Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
4,4'-DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
4,4'-DDD	0.10	3.3
Endosulfan sulfate	0.10	3.3
4,4'-DDT	0.10	3.3
Methoxychlor	0.50	17
Endrin ketone	0.10	3.3
Endrin aldehyde	0.10	3.3
α-Chlordane	0.05	1.7
γ-Chlordane	0.05	1.7
Toxaphene	5.0	170
Aroclor-1016	0.50	33
Aroclor-1221	0.50	67
Aroclor-1232	0.50	33
Aroclor-1242	0.50	33

Table 5.1 - Pesticides/PCBs Analysis - CLP SOW OLM01.8 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Aroclor-1248	0.50	33
Aroclor-1254	0.50	33
Aroclor-1260	0.50	33

ATTACHMENT A FOR METHOD A-004

Attachment A for Method A-004

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-004.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-005

CLP Metals/ILM03.0

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Water and soil/sediment samples will be analyzed for the target metals according to the CLP SOW (EPA 1990b). Inductively coupled plasma (ICP) will be used to detect all the TAL metals with the exception of mercury, lithium arsenic, lead, selenium, thallium, and potassium, which will be detected by atomic absorption (AA) see Table 1.1. Additional elements to be detected by ICP are: bismuth, molybdenum and tin. The additional element lithium will be detected by flame AA. Modifications to the method have been prepared as Attachment A to this procedure. ICP metals will also be digested according to EPA Method 200.7 with a fourfold concentration in order to reach lower detection limits for aluminum, antimony, beryllium, and vanadium.

1.2 References

EPA 1990b. "U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multimedia, Multi-Concentration." Document No. ILM1.0 including Revisions 1.1 through 1.8. Environmental Protection Agency, March, 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

Table 1.1 - Method of Analysis for Target Analysis

Target Analyte	Analysis Method
Aluminum	ICP
Antimony	ICP
Arsenic	GFAA
Barium	ICP
Beryllium	ICP
Cadmium	ICP
Calcium	ICP
Chromium	ICP
Cobalt	ICP
Copper	ICP
Iron	ICP
Lead	GFAA
Magnesium	ICP
Manganese	ICP
Mercury	CVAA
Nickel	ICP
Potassium	FAA
Selenium	GFAA
Silver	ICP
Sodium	ICP
Thallium	GFAA
Vanadium	ICP
Zinc	ICP
Molybdenum	ICP
Tin	ICP
Bismuth	ICP
Lithium	FAA

2. PRESERVATION

CLP Metals Analysis - ILM03.0
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Metals	CLP SOW Attachment A	Polyethylene bottle	1000 mL	HNO_3 to pH <2, Cool 4°C	6 months, 28 days (Mercury)
Soil	Metals	CLP SOW Attachment A	Wide-mouth polyethylene bottle	100 grams	Cool 4°C	6 months, 28 days (Mercury)

3. CALIBRATION

TAL metals and four additional elements will be analyzed according to the procedures presented in the CLP SOW for inorganic analyses. Inductively coupled plasma (ICP) and AA instruments are calibrated daily, or once every 24 hours, and each time the instrument is set up. The AA instrument is calibrated with a blank and at least three concentrations of standards prepared each time for analysis. Minimum linearity for AA analysis is a correlation of coefficient of 0.995. The ICP must be calibrated with at least two standards, with one being a blank. The minimum correlation coefficient for cyanide calibration is 0.996.

An initial calibration verification (ICV) is performed to assess the accuracy of the initial calibration using a standard of a certified concentration form an external source. When the measurement exceeds the CLP-established control limits, the problem is corrected, the instrument is re-calibrated, and the ICV is run again. The initial calibration is verified by analysis of a continuing calibration verification (CCV) standard every two hours during an analysis run or at a frequency of 10%, whichever is more frequent (once very five samples for residential well samples for analysis by graphite AA. This standard is also analyzed for at the beginning and end of each sample analysis run. The concentration and source of the CCV and acceptance criteria are specified in the CLP SOW. For ICP and AA CLP analysis, linearity is required near the contract-required detection limit (CRDL). An ICP standard (CRI) at two times the CRDL or two times the instrument detection limit (IDL), whichever is greater, is analyzed for at the beginning and end of each samples analysis ruin or twice per 8-hour working shift, whichever is more frequent, but not before the ICV. An AA standard (CRA) at the CRDL or IDL, whichever is greater, is analyzed for at the beginning of each sample analysis run, but not before the ICV.

4. QC CRITERIA

Table 4.1 - Metals Analysis - ILM03.0
Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Metals , CLP ILM03.0	Field Duplicate	1 every 10 or fewer field samples (water) 1 every 10 or fewer field samples (soil)	≤ 25% RPD NA	Evaluate data for usability. Evaluate variability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.

Table 4.2 - Metals Analysis
Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Metals CLP SOW ILM03.0	Initial and continuing calibration blanks (ICB, CCB)	After every ICV and CCV or 10% or every 2 hours, whichever is more frequent	≤ CRDL	Correct problem; recalibrate; reanalyze preceding 10 samples or all since last good blank.
	Preparation blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent; see CLP SOW	≤ CRDL	If samples results < 10 x CRDL, but >CRDL, redigest and reanalyze.
	Laboratory control sample (LCS)	1 per group of samples in a delivery group or batch, whichever is more frequent.	80-120% recovery	Correct problem; redigest and reanalyze associated samples.
	Initial calibration verification std. (ICV)	CLP SOW	CLP SOW	See CLP SOW
	Continuing calibration verification (CCV)	CLP SOW	CLP SOW	See CLP SOW
	Linear range check standard (CRI, CRA) (ICP and AA only)	CLP SOW	Not established	None.
	Interference check samples (ICS) (ICP only)	Sample twice per 8-hour shift, or at beginning and end of analysis run, whichever is more frequent.	±20% of true value	Correct problem; recalibrate reanalyze samples since last good ICS.
	ICP Serial dilution (L) (ICP only)	1 per group of samples of a given matrix, concentration, or each delivery group, whichever is more frequent.	If result > 50 x IDL: ± 10% difference	Evaluate data for usability.
	Spike sample (S)	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	75-125% recovery	Evaluate data for usability.
	Sample dup.(D) (sample replicate)	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	If result ≥ 5 x CRDL ±20% RPD; f result ≤ 5 x CRDL: ± CRDL	Evaluate data for usability.
	Method of std. Addition for AA only (MSA)	CLP SOW	CLP SOW	See CLP SOW
	Linear range analysis (LRA) (for ICP only)	CLP SOW	CLP SOW	Reanalyze

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - CLP Metals - ILM03.0
Target Analyte List

Analyte	Water (µg/L)	Soil (mg/kg)
Aluminum	20	4
Antimony	10	2
Arsenic	10	2
Barium	200	40
Beryllium	1	0.2
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	0.6
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.1
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	10	2
Zinc	20	4
Additional Elements		
Molybdenum	20	2
Tin	50	10
Bismuth	150	30
Lithium	100	10

ATTACHMENT A FOR METHOD A-005

Attachment A for Method A-005

Contract Laboratory Program Statement Of Work Modifications

Modification to CLP SOW ILM03.0
"Statement of Work for Inorganic Analysis, Multi-media, Multi-concentration"

The purpose of this addendum is to outline modifications to the Contract Laboratory Program (CLP) Statement of Work (SOW) ILM03.0, "Statement of Work for Inorganic Analysis, Multi-media, Multi-concentration", which are project-specific to the QAPP prepared by Roy F. Weston, Inc. for investigative activities at the Department of Energy/LANL Mound Plant, Miamisburg, Ohio.

This addendum extends the analysis to include lithium, molybdenum, bismuth, and tin and requires lower detection limits for aluminum, antimony, beryllium, and vanadium. Molybdenum, bismuth, and tin must be analyzed by ICP.

Exhibit A - Summary of Requirements

No modifications

Exhibit B - Reporting and Deliverables Requirements

Section I: Contract Reports/Deliverable Distribution
No Modifications

Section II: Report Descriptions and Order of Data Deliverables
No Modifications

Section III: Form Instruction Guide
No Modifications

Section IV: Data Reporting Forms

The following elements have been added to the CLP SOW by this addendum:

CAS No.	Analyte
7439-93-2	Lithium
7439-98-7	Molybdenum
7440-69-9	Bismuth
7440-31-5	Tin

These four elements and the lower CRDLs must be added to the following forms:

Form	Description
I	Data Sheet
II (A)	Initial and Continuing Calibration Verification
II (B)	CRDL Standard for AA and ICP
III	Blanks
IV	ICP Interference Check Sample

V (A)	Spike Sample Recovery
V (B)	Post Digest Spike Sample Recovery
VI	Duplicates
VII	Laboratory Control Sample
IX	ICP Serial Dilutions
X	Instrument Detection Limit (Quarterly)
XI (A)	ICP Interelement Correction Factors (Annually)
XI (B)	ICP Interelement Correction Factors (Annually)
XII	ICP Linear Ranges (Quarterly)
XIV	Analysis Run Log

Exhibit C - Inorganic Target Analyte List (TAL)

This table is modified to include the following additional elements with the estimated CRDL values:

Analyte	Water CRDL (ug/L)	Soil CRDL (mg/kg)
Lithium	100	10
Molybdenum	20	2
Bismuth	150	30
Tin	50	10

Lower CRDLs are required for the following elements:

Analyte	Water CRDL (ug/L)	Soil CRDL (mg/kg)
Aluminum	20	4
Antimony	10	2
Beryllium	1	0.2
Vanadium	10	2

Exhibit D - Analytical Methods

Section I: Introduction
No Modifications

Section II: Sample Preservation and Holding Times
No Modifications

Section III: Sample Preparation

A. WATER SAMPLE PREPARATION

1. Acid Digestion Procedure for Furnace Atomic Absorption Analysis
No Modifications

2. Acid Digestion Procedure for ICP and Flame AA Analyses
A four-fold concentration of the sample or the use of 4 grams of sample instead of 1 gram is necessary to meet required detection limits for aluminum, antimony, beryllium, and vanadium. These metals are designated for analysis by ICP. This four-fold concentration preparation is detailed in Method 200.7 with revision 1.3 (1987) in the "Methods of Chemical Analysis of Water and Waste" (EPA-600/4-79-020). Briefly, one mL of (1+1) HNO_3 and five mL of (1+1) HCl is added to a 200 mL aliquot of the sample. The sample is digested until the volume is reduced to approximately 20 mL. When cool, the digestate is transferred to a 50-mL volumetric flask and brought up to volume with deionized distilled water. Analyte recovery data and sample preparation bias for these elements shall be evaluated prior to implementation of this technique.

B. SOIL/SEDIMENT PREPARATION

1. Acid Digestion Procedure for ICP, Flame AA, and Furnace AA Analyses

The laboratory is required to meet the required detection limits for aluminum, antimony, beryllium and vanadium. This may be accomplished through concentration of the sample (up to four-fold) or through digestion of up to 4 grams of soil instead of 1 gram. All analytes must be run within the linear range of the instrument.

C. TOTAL METALS SAMPLE PREPARATION USING MICROWAVE DIGESTION

Not Applicable

Section IV: Sample Analysis

Part A - Inductively Coupled Plasma - Atomic Emission Spectrometric Method

1.0 Scope and Application

1.1 No modification.

1.2 No modification.

1.3 Table I is modified to include:

Element	Wavelength (nm)	Estimated Detection Limit (ug/L)
Molybdenum	202.030	20
Bismuth	223.061	150
Tin	189.989	50
Aluminum	-	20
Antimony	-	10
Beryllium	-	1
Vanadium	-	10

1.4 No modification.

2.0 Summary of Method
No modification.

3.0 Definitions
No modifications.

4.0 Safety
No modifications.

5.0 Interferences

Table 2 contains information regarding molybdenum. No information is available at this time for lithium, bismuth, and tin and will be evaluated before sample analysis is conducted.

6.0 Apparatus
No modifications.

7.0 Reagents and Standards
7.1 No modifications.
7.2 No modifications.
7.3 Stock standard solutions - Modified to include:
7.3.26 Bismuth solution, stock, 1 mL = 100 ug Bi:

Dissolve 0.1000 g of bismuth metal in a minimum amount of (1+1) HNO_3. Dilute to 1000 mL with deionized, distilled water.

7.3.27 Tin solution, stock, 1 mL = 100 ug Sn:

Dissolve 0.1000 g of tin metal in 100 mL of conc. HCl and dilute to 1000 mL with deionized, distilled water. This standard is prepared fresh weekly.

7.4 Mixed calibration standard solutions - Modify to include:
7.4.6 Mixed standard solution VI - Lithium, bismuth, and tin.
7.5 No modifications.
7.6 Instrumental and calibration check standards must include all analytes of interest in Table 1.

8.0 Procedure
No modifications.

9.0 Calculation
No modifications.

10.0 Quality Control (Instrumental)
No modifications.

Part B - Atomic Absorption Methods, Furnace Techniques

If tin is analyzed by GFAA, the peroxide used for digestion must be verified by the laboratory to be free of tin contamination.

Part C - Atomic Absorption Methods, Flame Techniques

Lithium will be analyzed by SW7430; "Test Methods for Evaluating Solid Wastes," U.S. EPA. Quality control must be implemented as required by CLP SOW for Flame AA analyses and outlined in QAPP Table III.2.

Part D - Cold Vapor Methods for Mercury Analysis

No Modifications

Part E - Methods for Cyanide Analysis

No Modifications

Part F - Percent Solids Determination Procedure

No Modifications

Part G - Alternate Methods (Catastrophic ICP Failure)

Bi method 3500-Bi; "Standard Methods for Analysis of Water and Waste Waters," 17th edition
Sn method 282.2; "Methods for Chemical Analysis of Water and Wastes," U.S. EPA EPA 600/4-79-020, March 1983.
Mo method 246.1; "Methods for Chemical Analysis of Water and Wastes," U.S. EPA EPA 600/4-79-020, March 1983.

Exhibit E - Quality Assurance/Quality Control Requirements

Section I - General QA/QC Procedures
No Modifications
Section II - Specific QA/QC Procedures
No Modifications
Section III - Quality Assurance Plan
No Modifications
Section IV - Data Management
No Modifications
Section V - Required QA/QC Operations

1.0 Instrument Calibration

All analytes from Table 1 must be included in calibration standards.

2.0 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

All analytes from Table 1 must be included. If sample pre-concentration is to be utilized, analyte recovery data and sample preparation bias for these elements shall be evaluated and found to be acceptable prior to the implementation of this technique.

3.0 CRDL Standards for ICP (CRI) and AA (CRA)

All analytes from Table 1 must be included.

4.0 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

References to Exhibit C includes the modifications to Exhibit C presented in this addendum.

5.0 ICP Interference Check Sample (ICS) Analysis

Table 2 is modified to include the following analytes in solution AB:

Element	mg/L
Molybdenum	1.0
Bismuth	1.0
Tin	1.0

6.0 Spike Sample Analysis (S)

Table 3 is modified to include:

Element	ICP/Flame AA		Furnace AA	
	Water (ug/L)	Soil (mg/kg)	Water (ug/L)	Soil (mg/kg)
Lithium	2000	500	-	-
Molybdenum	300	200	-	-
Bismuth	2000	500	-	-
Tin	500	200	100	50

7.0 Duplicate Sample Analysis (D)
Reference to Exhibit C includes modifications to Exhibit C presented in this addendum.

8.0 Laboratory Control Sample (LCS) Analysis
All of the four new elements (Li, Mo, Sb, Sn) will be included in the LCS analysis.

9.0 ICP Serial Dilutions Analysis (L)
No modifications.

10.0 Instrument Detection Limit (IDL) Determination
Reference to Exhibit C includes the modifications to Exhibit C present in this addendum.

11.0 Interelement Corrections for ICP
ICS A and AB solutions for titanium must be monitored to insure adequate interelement correction factors between all elements.

12.0 Linear Range Analysis (LRA)
No modifications.

13.0 Furnace Atomic Absorption (AA) QC Analyses
No modifications.

Section VI - Laboratory Evaluation Process
No Modifications

Exhibit F - Chain-of-Custody, Document Control, and Standard Operating Procedures

No Modifications

Exhibit G - Glossary of Terms

No Modifications

Exhibit H - Data Dictionary and Format for Data Deliverables in Computer-Readable Format

The four additional elements addressed by this addendum must be included on all electronic deliverables.

ATTACHMENT B FOR METHOD A-005

Attachment B for Method A-005

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-005.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-006

Cyanide/CLP SOW ILM03.0

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Cyanide Analysis Description

Cyanide will be analyzed according to the CLP SOW ILM03.0 for water and soil samples. Soil samples must be prepared per Attachment A. This method uses spectrophotometry. The required detection limits for cyanide are 10µg/L for water and 2 mg/kg for soil.

1.2 References

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

EPA. 1996. "U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multi-media, Multi-Concentration." Document No. ILM03.0. U.S. Environmental Protection Agency, March 1990.

2. PRESERVATION

Cyanide Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Cyanide	CLP SOW	Polyethylene bottle	1500 mL	NaOH to pH ≥ 12 Cool 4°C	14 days
Soil	Cyanide	CLP SOW	Wide-mouth polyethylene bottle	100 grams	Cool 4°C	14 days

[1] ther analyses have been listed to ensure field personnel know which analyses can be taken from the same container.

3. CALIBRATION

Cyanide will be analyzed according to the procedures presented in the CLP SOW for inorganic analyses. The spectrophotometer is calibrated daily, or once every 24 hours, and each time the instrument is set up. The minimum correlation coefficient for cyanide calibration is 0.996.

A initial calibration verification (ICV) is performed to assess the accuracy of the initial calibration using a standard of a certified concentration from an external source. When the measurement exceeds the CLP-established control limits, the problem is corrected, the instrument is re-calibrated and the ICV is run again. The initial calibration is verified by analysis of a continuing calibration verification (CCV) standard every two hours during an analysis run or at a frequency of 10%, whichever is more frequent. This standard is also analyzed for at the beginning and end of each sample run. The concentration and source of the CCV and acceptance criteria are specified in the CLP SOW.

4. QC CRITERIA

Table 4.1 - Cyanide Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Cyanide, CLP SOW ILM03.0	Duplicate	1 every 10 or fewer field samples (**water**) 1 every 10 or fewer field samples (**soil**)	≤ 25% RPD Not applicable	Evaluate data for usability. Evaluate variability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 × level in associated samples	Evaluate potential sources; Evaluate associated data for usability.

Table 4.2 - Cyanide Analysis CLP SOW ILM03.0 aboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Cyanide, CLP SOW ILM03.0	Inital and continuing calibration blanks (ICB,CCB)	After every ICV and CCV or 10% or every 2 hours, whichever is more frequent.	≤ CRDL	Correct problem; recalibrate; reanalyze preceding 10 samples or all since last good blank.
	Spike sample (S)	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	75-125% Recovery	Evaluate data for usability.
	Sample duplicate (D)	1 per group of samples of a given matrix, concentration, or sample delivery group, whichever is more frequent.	If result ≥ 5 × CRDL: ± 20% RPD If result ≤ 5 × CRDL: ± CRDL	Evaluate data for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Cyanide - CLP SOW ILM03.0 Target Analyte List

Analyte	Water (µg/L)	Soil (mg/kg)
Cyanide	10	2

ATTACHMENT A FOR METHOD A-006

Attachment A for Method A-006

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-006.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-007

General Chemistry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

1.1.1 *Chloride, Nitrate-Nitrite, Sulfate, Ammonia, Fluoride and Total Phosphorus*

Soil/sediment or water samples will be analyzed for chloride, nitrate-nitrite, fluoride, and sulfate. Water samples will also be analyzed for ammonia and total phosphorus. Analyses will be performed by the methods specified in Table 1.1. The laboratory will perform only one of the identified methods in Table 1.1 per analyte. Soil/sediment samples will be extracted with deionized water for the dissolution of the desired anions prior to analysis, according to the statement of work in Attachment B. Soil detection limits are based on a 10-gram soil samples, 10 ml or extractant, and a soil moisture content between 0 and 10 percent. The actual detection limit will vary depending upon these variables.

Table 1.1 General Chemistry Method of Analysis

Analyte	Water Method	Soil Method
Nitrate-Nitrite	E353.2[a]	E353.2
Chloride	E325.1/E325.2[a]	SW9250/SW9251[a]
Sulfate	E375.2[a]	E375.2[b]
Nitrite	E354.1[a]	NA
Fluoride	E340.2[a]	E340.2[a]
Ammonia	E350.1[a]	NA
Total Phosphorus	E365.1[a]	NA
Total Nitrogen	E351.3[a]	NA

[a] Methods of Chemical Analysis of Water and Wastes
[b] Test Methods for Evaluating Solid Waste

1.1.2 Total Kjeldahl Nitrogen

Groundwater and surface water samples will be analyzed for total Kjeldahl nitrogen by the method specified in Table 1.1. Analysis consists of converting nitrogen to ammonia, then detecting the ammonia by colorimetry using Nesslerization.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

EPA. 1993. "Methods for Chemical Analysis of Water and Wastes." U.S. Environmental Protection Agency, EPA - 600/4-79-020. March 1983.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

General Chemistry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Sulfate Chloride	E375.2; E325.1/325.2/ SW9250/9251	Polyethylene bottle	500 mL	Cool 4°C	28 days
	Nitrite	E354.1	Polyethylene bottle	150 mL	Cool 4°C	48 hours
	Nitrate-Nitrite	E353.2	Polyethylene bottle	500 mL	H_2SO_4 to pH <2 Cool 4°C	28 days
	Fluoride	E340.2	Polyethylene bottle	500 mL	Cool 4°C	28 days
	Ammonia	E350.1/350.3	Polyethylene bottle	500 mL	Cool 4°C H_2SO_4 to pH <2	28 days
	Total Nitrogen Total Phosphorus	E351.3 E365.1	Polyethylene bottle	500 mL	H_2SO_4 to pH <2 Cool 4°C	28 days
Soil	Fluoride	E340.2	Wide-mouth polyethylene bottle	50 grams	Cool 4°C	28 days
	Nitrate-Nitrite Chloride Sulfate	E353.2 SW9250-9251/ 325.1/325.2 E375.2	Wide-mouth polyethylene bottle	100 grams	Cool 4°C	28 days

3. CALIBRATION

Nitrate/nitrite, sulfate, chloride, total nitrogen, nitrite, ammonia, and total phosphorus will be analyzed for by spectrophotometric methods, which use a colorimeter to identify the analyte when the analyte is complexed with, or created the formation of, a light-absorbing compound. Fluoride will be detected using an ion selective electrode. Calibration of the colorimeter and electrode is accomplished with a minimum of three concentrations of standards and is performed when instrument conditions are changed or when the calibration standard exceeds acceptance criteria. The calibration curve is plotted or a minimum correlation coefficient of 0.995 is required for acceptable linearity of the resulting calibration curve.

The initial calibration is verified with the analysis of a midrange calibration standard prior to sample analysis and for every 20 samples analyzed. The standard result must be less than or

equal to a 15% difference from the response of the initial calibration. If this acceptance criterion is exceeded, then the instrument is recalibrated.

4. QC CRITERIA

Table 4.1 - General Chemistry Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Chloride Nitrate-Nitrite Sulfate	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
Total nitrogen Total phosphorus Nitrite	Field Duplicate	1 every 10 or fewer field samples (water)	≤25% RPD	Evaluate data for usability
Fluoride Ammonia		1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability

Table 4.2 - General Chemistry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Chloride Nitrate-Nitrite Sulfate Total nitrogen Total phosphorus Nitrite Fluoride Ammonia	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Correct problem; reanalyze.
	Calibration (3 points) and Reagent blank	When instrument conditions change or when calibration check criteria exceeded.	Correlation coefficient ≥0.995 or plot curve for nonlinear analytes	Recalibrate
	Calibration check	Prior to sample analysis and one per 20 samples analyzed	± 15% of initial calibration responses	Identify and correct problem; recalibrate
	Matrix spike	1 per 20 samples of a given matrix	75-125% Recovery	Evaluate data for useability
	Matrix spike duplicate	1 per 20 samples of a given matrix	≤ 20% RPD	Evaluate data for useability
	Laboratory Control Sample (chloride, nitrate)	1 for each calibration	Vendor specification	Evaluate data for useability

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 General Chemistry Analysis Target Analyte List

Analyte	Water (mg/L)	Soil (mg/kg)
Nitrate-Nitrite[1]	0.2	2
Chloride[1]	1.0	5
Sulfate[1]	5	50
Fluoride[1]	0.1	2.5
Nitrite	0.01	NA
Ammonia	0.1	NA
Total Nitrogen	0.1	NA
Total Phosphorus	0.1	NA

[1] Attachment A and B include descriptions of required method modifications

ATTACHMENT A FOR METHOD A-007

Attachment A for Method A-007

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-007.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

ATTACHMENT B FOR METHOD A-007

Attachment B for Method A-007

Statement of Work for
Soil Preparation for Anion Analysis

1.0 Scope and Application

This procedure is used to prepare soil samples for anion analysis. Only water soluble anions can be analyzed from soils prepared by this method.

2.0 Summary of Method

Reagent water is added to the soil sample and used to leach the anions from the soil. The water leachate is then analyzed for analyte of interest.

3.0 Interferences

None

4.0 Apparatus and Equipment

4.1 Bottle, plastic
4.2 Spatula
4.3 Electronic Balance (minimum accuracy ± 0.01)
4.4 Stir Bar
4.5 Electronic Stirrer
4.6 Ultrasonic bath

5.0 Reagents/Supplies

5.1 Water, ASTM Type II

6.0 Sample Collection, Preservation, and Handling

See Section 2.0 of Method A-007.

7.0 Sample Preparation Procedure

7.1 Determine the percent moisture in the sample per CLP SOW Document No. OLM01.8.
7.2 Take a representative soil aliquot as described in SOW-003 and weigh 20 g of soil into a 250 mL plastic bottle. (If more than 200 mL of leachate is required for the tests, then use the ratio of 1:10 (soil to water) to generate the needed amount of leachate).

7.3 Add 200 mL of water to the bottle, a magnetic stir bar, and then mix the slurry for 10 minutes.

7.4 Sonicate the water/soil mixture for 10 minutes.

7.5 Filter the slurry and the filtrate is ready for analysis. Results to be reported on dry weight basis.

8.0 Quality Control

8.1 See Method A-007.

9.0 References

"The Determination of Inorganic Ions by Ion Chromatography," USEPA Method 300.0.

MOUND

Environmental
Restoration
Program

Method: A-008

Total Dissolved Solids/ Total Suspended Solids

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) for groundwater and surface water samples will be analyzed according to EPA Methods 160.1 and 160.2, respectively.

1.2 References

EPA. 1993. "Methods for Chemical Analysis of Water and Waste," U.S. Environmental Protection Agency, EPA - 600/4-79-020 revised March 1983.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Total Dissolved Solids/Total Suspended Solids Analysis Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Total Dissolved Solids	E160.1	Polyethylene bottle	1000 mL	Cool 4°C	7 days
	Total Suspended Solids	E160.2	Polyethylene bottle	1000 mL	Cool 4°C	7 days

3. CALIBRATION

Calibration is not specifically addressed in the Remedial Investigation/Feasibility Study, Operable Unit 9, Site-Wide Quality Assurance Project Plan for these analyses and is not included in this procedure.

4. QC CRITERIA

Table 4.1 - Total Dissolved Solids/Total Suspended Solids Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Total Dissolved Solids (TDS)	Field Duplicate	1 every 10 or fewer field samples (water)	≤25% RPD	Evaluate data for usability
Total Suspended Solids (TSS)	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate associated data for usability.

Table 4.2 - Total Dissolved Solids/Total Suspended Solids Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Total Dissolved Solids (TDS) Total Suspended Solids (TSS)	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Identify and correct problem; Reanalyze blank.
	Replicate sample	1 per 20 samples analyzed	≤20% RPD	Reanalyze a replicate sample; Report both results.
	Laboratory control sample (LCS)	1 per 20 samples analyzed	80-120%	Identify and correct problem.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - TDS/TSS Analysis Target Analyte List

Analyte	Water (mg/L)	Soil (mg/kg)
Total Dissolved Solids	4	NA
Total Suspended Solids	10	NA

Method: A-009

Total Organic Carbon

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Soil/sediment samples and groundwater/surface water samples will be analyzed for total organic carbon (TOC), using EPA Methods 415.1 or 415.2. Analysis consists of converting organic carbon to carbon dioxide, which is detected by a nondispersive infrared detector. Soil/sediment samples undergo a pyrolysis to release the carbon dioxide to be detected. The soil preparation procedure is described in a statement of work in Attachment B.

1.2 References

EPA. 1983. "Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, EPA - 600/4-79-020, March 1983.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Total Organic Carbon Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Total Organic Carbon	E415.1/E415.2	Amber glass bottle with Teflon-lined lid (no headspace)	250 mL	H_2SO_4 to pH ≤ 2 Cool 4°C	28 days
Soil	Total Organic Carbon	E415.1/E415.2	Amber glass jar with Teflon-lined lid	50 grams	Cool 4°C	28 days

3. CALIBRATION

3.1 Total Organic Carbon (TOC) Analysis

Total organic carbon (TOC) is analyzed using a spectrophotometer with an infrared detector. The TOC analyzer is calibrated with a single concentration standard. The initial calibration is verified with the analysis of a midrange calibration standard prior to sample analysis and for every 20 samples analyzed. The mid-range standard result must be less than or equal to 15% difference from the response of the initial calibration. If this acceptance criterion is exceeded, then the instrument is recalibrated.

4. QC CRITERIA

Table 4.1 - Total Organic Carbon Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Total Organic Carbon	Field Duplicate	1 every 10 or fewer field samples (water) 1 every 10 or fewer field samples (soil)	≤35% RPD Not Applicable	Evaluate data for usability. Evaluate variability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate associated data for usability.

Table 4.2 - Total Organic Carbon Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Total Organic Carbon	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Correct problem; Reanalyze blank.
	Calibration	When instrument conditions change or when calibration check criteria exceeded.	Second reading must be within 25% of initial	Recalibrate
	Calibration check	1 per 20 samples analyzed	±15% of initial calibration response	Recalibrate
	Matrix spike (MS)	1 per 20 samples of a given matrix.	75-125% recovery	Evaluate data for usability.
	Matrix spike duplicate (MSD)	1 per 20 samples of a given matrix.	≤20% RPD	Evaluate data for usability.
	Replicate sample	**4 analyses for every sample.**	≤25% RSD	Reanalyze aqueous samples once. Evaluate soil samples considering inhomogeneity. Reanalyze aqueous samples is no matrix effects.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Total Organic Carbon Analysis Target Analyte List

Analyte	Water (µg/L)	Soil (mg/kg)
Total Organic Carbon[1]	1	25

[1] Attachment A and B includes modifications to this analysis method.

ATTACHMENT A FOR METHOD A-009

Attachment A for Method A-009

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-009.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

ATTACHMENT B FOR METHOD A-009

Attachment B for Method A-009

Statement of Work
Total Organic Carbon Soil Preparation

1.0 Scope and Application

This procedure describes the process of preparing a soil for Total Organic Carbon (TOC) analysis.

The percent moisture will be determined prior to preparing the sample to allow the results to be reported on a dry-weight basis.

2.0 Summary of Method

The sample is prepared by reacting inorganic carbon with phosphoric acid. This reaction causes the inorganic carbon to be driven from the sample. The treated soil is then ready for analysis in a combustion tube by EPA 415.1 or EPA 415.2.

3.0 Interferences

This preparation procedure has a minor potential to drive off some organic carbon as well as the inorganic carbon causing the reported result to be biased low.

4.0 Apparatus and Equipment

4.1 Platinum Weigh Boat

4.2 Hot plate

4.3 Electronic Balance (accurate to $\pm$ 0.0001 g)

5.0 Reagents/Supplies

5.1 Spatula

5.2 Hotplate (75° C)

5.3 Combustion tube

5.4 Phosphoric Acid, 50%

6.0 Sample Collection, Preservation, and Handling

6.1 See Section 2.0 of Method A-009.

7.0 Sample Preparation Procedure

7.1 Determine the percent moisture in the sample per CLP SOW Document No. OLM018.

7.2 A representative sample aliquot will be taken as described in SOW-003. Weigh 1-2 mg of sample into a platinum boat and record the weight.

7.3 Add 0.05 mL (one drop) phosphoric acid to the soil and heat the boat to 75° C on a hot plate for about 30 minutes (near dryness). This procedure drives off inorganic carbon (carbonates) from the sample. Volatile organics will also be driven off during this procedure.

7.4 Place the treated soil into a combustion tube assembly for pyrolysis. The sample can now be analyzed as normal at 800° C. (EPA method 415.1 or 415.2).

7.5 The determined TOC result must be reported on a dry weight basis, as given by:

$$\frac{\text{TOC result (mg/Kg)}}{1 - (\%\ \text{moisture}/100)} = \text{TOC result (dry weight, mg/Kg)}$$

8.0 Quality Control

8.1 See OU9 Site-Wide QAPP, most recent version

9.0 References

None.

MOUND

Environmental
Restoration
Program

Method: A-010

Explosives Analysis by USEPA Method 8330

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Both soils/sediments and water samples will be analyzed for ten SW8330 explosives and PETN using high performance liquid chromatography (HPLC). Analysis will be performed according to laboratory SOPs which are based on USEPA SW846, Method 8330 (EPA 1990). Second column confirmation will be performed if positive results are obtained on the primary column. PETN will be detected at a different wavelength (220 nm) on a separate analytical run. A statement of work for analysis of PETN is provided in Attachment B describing the required variation from method 8330.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Explosives Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Explosives	SW8330	Amber glass bottle with Teflon-lined lid	1 liter	Cool 4°C	7 days extraction/ 40 days analysis
Soil	Explosives	SW8330	125-mL wide-mouth amber glass jar with Teflon-lined lid.	100 grams	Cool 4°C	14 days extraction/ 40 days analysis

3. CALIBRATION

3.1 High Performance Liquid Chromatography (HPLC)

High-performance liquid chromatography (HPLC) is used for analysis of explosives. Initial calibration is performed with a minimum of five concentrations of standards whenever there is change in chromatographic conditions or when the check standard is outside acceptance criteria. The resulting calibration curve must have an average response factor with a relative standard deviation less than or equal to 20%.

The initial calibration is checked prior to sample analysis and once every 10 samples analyzed with a midrange standard for each analyte. The response of the check standard must be within 15% of the predicted response in order for the initial calibration to be valid. If the calibration check is outside this criteria, a new calibration curve will be performed. The retention times and peak heights of the check standard for every 10 samples are compared to those of the check standard run at the beginning of the day. If significant deviation or visible chromatographic abnormalities are observed, then all samples analyzed after the last acceptable standard check will be reanalyzed.

4. QC CRITERIA

Table 4.1 - Explosives Analysis - EPA Method SW8330 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Explosives	Field Duplicate	1 every 10 or fewer field samples (**water**) 1 every 10 or fewer field samples (**soil**)	≤35% RPD Not Applicable	Evaluate data for usability. Evaluate variability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (**water**)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.

Table 4.2 - Explosives Analysis - EPA Method SW8330 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Explosives	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Reanalyze blank.
	Calibration	(5 pt.) when calibration check limit criteria exceeded.	≤20% RSD	Recalibrate
	Surrogate Spike	All lab and field samples	See Table 4.3	Reanalyze
	Matrix spike (MS)	1 per 20 samples of a given matrix.	See Table 4.3	Evaluate data for usability
	Matrix spike duplicate (MSD)	1 per 20 samples of a given matrix.	See Table 4.3	Evaluate data for usability
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Evaluate data for usability
	Retention time window	With every calibration check	Column and Compound Specific	Identify source, correct problem; reanalyze samples since last good calibration check
	Calibration check	Prior to sample analysis and 1 per 10 samples analyzed.	±16% of peak height of initial calibration	Recalibrate
	Secondary column confirmation	Every positive detection ≥ PQL	Not applicable	Evaluate positive identification of analyte.

Table 4.3 - Explosives Analysis- EPA Method SW8330 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
	Matrix Spike/LCS (low concentration)						
Explosives	RDX	11.6	N/A	62-87	N/A	32	N/A
	1,3,5-TNB	28	N/A	85-100	N/A	19	N/A
	2,4,6-TNT	5.8	N/A	78-102	N/A	29	N/A
	2,6-DNT	1.0	N/A	66-102	N/A	45	N/A
	2,4-DNT	0.8	N/A	74-99	N/A	31	N/A
	Matrix Spike/LCS (high concentration)						
	RDX	140	13	85-108	40-160	20	30
	1,3,5-TNB	29	13	83-104	40-160	19	30
	2,4,6-TNT	5.0	13	74-96	40-160	19	30
	2,6-DNT	4.0	3.0	77-100	40-160	20	30
	2,4-DNT						
	Surrogates						
	4-Nitrotoluene	4.0	25	50-150	50-150	N/A	N/A
	4-Nitroglycern	*	*	50-150	50-150	N/A	N/A

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Explosives Analysis Target Analyte List

Analyte	Water (µg/L)	Soil (mg/kg)
HMX	20	3.0
RDX	6.0	2.5
NB	15	1.5
1,3-DNB	15	1.5
1,3,5-TNB	15	1.5
2,4-DNT	0.5	0.5
2,6-DNT	0.5	1.5
TNT	3.0	1.5
2A,4,6-DNT	3.0	1.5
Tetryl	3.0	2.5
PETN	1	1

ATTACHMENT A FOR METHOD A-010

Attachment A for Method A-010

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-010.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

ATTACHMENT B FOR METHOD A-010

Attachment B for Method A-010

Statement of Work for
PETN Water Extraction and Analysis
Using EPA Method 8330

1.0 Scope and Application

This procedure describes the required modification to extract and analyze pentaerythritol tetranitrate (PETN) from water by EPA Method 8330, (EPA 1990). This procedure also describes the surrogates required for the analysis of explosives.

2.0 Summary of Method

No change.

3.0 Interferences

No change.

4.0 Apparatus and Equipment

No change.

5.0 Reagents/Supplies

5.2.19 PETN - Reagent Grade
5.2.20 4-Nitrobutene - Reagent Grade
5.4.3 Surrogate Spiking Solution
Prepare a 2000 µg/mL solution of 4-nitrotoluene and 4-nitrobutene in acetonitrile. 50 µL of solution will be added to each sample.

6.0 Sample Collection, Preservation, and Handling

No change.

7.0 Sample Analysis Procedure

7.1 Instrument Conditions

Primary HPLC Column

Column:	LC-18
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.

Detector:	UV 220
Injection Size:	100 µl loop

Confirmation HPLC Column

Column:	LC-CN
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

8.0 Quality Control

8.7 Two surrogates, 4-nitrotoluene and 4-nitrobutene, are added to each sample.

8.8 A control spike (blank spike) is required for every 20 samples extracted or each batch of samples, whichever is more frequent.

9.0 References

No change.

Statement of Work for
PETN Soil Extraction and Analysis
Using EPA Method 8330

1.0 Scope and Application

This procedure describes the required modification to extract and analyze pentaerythritol tetranitrate (PETN) from soil and other solid matrices by EPA Method 8330, (EPA 1990). This procedure also describes the surrogates required for the analysis of explosives.

2.0 Summary of Method

No change.

3.0 Interferences

No change.

4.0 Apparatus and Equipment

No change.

5.0 Reagents/Supplies

5.2.19 PETN - Reagent Grade
5.2.20 4-Nitrobutene - Reagent Grade
5.4.3 Surrogate Spiking Solution
Prepare a 2000 µg/mL solution of 4-nitrotoluene and 4-nitrobutene in acetonitrile. 12.5 µL of solution will be added to each sample.

6.0 Sample Collection, Preservation, and Handling

No change.

7.0 Sample Analysis Procedure

7.1 Instrument Conditions

Primary HPLC Column

Column:	LC-18
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

Confirmation HPLC Column

Column:	LC-CN
Mobile Phase:	1:1 Methanol/Water
Flow:	1.5 ml/min.
Detector:	UV 220
Injection Size:	100 µl loop

8.0 Quality Control

8.7 Two surrogates, 4-nitrotoluene and 4-nitrobutene, are added to each sample.

8.8 A control spike (blank spike) is required for every 20 samples extracted or each batch of samples, whichever is more frequent.

9.0 References

No change.

MOUND

Environmental
Restoration
Program

Method: A-011

Alkalinity

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Alkalinity will be measured using the tritrimetric method in EPA Method 310.1. Carbonate and bicarbonate species will be calculated based on the measurement and the pH of the sample and by making assumptions on alkalinity relationships.

1.2 References

EPA. 1983 "Methods for Chemical Analysis of Water and Wastes, " U.S. Environmental Protection Agency, EPA 600/4-79-020, March 1983.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Alkalinity Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Alkalinity	E310.1	Polyethylene bottle	200 mL	Cool 4°C	14 days

3. CALIBRATION

3.1 Alkalinity

The remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan doesn't specifically address calibration, and therefore is not included in this section.

4. QC CRITERIA

Table 4.1 - Alkalinity Analysis
Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Alkalinity	Field Duplicate	1 every 10 field samples	≤35% RPD	Evaluate data for usability.

Table 4.2 - Alkalinity Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Alkalinity	Method blank	1 per 20 samples of given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	<PQL	Identify and correct source; Reanalyze blank prior to sample analysis.
	Calibration	pH 4 and 7 standards; once every 10 samples	≤0.1 units of true value	Recalibrate; check pH meter, replace probe and meter if necessary
	Calibration check	After initial calibration with pH 4 standard	≤0.1 units of true value	Recalibrate

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Alkalinity Analysis Target Analyte List

Analyte	Water (mg/L)	Soil (mg/kg)
Alkalinity	5	NA

MOUND

Environmental
Restoration
Program

Method: A-012

Isotopic Uranium, Isotopic Plutonium, and Isotopic Thorium by Alpha Spectrometry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Alpha Spectrometry

Specific isotopes from alpha spectrometry include plutonium^{238}, plutonium$^{239/240}$, uranium^{234}, uranium^{235}, uranium^{238}, thorium^{227} (for calculation of actinium^{227}), thorium^{228}, thorium^{230}, and thorium^{232}. Soil samples are prepared using acid digestion procedures to concentrate the isotopes of interest in an aqueous matrix. The alpha emitting isotopes in these acid extracts and in water samples are precipitated from the aqueous solution. The precipitates are re-dissolved and subjected to a sequential separation of alpha isotopes by elution from anion/cation exchange resins. The separated alpha isotopes are counted using a surface barrier detector.

1.2 References

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

Coleman, G.H., "The Radiochemistry of Plutonium", NAS-NS-3058, National Academy of Sciences. September, 1965.

Grindler, J.E., "The Radiochemistry of Uranium", NAS-NS-3050, National Academy of Sciences. March, 1962.

Hyde, E.K., "The Radiochemistry of Thorium", NAS-NS-3004, National Academy of Sciences. January 1960.

2. PRESERVATION

Alpha Spectrometry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Gamma Spectrometry **Plutonium Isotopes** **Thorium Isotopes** Radium^{226} Americium^{241} **Uranium Isotopes** Strontium^{90}	Nuclear Data, Inc. 1986 NAS 1965 NAS 1960 ASTM D2460-70 EML Am-01 NAS 1962 NAS 1960	Plastic cubetainer	2x4 liter	HNO_3 to pH ≤ 2 (15 mL 1N HNO_3 per liter)	NA
Soil	Gamma Spectrometry Tritium **Plutonium Isotopes** **Thorium Isotopes** **Uranium Isotopes** Strontium^{90}	Nuclear Data, Inc.1986 E906.0 NAS 1965 NAS 1960 NAS 1962 BAS 1960	Wide-mouth nalgene bottle	750 grams	None	NA

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken rom the same container by the laboratory.

3. CALIBRATION

Alpha spectrometry will be used for measurement of isotopic plutonium, thorium and uranium. A pulse check is performed once every day to determine if the detection system is functioning correctly. The background level is checked for gross contamination at a minimum of once per week with a 1000-minute count.

4. QC CRITERIA

Table 4.1 - Alpha Spectrometry Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Isotopic uranium Isotopic plutonium Isotopic thorium	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	± 4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Alpha Spectrometry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Isotopic uranium Isotopic plutonium Isotopic thorium	Background (1000 minutes)	Once per week	For background subtraction; minimum detectable activity.	Identify and correct problem; recount
	Pulse check	Once per day	Peak counts at 5 meV ± 3 x SD	Identify and correct problem. Recheck.
	Method blank	1 per 20 samples of a similar matrix	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 per 20 samples of a similar matrix	± 3 x SD normalized deviations	Evaluate data for usability.
	Replicate sample	1 per 20 samples of a similar matrix	± 4 x SD normalized range	Evaluate data for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Alpha Spectrometry Analysis Target Analyte List

Analyte	Water (pCi/L)	Soil (pCi/L)
$Plutonium^{238}$	1.0	0.01
$Plutonium^{239/240}$	1.0	0.01
$Thorium^{227}$	1.0	0.10
$Thorium^{228}$	1.0	0.10
$Thorium^{230}$	1.0	0.10
$Thorium^{232}$	1.0	0.10
$Uranium^{234}$	1.0	0.10
$Uranium^{235}$	1.0	0.10
$Uranium^{238}$	1.0	0.10

The reporting limits in Table 5.1 reflect the limits required by EG&G's most recent laboratory statement of work, and are not reflective of the QAPP limits.

ATTACHMENT A FOR METHOD A-012

Attachment A for Method A-012

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-012.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-013

Isotopic Americium ^{241} in Water by Alpha Spectrometry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Description

Americium^{241} in the water samples is precipitated from the aqueous solution. The precipitates are re-dissolved and subjected to a sequential separation of alpha isotopes by elution from anion/cation exchange resins. The separated americium^{241} is counted using a surface barrier detector.

1.2 References

DOE. "EML Procedure Manual," HASL-300, Environmental Measurements Laboratory, U.S. Department of Energy, 27th edition.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Americium^{241} Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Gamma Spectrometry	Nuclear Data, Inc. 1986	Plastic cubetainer	2x4 liter	HNO_3 to pH < 2 (15 mL 1N HNO_3 per liter)	NA
	Plutonium Isotopes	NAS 1965				
	Thorium Isotopes	NAS 1960				
	Radium^{226}	ASTM D2460-70				
	Americium^{241}	EML Am-01				
	Uranium Isotopes	NAS 1962				
	Strontium^{90}	NAS 1960				

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

Alpha spectrometry will be used for measurement of Americium^{241}. A pulse check is performed once every day to determine if the detection system is functioning correctly. The background level is checked for gross contamination at a minimum of once per week with a 1000-minute count.

4. QC CRITERIA

Table 4.1 - Americium^{241} Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Americium^{241}	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	+ 4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Alpha Spectrometry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Americium^{241}	Background (1000 minutes)	Once per week	For background subtraction; minimum detectable activity.	Identify and correct problem; recount
	Pulse check	Once per day	Peak counts at 5 meV ± 3 x SD	Identify and correct problem. Recheck.
	Method blank	1 per 20 samples of a similar matrix	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 per 20 samples of a similar matrix	± 3 x SD normalized deviations	Evaluate data for usability.
	Replicate sample	1 per 20 samples of a similar matrix	±4 x SD normalized range	Evaluate data for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Americium^{241} Analysis Target Analyte List

Analyte	Water (*p*Ci/L)	Soil (*p*Ci/L)
Americium^{241}	1.0	NA

MOUND

Environmental
Restoration
Program

Method: A-014

Tritium

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Tritium Analysis Description

Groundwater, surface water and soil/sediment samples will be analyzed for tritium according to EPA method 906.0A (EPA 1980). Beta emissions are detected using a liquid scintillation method with a fluorescence detector. A statement of work for preparation of soil samples for tritium analysis is provided in Attachment A.

1.2 References

EPA. "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," U.S. Environmental Protection Agency, EPA-600/4-80-032, latest version.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Tritium Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Tritium	E906.0	Glass bottle	250 mL	None	None
Soil	Tritium Plutonium Isotopes Thorium Isotopes Uranium Isotopes Strontium^{90}	E906.0	Wide-mouth nalgene bottle	750 grams	None	None

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

Liquid scintillation is used to measure beta particle activity from tritium. A source check is performed daily to verify calibration and efficiency. The background level is also checked daily.

4. QC CRITERIA

Table 4.1 - Tritium Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Tritium	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	±4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Tritium Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Tritium	Background	Once per day	+ 3 x SD, limit-gross contamination; background subtracts.	Identify and correct problem.
	Source check	Once per day	± 3 x SD	Identify and correct problem.
	Method blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 every 20 or fewer field samples of a similar matrix.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 every 20 or fewer field samples of a similar matrix.	+ 4 x SD normalized deviation	Identify and correct problem; evaluate associated sample results for usability.
	Replicate sample	1 every 20 or fewer field samples of a similar matrix.	+ 4 x SD normalized range	Identify and correct problem; evaluate associated sample results for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Tritium Analysis Target Analyte List

Analyte	Water (pCi/L)	Soil (pCi/g)
Tritium	500	50

ATTACHMENT A FOR METHOD A-014

Attachment A for Method A-014

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop

4.2 Glass tray, plastic tray, or other material for containing spilled soil

4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-014.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-015

Gamma Spectrometry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Gamma Spectrometry Analysis Description

Gamma spectrometry measures gamma radiation over a given spectrum and will be used to determine the gamma radiation levels in water and soil/sediment samples. Particular isotopes of interest that will be detected as gamma radiation are radium^{226} (soil samples), bismuth^{210} metastable, americium^{241} (soil samples), cobalt^{60}, cesium^{137}, bismuth^{207}, polonium^{210}, and potassium^{40}. Analysis will be performed according to the instrument's spectroscopy application user's manual. Sample preparation and analysis procedures are provided in the laboratory SOPs and Attachment A of this procedure. The detection limits listed on Table 5.1 are based on cesium^{137} and assume no interfering lines. Detection limits of individual isotopes may vary. These methods are based on procedures outlined in HASL-300 (DOE 1982) and in USEPA method 901.1 (EPA 1980).

1.2 References

DOE. "EML Procedures Manual", HASL-300, Environmental Measurements Laboratory, U.S. Department of Energy, 27th Edition.

ND9900 VAX/VMS Spectroscopy Application Package User's Manual (09-0196), Nuclear Data, Inc., Schaumberg, IL. August 1986.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Gamma Spectrometry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	**Gamma Spectrometry** Plutonium Isotopes Thorium Isotopes Radium^{226} Americium^{241} Uranium Isotopes Strontium^{90}	Nuclear Data, Inc. 1986 NAS 1965 NAS 1960 ASTM D2460-70 EML Am-01 NAS 1962 NAS 1960	Plastic cubetainer	2x4 liter	HNO_3 to pH ≤ 2 (15 mL 1N HNO_3 per liter)	NA
Soil	**Gamma Spectrometry** Tritium Plutonium Isotopes Thorium Isotopes Uranium Isotopes Strontium^{90}	Nuclear Data, Inc. 1986 E906.0 NAS 1965 NAS 1960 NAS 1962 BAS 1960	Wide-mouth nalgene bottle	750 grams	None	NA

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

3.1 Gamma Spectrometry

For gamma spectrometry, the counting efficiency is verified once per day with a source check. A mixed standard consisting of selected radionuclides of interest is used with initial instrument setup and when necessary, to perform an energy and efficiency calibration of the detection system. The background level is checked for contamination once per day with a 10-minute count. Background is established with a 100-minute count performed once per month.

4. QC CRITERIA

Table 4.1 - Gamma Spectrometry Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	± 4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Gamma Spectrometry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry	Background (10 minutes)	Once per day	No identifiable peaks; ± 20% error	Identify and correct problem; recount
	Background (1000 minutes)	Once per month	Not applicable; stored for background subtraction	No applicable
	Source check	Once per day	±3 x SD	Identify and correct problem; recount.
	Mixed standard	Initial setup and as necessary	Full range energy, linearity and efficiency calibration ± 5% of known standard	Not applicable.
	Replicate sample	1 per 20 samples of a similar matrix	± 4 x SD normalized range	Identify and correct problem; evaluate associated sample results for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected reporting limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Gamma Spectrometry Analysis Target Isotope List

Analyte	Water (*p*Ci/L)	Soil (*p*Ci/g)
Americium^{241}	NA	1
Cobalt^{60}	20	1
Cesium^{137}	20	1
Bismuth^{210} metastable	15	1
Bismuth^{207}	15	1
Potassium^{40}	350	10
Radium^{226}	NA	0.3

ATTACHMENT A FOR METHOD A-015

Attachment A for Method A-015

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-015.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-016

Strontium ^{90}

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Strontium^{90} Analysis Description

All strontium present in the sample is assumed to be strontium^{90}, due to the short half-life of strontium^{89} and the knowledge of the processes at Mound Plant. Soil samples are subjected to acid digestions to remove interferences and concentrate the strontium as an aqueous matrix. Strontium^{90} is precipitated from aqueous samples and soil acid extracts. Interferences are reduced by continued precipitations of the strontium carrier. The beta activity of yttrium^{90} is determined with a gas flow proportional detector immediately after its removal from the strontium^{90} determination.

1.2 References

Martin, D.B., "Determination of Strontium^{89} and Strontium^{90} in Soil with Total Sample Decomposition", Analytical Chemistry, October 1979.

Sunderman, D.N. and Townley, D.W., "The Radiochemistry of Barium, Calcium, and Strontium," NAS-NS-3010, National Academy of Sciences, January 1960.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Strontium^{90} Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Gamma Spectrometry Plutonium Isotopes Thorium Isotopes Radium^{226} Americium^{241} Uranium Isotopes **Strontium^{90}**	Nuclear Data, Inc. 1986 NAS 1965 NAS 1960 ASTM D2460-70 EML Am-01 NAS 1962 NAS 1960	Plastic cubetainer	2x4 liter	HNO_3 to pH ≤ 2 (15 mL 1N HNO_3 per liter)	NA
Soil	Gamma Spectrometry Tritium Plutonium Isotopes Thorium Isotopes Uranium Isotopes **Strontium^{90}**	Nuclear Data, Inc.1986 E906.0 NAS 1965 NAS 1960 NAS 1962 BAS 1960	Wide-mouth nalgene bottle	750 grams	None	NA

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

3.1 Strontium^{90}

There was no description in the Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan.

4. QC CRITERIA

Table 4.1 - Strontium^{90} Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Strontium^{90}	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	±4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Strontium^{90} Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Strontium^{90}	Method blank	Once per day	≤ 2 x MDA	Identify and correct problem; reanalyze
	Background check	Once per week	±3 x SD, limit-gross contamination	Identify and correct problem; recheck
	Instrument reliability	Once per day	±3 x SD	Identify and correct problem; recheck
	Method spike	1 per 20 samples of a similar matrix	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability
	Matrix spike	1 per 20 samples of a similar matrix	±3 x SD normalized deviations	Evaluate data for usability
	Replicate sample	1 per 20 samples of a similar matrix	±4 x SD normalized range	Evaluate data for usability
	Plateau	Once per year	Not applicable	Not applicable
	Efficiency determination	Once per year	Not applicable	Not applicable

5. ANALYTE LIST AND REPORTING LIMITS

These are expected reporting limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Strontium^{90} Analysis Target Isotope List

Analyte	Water (*p*Ci/L)	Soil (*p*Ci/g)
Strontium^{90}	5	1.0

ATTACHMENT A FOR METHOD A-016

Attachment A for Method A-016

Statement of Work for Soil Preparation for Common Organic, Inorganic, and Selected Radiological Analyses

1.0 Scope and Application

This procedure describes how to aliquot Mound soil and soil-like samples for laboratory preparation and analysis. This procedure applies to soil analysis for metals, semi-volatiles, pesticide/PCBs, cyanide, anions, explosives, and radiological analyses which do not have a prescribed soil preparation procedure. This procedure should not be used for volatile organic analysis. Soils for volatile organic analysis will be prepared and homogenized as described in the method of analysis.

2.0 Summary

A representative aliquot of a sample is taken in the laboratory by either visually examining and taking a representative portion from each layer in a sample or taking a core of the sample.

3.0 Interferences

Soil samples are heterogeneous by nature. Because of this nature, target analytes are often channeled and concentrated in the soil in specific layers or locations. This heterogeneity may affect both how representative the sample is of the field location and how representative the laboratory aliquot is of the sample.

Heterogeneous nature of soils can sometimes be eliminated in laboratory aliquoting by visually inspecting the sample for layering and selecting a representative aliquot or by taking a core of the sample.

4.0 Equipment

4.1 Spatula or Scoop
4.2 Glass tray, plastic tray, or other material for containing spilled soil
4.3 Large container, i.e. 1000 mL Pyrex beaker

5.0 Reagents/Supplies

5.1 Disposable gloves

6.0 Sample Collection/Holding Time/Preservation

6.1 See Section 2.0 of Method A-016.

7.0 Procedure

7.1 Place a glass tray, plastic tray, or disposable paper beneath the sample container. The tray or paper will be used to contain any soil which accidentally falls off the bottle lip when the cap is opened or falls out while the sample is taken.

7.2 Visually examine the contents of the sample container. If obvious layering is present, then representative portions of each layer must be taken for the aliquot.

7.2.1 If the sample is obviously a core sample (cylindrical soil mass), then use the spatula to core from the top of the sample to the bottom of the sample. This procedure should be representative of the entire core.

7.2.2 If the sample cannot be easily cored, it may be necessary to transfer the sample to a large container and thoroughly and carefully mix the sample with a spatula or scoop. Mixing will not be performed on soil samples for volatile and semi-volatile analyses.

7.2.3 If the sample is neither layered nor a core sample, then use a spatula to core through the middle of the sample. The core should be representative of the entire sample.

7.3 Process the sample as specified in the applicable method.

8.0 Quality Control

8.1 Each analytical method has specific types of quality control samples introduced to evaluate laboratory precision and reproducibility of sample results. Typically, these quality control samples are laboratory duplicates or matrix spike duplicates. These quality control samples permit the laboratory to calculate the relative percent difference and evaluate the soil aliquoting procedure and the precision of the method.

9.0 References and Associated Standard Operating Procedures

None

MOUND

Environmental
Restoration
Program

Method: A-017

Isotopic Radium ^{226} in Water

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Radium^{226} Analysis Description

Radium^{226} in water samples is precipitated from the aqueous solution. The precipitates are re-dissolved and subjected to a sequential separation of alpha isotopes by elution from anion/cation exchange resins. The separated alpha isotopes are counted using a surface barrier detector.

1.2 References

ASTM. 1991. "Annual Book of American Society of Testing Materials Standards." Section II, Water and Environmental Technology. Volume 11.02 Water II.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Radium^{226} Analysis
Sample Containers, Volumes, Preservation, and Holding Times:

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Alpha Spectrometry Gamma Spectrometry Plutonium Isotopes Thorium Isotopes **Radium^{226}** Americium^{241} Uranium Isotopes Strontium^{90}	Nuclear Data, Inc. 1986 NAS 1965 NAS 1960 ASTM D2460-70 EML Am-01 NAS 1962 NAS 1960	Plastic cubetainer	2x4 liter	HNO_3 to pH ≤ 2 (15 mL 1N HNO_3 per liter)	NA

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

Alpha spectrometry will be used for measurement of radium^{226}. A pulse check is performed once every day to determine if the detection system is functioning correctly. The background level is checked for gross contamination at a minimum of once per week with a 1000-minute count.

4. QC CRITERIA

Table 4.1 - Radium^{226} Analysis Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Radium^{226}	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	±4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.

Table 4.2 - Radium^{226} Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Radium^{226}	Background (1000 minutes)	Once per week	For background subtraction; minimum detectable activity.	Identify and correct problem; recount
	Pulse check	Once per day	Peak counts at 5 meV ± 3 x SD	Identify and correct problem. Recheck.
	Method blank	1 per 20 samples of a similar matrix	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 per 20 samples of a similar matrix	± 3 x SD normalized deviations	Evaluate data for usability.
	Replicate sample	1 per 20 samples of a similar matrix	±4 x SD normalized range	Evaluate data for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Radium^{226} Analysis Target Analyte List

Analyte	Water (pCi/L)	Soil (pCi/kg)
Radium^{226}	1.0	NA

MOUND

Environmental
Restoration
Program

Method: A-018

Volatiles Organic Analysis/ EPA Method 8030

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

1.1 Acetonitrile/Acrylonitrile Analysis Description

EPA Method 8030 with the purge and trap technique (5030) will be used to identify acrylonitrile and acetonitrile in groundwater samples. This gas chromatography method uses a flame ionization detector to detect these volatile compounds.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

2. PRESERVATION

Volatile Organic Analysis - EPA Method 8030
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Volatile Organic Compounds	SW5030/SW8030	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of volatile organic compounds in groundwater (Method 8030). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column). A minimum of five external standards for volatile organic analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for volatile organic compounds (VOCs) are valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for volatile organic analysis is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time windows for VOCs are established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis - EPA Method 8030 Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8030	Trip Blank	1 per shipping container to lab	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Sample bank blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Ambient blank	1 every 20 or fewer field samples	≤ 10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water)	≤ 35% RPD	Evaluate data for usability.

Table 4.2 - Volatile Organic Analysis - EPA Method 8030 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8030	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3 x SD of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Volatile Organic Analysis - EPA Method 8030 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
Volatile Organic	*Matrix Spike/LCS*						
Compounds	Acetonitrile	*	NA	70-136	NA	≤15	NA
SW8030	Acrylonitrile	*	NA	70-135	NA	≤15	NA

* Per standard laboratory specification (mid-range response).

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Volatile Organic Analysis - EPA Method 8030 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Acetonitrile	10	NA
Acrylonitrile	10	NA

MOUND

Environmental
Restoration
Program

Method: A-020

Volatiles Organic Analysis/ EPA Method 8020

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document:

1. INTRODUCTION

1.1 Description

Soil samples will be analyzed for aromatic VOCs using gas chromatography with a photoionization detector. The methodology to be followed is EPA Method 8020 (EPA 1987). This method was added to satisfy State of Ohio Buried Underground Storage Tank Requirements. The method is appropriate for verifying compliance to the Ohio BUSTR regulations.

1.2 References

EPA. 1986. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. November 1986.

EPA. 1987. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. December 1987.

EPA. 1990. "Test Methods for Evaluating Solid Waste." Laboratory Manual/Physical Methods, SW-846, Volumes 1A, 1B and 1C, third edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. March 1990.

2. PRESERVATION

Volatile Organic Analysis - EPA Method 8020
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Volatile Organic Compounds	SW5030/SW8020	Glass vial with Teflon-lined septum (no headspace)	Two 40 mL vials	HCl to pH<2 Cool 4°C	14 days
Soil	Volatile Organic Compounds	SW5030/SW8020	Glass bottle with Teflon-lined lid (no headspace)	120g bottle	Cool 4°C	14 days

3. CALIBRATION

Gas chromatography will be used for analysis of volatile organic compounds in groundwater (Methods SW-8020). Initial calibration is performed when chromatographic conditions are changed (e.g., change in flow rate, detectors, new column. A minimum of five external standards for volatile organic analysis are analyzed to determine the linearity of the gas chromatograph. Response factors for each compound are calculated (as specified in the methods) from the results, and a calibration curve generated. Linearity criteria for volatile organic compounds (VOCs) are

valid if there is less than or equal to 20% relative standard deviation among the calibration factors. A quadratic curve may also be used.

The linearity of the gas chromatograph for volatile organic analysis is checked by analysis of a check standard after every 10 sample analyses. The response for any analyte must be within a 15% difference of the response from the initial calibration. If the percent difference exceeds this criterion, then the instrument is checked and a new calibration curve is performed before samples are analyzed.

Retention time windows for VOCs are established when a column is changed or after other changes are made in instrument conditions that will alter the retention times of the analytes of interest. The windows are established according to procedures defined in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA (EPA 1987).

4. QC CRITERIA

Table 4.1 - Volatile Organic Analysis - EPA Method 8020 Field QC Sample Frequency

Parameters	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8020	Trip Blank	1 per shipping container to lab	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ 0.10 x level in associated samples, or ≤ PQL	Evaluate potential sources; Evaluate associated data for usability.
	Field Duplicate	1 every 10 or fewer field samples (water)	≤ 35% RPD	Evaluate data for usability.

Table 4.2 - Volatile Organic Analysis - EPA Method 8020 Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
VOC, SW8020	Method Blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ PQL	Identify and correct source. Reanalyze blank and associated samples.
	Calibration	5 points; when calibration check criteria exceeded.	≤ 20% RSD for calibration factors	Recalibrate
	Calibration check	Once per 10 samples analyzed.	± 15% from initial response factor	Recalibrate
	Matrix spike	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Matrix spike duplicate	1 per 20 samples of a given matrix	See Table 4.3	Evaluate data for usability.
	Surrogate spikes	All field and lab samples	See Table 4.3	Check calculations, surrogate and standard solutions, and instrument. If problem not identified then reanalyze sample.
	Retention time window	When new column installed and as needed	±3 x SD of three retention times for each analyte as per SW 846.	Identify source, correct problem.
	Laboratory control sample (LCS)	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	See Table 4.3	Identify and correct problem prior to further sample analyses, reanalyze.

Table 4.3 - Volatile Organic Analysis - EPA Method 8020 Laboratory Surrogate and Matrix Spike Limits

Analytical Method	Spiking Compounds	Spike Concentration		Advisory Limits Percent Recovery		Advisory Limits Relative Percent Difference (%)	
		Water (µg/L)	*Soil (µg/kg)*	*Water*	*Soil*	*Water*	*Soil*
Volatile Organic Compounds, SW8020	*Matrix Spike/LCS*						
	Benzene	*	*	80-120	80-120	≤15	≤30
	Toluene	*	*	80-120	80-120	≤15	≤30
	Ethyl Benzene	*	*	80-120	80-120	≤15	≤30
	Xylenes	*	*	80-120	80-120	≤15	≤30
	Surrogates						
	Fluorobenzene	30	30	80-120	80-120	≤15	≤30

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 - Volatile Organic Analysis - EPA Method 602 Target Analyte List

Analyte	Water (µg/L)	Soil (µg/kg)
Benzene	2.0	NA
Ethylbenzene	2.0	NA
Toluene	2.0	NA
Xylene	2.0	NA

FIELD METHODS

FIELD METHODS

Field methods describe the quality control requirements for methods of analysis performed on-site at the Mound Plant. Typically, field methods will be used when there are less stringent data reporting requirements, fast turn around time is needed, or the on-site method is capable of meeting the designated data quality objective and is cost competitive. Because the field methods described in the Remedial Investigation/Feasibility Study Operable Unit 9, Site-wide Quality Assurance Project Plan (QAPP) were only intended to provide basic field screening information and lacked many specific quality control requirements, the methods were not incorporated into this section of the compendium.

As new field methods are identified, Section 1.1 of each of the methods must briefly describe how the method will be used to meet the data quality objective for the potential release site. This description is required to facilitate using the method by reference for other potential release site investigations. When a new method is approved for use with a specific release site, then:

- the Source Document and Document Date on the title page of the method must be updated, and
- both the method and a revised table of contents for the section must distributed to the copy holders.

FIELD METHODS — TABLE OF CONTENTS

MOUND

Environmental
Restoration
Program

Method: F-001

Isotopic Uranium, Plutonium, and Thorium by Alpha Spectrometry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document:

1. INTRODUCTION

1.1 Description

Specific isotopes from alpha spectrometry include plutonium^{238}, plutonium$^{239/240}$, uranium^{234}, uranium^{235}, uranium^{238}, thorium^{228}, thorium^{230}, and thorium^{232}. These analyses will be performed at the DOE Mound facility in Miamisburg, Ohio. Soil samples will be digested in acid, passed through an ion exchange column, electrodeposited, and analyzed using a surface barrier alpha particle detector. Water samples are precipitated, the precipitate is dissolved in acid, passed through an ion exchange column, electrodeposited, and analyzed using a surface barrier alpha particle detector.

1.2 References

DOE 1995. "Uranium in Well Water by Co-precipitation Anion Exchange Method", Operation Number 3266, Technical Manual MD-80030, Issue 23, U.S. Department of Energy, July 1995.

DOE 1995. "Environmental/Bioassay Sample Counting Procedure", Operation Number 0054, Technical Manual MD-80030, Issue 23, U.S. Department of Energy, July 1995.

DOE 1995. "Plutonium Activity in Liquid Effluents", Operation Number 1272, Technical Manual MD-80030, Issue 23, U.S. Department of Energy, July 1995.

DOE 1995. "Leachable Plutonium in Solid Matrix," Operation Number 1385, Technical Manual MD-80030, Issue 23, U.S. Department of Energy, July 1995.

2. PRESERVATION

Alpha Spectrometry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Gamma Spectrometry	Nuclear Data, Inc. 1986	Plastic cubetainer	2 liter	HNO_3 to pH ≤ 2 (15 mL 1N HNO_3 per liter)	NA
	Plutonium Isotopes	NAS 1965				
	Thorium Isotopes	NAS 1960				
	Radium^{226}	ASTM D2460-70				
	Americium^{241}	EML Am-01				
	Uranium Isotopes	NAS 1962				
	Strontium^{90}	NAS 1960				

Alpha Spectrometry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Soil	Gamma Spectrometry	Nuclear Data, Inc.1986 E906.0	Wide-mouth nalgene bottle	500 grams	None	NA
	Tritium	NAS 1965				
	Plutonium Isotopes	NAS 1960				
	Thorium Isotopes	NAS 1962				
	Uranium Isotopes	BAS 1960				
	Strontium^{90}					

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

3.1 Alpha Spectrometry

Alpha spectrometry will be used for measurement of isotopic plutonium, thorium and uranium. A pulse check is performed once every day to determine if the detection system is functioning correctly. The background level is checked for gross contamination at a minimum of once per week with a 1000-minute count.

4. QC CRITERIA

Table 4.1 - Alpha Spectrometry Analysis
Recommended Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Isotopic uranium *Isotopic plutonium* *Isotopic thorium*	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤ one-fifth concentration in samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	RPD < 35%	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Alpha Spectrometry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Isotopic uranium *Isotopic plutonium* *Isotopic thorium*	Background (1000 minutes)	Once per week	For background subtraction; minimum detectable activity.	Identify and correct problem; recount
	Pulse check	Once per day	Peak counts at 5 meV ± 3 x SD	Identify and correct problem. Recheck.
	Method blank	1 per 20 samples of a similar matrix	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 per 20 samples of a similar matrix		Evaluate data for usability.
	Replicate sample	1 per 20 samples of a similar matrix	RPD ≤ 50%	Evaluate data for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Alpha Spectrometry Analysis Target Analyte List

Analyte	Water (pCi/L)	Soil (pCi/L)
Plutonium^{238}	1.0	0.01
Plutonium$^{239/240}$	1.0	0.01
Thorium^{227}	1.0	0.10
Thorium^{228}	1.0	0.10
Thorium^{230}	1.0	0.10
Thorium^{232}	1.0	0.10
Uranium^{234}	1.0	0.10
Uranium^{235}	1.0	0.10
Uranium^{238}	1.0	0.10

MOUND

Environmental
Restoration
Program

Method: F-002

Gamma Spectrometry

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document:

1. INTRODUCTION

1.1 Gamma Spectrometry Analysis Description

Gamma spectrometry measures gamma radiation over a given spectrum and will be used to determine the gamma radiation levels in water and soil/sediment samples. Particular isotopes of interest that will be detected as gamma radiation are radium^{226} (soil samples), bismuth^{210} metastable, americium^{241} (soil samples), cobalt^{60}, cesium^{137}, bismuth^{207}, polonium^{210}, and potassium^{40}.

This analysis will be performed at the DOE Mound Facility in Miamisburg, and according to the instrument's spectroscopy application user's manual. Sample preparation and analysis procedures are provided in the laboratory SOPs. The detection limits listed on Table 5.1 are based on cesium^{137} and assume no interfering lines. Detection limits of individual isotopes may vary. These methods are based on procedures outlined in HASL-300 (DOE 1982) and in USEPA method 901.1 (EPA 1980).

1.2 References

DOE. "EML Procedures Manual", HASL-300, Environmental Measurements Laboratory, U.S. Department of Energy, 27th Edition.

ND9900 VAX/VMS Spectroscopy Application Package User's Manual (09-0196), Nuclear Data, Inc., Schaumberg, IL. August 1986.

2. PRESERVATION

Gamma Spectrometry Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters[1]	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Gamma Spectrometry	Nuclear Data, Inc. 1986	Plastic cubetainer	750mL	HNO_3 to pH < 2 (15 mL 1N HNO_3 per liter)	NA
Soil	Gamma Spectrometry	Nuclear Data, Inc.1986	Wide-mouth nalgene bottle	750 grams	None	NA

[1] Other radiological analyses have been listed to ensure field personnel know which analyses can be taken from the same container by the laboratory.

3. CALIBRATION

For gamma spectrometry, the counting efficiency is verified once per day with a source check. A mixed standard consisting of selected radionuclides of interest is used with initial instrument setup and when necessary, to perform an energy and efficiency calibration of the detection system. Background is established with a 600-minute count performed once per week.

4. QC CRITERIA

Table 4.1 - Gamma Spectrometry Analysis Recommended Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry	Field duplicate	1 every 10 or fewer field samples (water)	± 4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Gamma Spectrometry Analysis Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry	Background (600 minutes)	Once per week	Not applicable; stored for background subtraction	No applicable
	Source check	Once per day	10% of expected	Identify and correct problem; recount.
	Mixed standard	Daily	Full range energy	Not applicable.
	Mixed standard	Once per year or with equipment change	Efficiency calibration ± 5% of known standard	Not applicable.
	Replicate sample	1 per 20 samples of a similar matrix	±4 x SD normalized range	Identify and correct problem; evaluate associated sample results for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected reporting limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Gamma Spectrometry Analysis Target Isotope List

Analyte	Water (*p*Ci/L)	Soil (*p*Ci/g)
Americium^{241}	NA	0.1
Cobalt^{60}	20	0.05
Cesium^{137}	20	0.05
Bismuth^{210} metastable	15	0.05
Bismuth^{207}	15	0.03
Potassium^{40}	350	0.9
Radium^{226}	NA	0.7

MOUND

Environmental
Restoration
Program

Method: F-003

Plutonium ^{238} and Thorium ^{232} by Thin Sodium Iodide Detector

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document:

1. INTRODUCTION

The bicron detector, a thin sodium iodide detector, is used to measure plutonium^{238} concentrations in soil. The analysis is performed in accordance with procedure 1355 from the Mound Plant Technical Manual of Environmental Analytical Procedures.

1.1 References

DOE. "EML Procedures Manual", HASL-300, Environmental Measurements Laboratory, U.S. Department of Energy, 27th Edition.

DOE 1995. "Remedial Investigation/Feasibility Study Operable Unit 9, Site-Wide Quality Assurance Project Plan," Final Revision 4, U.S. Department of Energy, April 1995.

DOE 1995. "Technical Manual", MD-80030, Issue 23. Environmental Analytical Procedures, U.S. Department of Energy, August 1995.

2. PRESERVATION

Gamma Spectrometry Analysis - Bicron Detector Sample Container, Volume, Preservation, and Holding Time

Matrix	Parameters[1]	Analytical Method	Container	Minimum Amount	Preservation	Holding Time
Soil	Plutonium^{238}	Mound Op. 1355	Wide-mouth plastic bottle	700 grams	None	NA
	Thorium^{232}					

3. CALIBRATION

Three plutonium^{238} NIST traceable standards are used to develop a density correction curve for efficiency. The curve will be prepared annually, or when the instrument is changed or a verification check fails criteria. A verification check will be performed daily by analyzing an NIST traceable standard. If the verification check is not within 10% of the expected result, the need to re-establish a density correction curve will be evaluated. A ten minute background and energy calibration will be performed each week.

4. QC CRITERIA

Table 4.1 - Gamma Spectrometry Analysis - Bicron Detector Recommended Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry	Field duplicate	1 every 10 or fewer field samples (soil)	Not applicable	Evaluate variability.

Table 4.2 - Gamma Spectrometry Analysis - Bicron Detector Laboratory QC Sample Frequency

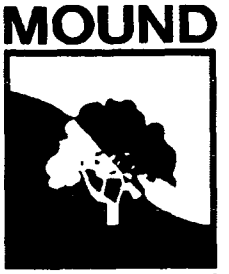
Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Gamma spectrometry, Bicron detector	Density Correction Curve	Annually, or when instrument changed or verification sample fails.	NA	NA
	Background (10 minutes)	Weekly	Not applicable; stored for background subtraction	Not applicable
	Verification	Once per day	±10% of true	Identify and correct problem; recount.
	Energy Calibration	Weekly	Verify peak within region of interest.	Not applicable.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected reporting limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Gamma Spectrometry Analysis - Bicron Detector Target Isotope List

Analyte	Soil (pCi/g)
Plutonium^{238}	25
Thorium^{232}	5



Method: F-004

Tritium

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document:

1. INTRODUCTION

1.1 Tritium Analysis Description

Groundwater, surface water and soil/sediment samples will be analyzed for tritium. Beta emissions are detected using a liquid scintillation method with a fluorescence detector. Soil samples will be prepared using distillation to separate the water from the soil. This analysis will be performed at the DOE Mound facility in Miamisburg.

1.2 References

DOE 1995. "Tritium in Environmental Water Samples," Operation Number 2261, Technical Manual MD-80030, Issue 23, July 1995.

DOE 1995. "Distillation of Tritium in Solids," Operation Number 2722, Technical Manual MD-80030, Issue 23, July 1995.

DOE 1995. "Distillation of Tritium in Water and Other Aqueous Base Liquids," Technical Manual MD-80030, Issue 23, July 1995.

2. PRESERVATION

Tritium Analysis
Sample Containers, Volumes, Preservation, and Holding Times

Matrix	Parameters	Analytical Method	Container	Minimum Volume	Preservation	Holding Time
Water	Tritium	Mound Op. 2261	Glass bottle	250 mL	None	None
Soil	Tritium	Mound Op. 2722	Wide-mouth nalgene bottle	150 g	None	None

3. CALIBRATION

Liquid scintillation is used to measure beta particle activity from tritium. A source check is performed daily to verify calibration and efficiency. The background level is also checked daily.

4. QC CRITERIA

Table 4.1 - Tritium Analysis
Recommended Field QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Tritium	Equipment (rinsate) blank	1 every 10 or fewer field samples (water)	≤10 x level in associated samples	Evaluate potential sources; Evaluate associated data for usability.
	Field duplicate	1 every 10 or fewer field samples (water)	± 4 x SD	Evaluate data for usability.
		1 every 10 or fewer field samples(soil)	Not applicable	Evaluate variability.

Table 4.2 - Tritium Analysis
Laboratory QC Sample Frequency

Parameter	Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Tritium	Background	Once per day	± 3 x SD, limit-gross contamination; background subtracts.	Identify and correct problem.
	Source check	Once per day	± 3 x SD	Identify and correct problem.
	Method blank	1 per 20 samples of a given matrix or 1 whenever a batch of samples is prepared in a day, whichever is more frequent.	≤ 2 x MDA	Identify and correct problem. Reanalyze blank.
	Method spike	1 every 20 or fewer field samples of a similar matrix.	±3 x SD normalized deviations	Identify and correct problem; evaluate associated sample results for usability.
	Matrix spike	1 every 20 or fewer field samples of a similar matrix.	± 4 x SD normalized deviation	Identify and correct problem; evaluate associated sample results for usability.
	Replicate sample	1 every 20 or fewer field samples of a similar matrix.	± 4 x SD normalized range	Identify and correct problem; evaluate associated sample results for usability.

5. ANALYTE LIST AND REPORTING LIMITS

These are expected quantitation limits based on reagent grade water or a purified solid matrix. Actual quantitation limits may be higher depending upon the nature of the sample matrix. The limit reported on final laboratory reports will take into account the actual sample volume or weight, percent moisture (where applicable), and the dilution factor, if any.

Table 5.1 Tritium Analysis
Target Analyte List

Analyte	Water (pCi/L)	Soil (pCi/g)
Tritium	500	50

QUALITY ASSURANCE METHODS

QUALITY ASSURANCE METHODS

Quality assurance methods were included in the compendium to provide consistency between and within different subcontractors who will perform sampling and analysis of the release sites at the Mound Plant. The first three procedures in this section, Q-001 to Q-003, were extracted from the Remedial Investigation/Feasibility Study Operable Unit 9, Site-wide Quality Assurance Project Plan (QAPP). The procedures describe documenting problems, maintaining chain-of-custody, and managing documentation records. To facilitate the use of these QAPP extracted documents by multiple subcontractors, some of the forms and text were modified, particularly in the Corrective Action Report procedure. The other two methods were changed only slightly to improve the readability of the extracted text. The forms described within the methods are available on electronic media as Microsoft ™ Word® documents. Each of the QAPP extracted methods list the Source Document as QAPP and the Document Date as April 1995.

As new program level quality assurance methods are required, the methods will be introduced into the compendium. If appropriate, the title page of the quality assurance procedure will reference a Source Document and Document Date. If the method is not introduced as part of a sample plan, then only the Document Date will be included. When a new method is added, the revised table of contents for the section and the method will be distributed to the copy holders of the compendium.

QUALITY ASSURANCE METHODS — TABLE OF CONTENTS

MOUND

Environmental
Restoration
Program

Method: Q-001

Corrective Action Reports

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

Corrective action reports are used to document errors and deviations from written plans. When errors are identified or changes to a written plan are required, a corrective action report must be initiated by the person who identified the problem. The corrective action report is then submitted to the project manager or quality assurance manager, and is ultimately included in the data report to the DOE Mound Plant.

2. RESPONSIBILITIES

2.1 Person Who Identified the Deficiency

The person who identified the deficiency must initiate the corrective action report. If the person takes an action to correct the problem, then the corrective action section of the report must be completed by the person.

2.2 Project Manager

The project manager must review all corrective action reports and verify that an appropriate corrective action has been taken, and that the form has been completed properly. The project manager is also responsible for signing the report and maintaining a log which summarizes the corrective action reports. The project manager may assign these duties to a quality assurance manager.

3. PROCEDURES

When an error is identified or a need to deviate from a written plan is identified by an employee the employee must create a corrective action report, Figure 3.1. The employee is responsible for completing the Task Name, Internal Project Number, Project Manager, Initiator, Date Initiated, Requirement, and the Finding/Observation. Requirement on the form is used to describe the condition or specification which was violated. The Finding/Observation describes how the requirement was violated. If the employee takes an action to correct the error, the remedial corrective action section of the report must also be completed, as in the following:

The sampling plan states "All soil samples will be collected using a stainless steel scoop and placed in a glass bottle." During the sampling event, the field technician used a steel shovel and placed the sample into a glass bottle. In this situation, the Requirement Finding/Observation, and corrective action sections of the form would be completed as:

Requirement: According to the sampling plan, all soil samples should be collected using a stainless steel scope.
Finding/Observation: Ten samples were collected using a steel shovel. The ten samples are listed on the attached sheet.
Corrective Action for Incident: The error was identified after the 10th sample was collected, and all field technicians were told to use a stainless steel scoop. The laboratory was directed by Jane Doe to not analyze the affected samples on March 10, 1996. The samples will be recollected.

The completed corrective action must be submitted to the project manager.

The project manager is responsible for reviewing each of the corrective actions and verifying that the report is complete and that the remedial corrective action is appropriate. If a remedial correction action has not been implemented, then the project manager will identify what corrective action is required. The project manager will also determine what actions can be taken to prevent recurrence by identifying the root cause and implementing a preventive corrective action. An example of a possible root cause and preventive corrective action for the previous example might be:

Root Cause: The field technician accidentally picked up a draft copy of the sampling plan. The draft copy of the sampling plan specified samples must be collected with steel shovels.
Corrective Action to Prevent Recurrence: Prior to this incident, draft copies were not collected back from the copy holders. The document distribution system has been revised to ensure that draft documents are returned when the final version of a document is distributed. Additionally, we have implemented a checklist for field team leaders to ensure properly documents are in use and the proper procedures are being followed.

When the project manager completes the report, he will assign a tracking number (CAR No.) to the report and record the report on a project specific tracking sheet, Figure 3.2. The tracking number is simply a sequential number assigned to the report. An example of a completed log entry for the example is:

CAR No.	Date	Description	I Impact on Date
001	10-Mar-96	A steel shovel was used to collect 10 samples instead of a stainless steel scoop.	The samples were re-collected and the data quality was not impacted.
002			

When the project has been completed, the corrective action log and corrective actions are part of the client deliverables and must be turned over to DOE Mound.

Mound Plant
Nonconformance/Corrective Action Report

Task Name:		**Initiator:**	
Internal Project Number:		**File Code:**	
Project Manager:		**Date Initiated:**	
CAR No:		**Revision:**	

Requirement:

Finding/Observation:

Corrective Action for Incident:

Initiator of Corrective Action: _____ *Date:*_____

Root Cause:

Corrective Action to Prevent Recurrence:

Initiator of Corrective Action: _____ *Date:*_____

Rejected By: ___ Date Rejected: _

Verifications/Approvals: Project Manager:	Date:

Mound Plant
Corrective Action Report Log

CAR No.	Date	Description	Impact on Data

MOUND

Environmental
Restoration
Program

Method: Q-002

Chain-of-Custody Procedures

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

Sample custody procedures to be followed during the activities require that the possession and handling of each sample from the moment of its collection through analysis be documented by written record. A sample is in someone's custody when one of the criteria listed below has been satisfied:

1. The sample is in one's actual possession.

2. The sample is in one's view after being in one's physical possession.

3. The sample is in one's physical possession and is then locked up so that no one can tamper with it.

4. The sample is kept in a secured area that is restricted to authorized personnel only.

Samples will consist of material collected in the field, such as water, soil, or sediments, and any reagents added for the purpose of sample preservation.

2. RESPONSIBILITIES

2.1 Field Crew and Support Staff

Field Technician: The field technician is responsible for properly collecting and handling samples, and properly documenting sample collection.

Field Sample Manager: The designated sample manager is responsible for properly packaging samples for shipment and completing chain-of-custody documentation.

Field Manager: The field manager is responsible for overseeing all aspects of field chain-of-custody, and resolving and documenting laboratory sample receipt problems. The field manager may delegate the resolution and documentation of sample receipt problems to a designated laboratory liaison as appropriate.

Laboratory Liaison: In the event the field team manager or project manager designates a laboratory liaison, the laboratory liaison is responsible for resolving and documenting sample receipt discrepancies.

2.2 Laboratory

Laboratory Sample Custodian: The sample custodian is responsible for verifying sample receipt requirements were met and sample custody is maintained within the laboratory. The custodian must report discrepancies to the laboratory project manager.

Laboratory Project Manager: The laboratory project manager is responsible for reporting sample discrepancies and working to resolve the discrepancy.

3. PROCEDURES

3.1 Field Custody Procedures

3.1.1 Sample Labels

All samples will be identified with a label attached directly to the container. Examples of sample labels are presented in Mound Plant ER Program SOP 1.3, Sample Control and Documentation. Sample label information will be completed using waterproof black ink. The labels will contain the following information:

- sample number,
- time and date of collection,
- installation name,
- parameters to be analyzed,
- preservative (if any),
- sample source/location, and
- sampler's initials.

3.1.2 Chain of Custody Record

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record (Figure 3.1) will be filled out for each sample as it is collected by the field sampler. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time of transfer, will be documented.

Chain-of-custody seals are used to determine if any tampering has occurred during shipment of samples. These signed and dated seals will be placed at the unction between the lid and the jar or cooler on all sample containers and shipment containers (coolers) by the person responsible for packaging. If the chain-of-custody seals are not intact, the laboratory project manager for the Mound Plant ER Program will notify the field manager within 24 hours of container receipt. The field manager will then complete a corrective action report Method Q-001.

3.1.3 Transfer of Custody and Shipment

Prior to shipment of samples, the chain-of-custody record will be signed and dated by a member of the field team who has verified that those samples indicated on the record are indeed being shipped. Mound Plant ER Program SOP 1.3, Sample Control Documentation describes the completion of this form and the steps necessary for sample control, sample identifications, and data recording. A copy of each chain-of-custody form will be retained in the project file at the site, and the original will be sent with the samples (sealed inside the sample cooler). After packaging has been completed, custody seals, signed and dated by a member of the field team, will be placed on the cooler.

After samples are collected and screened by Mound Plant, they will be transported by field personnel as soon as possible to the courier location for subsequent shipment to the laboratory or hand-delivered to the laboratory. (It should be noted that Federal Express® does not claim responsibility for samples and does not sign off on the chain of custody. However, the laboratory retains the shipping ticket, indicating acceptance and delivery of shipment.) Rental vehicles used by the field personnel may be used for transporting non-hazardous and non-radioactive samples from Mound Plant to the Federal Express® office only if the samples are properly packaged and labeled. Upon receipt of the samples at the laboratory, the receiver will complete the transfer by dating and signing the chain-of-custody record (Figure 3.1). This chain-of-custody record will remain with the sample at the laboratory.

3.2 Laboratory Custody Procedures

Sample custody procedures in the laboratory include the procedures for general security, sample receipt, storage, preparation, and analysis. The laboratory specifications attachments describe these procedures unique for the given laboratory. The following subsections describe the minimum general requirements for the laboratory.

3.2.1 Sample Receipt

- Samples will be checked for integrity and the temperature inside the coolers measured in the temperature blank, will be noted on the Chain of Custody. Sample containers for VOCs will be checked for bubbles. The field manager will be notified immediately of any discrepancies or broken bottles.
- The appropriate section managers and analysts will be notified of any short holding times.
- The laboratory will have a sample custodian who will assume custody of the samples by signing the chain of custody.
- The samples will be checked against the chain of custody and discrepancies will be resolved with the field manager.
- The completed chain of custody, with all relinquished signatures, will be returned to the subcontractor with the data package.

3.2.2 Sample Storage

Samples (except those samples designated for radiological or geotechnical analyses which do not have preservation requirements) will be stored in locked refrigerators which are maintained at 4° ± 2 °C. When samples, extracts, or digestates are retrieved from or returned to the refrigerator, a chain of custody record is signed by the analyst.

Unused samples, sample containers, sample extracts, and sample digests are stored for a minimum of 60 days after analysis and are not disposed of without written authorization from the Mound Plant subcontractor.

Laboratories will have controlled access to sample storage areas.

3.2.3 Sample Tracking

For samples requiring preparation, a sample preparation record is completed by the analyst/technician during the time of preparation. Sample extracts are maintained in secured refrigerator storage. Sample digests for metals and extracts for radiochemical analysis are stored at room temperature in a secured area.. Chemical and radiological sample preparation records are stored in a bound notebook.

3.2.4 Record Keeping

Sample preparation and analysis information are recorded in bound laboratory notebooks. Sample tracking information includes the following:

- project identification number;
- sample numbers;
- sample type;
- date received;
- date put into storage after analysis;
- date of extraction or digestion;
- date of analysis; and
- date of disposal.

Corrections to entries in laboratory notebooks are made by drawing a single line through the erroneous entry and entering the correct entry. Corrections are dated and initialed by the individual making the entry.

MOUND

Environmental
Restoration
Program

Method: Q-003

Documentation Requirements

Revision 1.0

Mound Plant
Miamisburg, OH

Source Document: QAPP (April 1995)

1. INTRODUCTION

This procedure describes the documentation requirements for Environmental Restoration projects.

2. RESPONSIBILITIES

Not applicable.

3. DOCUMENTATION

3.1 Records

3.1.1 Field Logs

All data collection activities performed at a site will be documented, using waterproof, non-erasable black ink, either in a field notebook or on ER Program forms. Field notebooks will be bound books and will be assigned to individual field personnel for the duration of their stay in the field. The required contents and procedures for entering into field notebooks are described in Subsection 3.1.2. In addition, all samples collected will be recorded in the field notebook with the following information:

- sample location,
- sample identification number,
- date and time of collection,
- sample matrix,
- any unusual appearances of the sample,
- parameters to be analyzed, and
- date and time sample was released or received.

3.1.2 Field Notebooks

Field notebook entries will include, on the inside cover and first pages, the following information:

- Name
- Company name and address
- Phone number
- Activity or location
- Phone numbers for supervisors, emergency response, etc.
- Table of contents
- The procedures (SOPs) used or followed for each field activity.

Daily entries will include the following information. Entries will be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory.

All records of numerical analyses performed on field and technical data will be legible, reproduction quality, and complete enough to permit logical reconstruction by a qualified individual other than the originator.

- Date and time
- Name of individual making the entry
- Description of test/activity
- Quantities of materials used
- Drawings and information related to the activity
- Conditions that might adversely affect the test/activity
- Names of witnesses or observers present
- Samples collected, received, or released
- Deviations from the procedures (SOPs) used or followed
- Calculations and sample collection information

For example, during drilling activities, the field team member supervising a rig will keep a chronological log of drilling activities, a vertical descriptive log of lithologies encountered, other pertinent drilling information (staining, odors, field screening, working conditions, water levels, geotechnical data), and a labor and materials accounting in the team member's bound notebook.

Ten percent of all calculations will be checked. Any inconsistencies or anomalies discovered will be resolved immediately, if possible, by seeking clarification from the filed personnel responsible for collecting the data.

Subjective field and technical data will also be reviewed for reasonableness and completeness by the installation manager. In addition, random checks of sampling and field conditions will be made by the field supervisor, who will check recorded data at that time to confirm the recorded observations. Whenever possible, peer review also will be incorporated into the data review process, particularly for subjective data, to maximize consistency among field personnel. For example, during drilling activities, the field manager will schedule periodic reviews of archived lithologic samples to ensure that the appropriate descriptions and codes are being consistently applied by all field personnel.

It will be the responsibility of all field personnel to photocopy all field logs (including notebook pages and ER Program forms) generated during a given field day, at the end of that day. Copies will be given to the site manager or field supervisor, who will maintain field log files. At the completion of a work shift, copies of all field logs, notebook pages, and ER Program forms will be returned to the EG&G subcontractor's office. These copies will be presented to the ER Program site manager and entered into the project file. At the completion of a field program, field logbooks will be returned to the project files. All field records will be kept on file by EG&G for a minimum of 10 years. EPA Region V and the EG&G will be notified prior to any intent to dispose of project files.

After the validity of data in the field notes and on ER Program forms has been evaluated according to the procedures described above, the data administrator will tabulate the data, wherever possible, by entering the data in computer data files. All data hand-entered into computer files will be checked 100 percent by another individual. Where appropriate, the data files will be set up for direct input into the project data base. Subjective data will be filed as hard copies for later review by the installation manger and for incorporation into technical reports, as appropriate.

3.1.3 Data Collection Forms

As an added means of ensuring the collection of accurate field and sampling information, standardized data collection forms will be used. These forms will be used to record data in a consistent format that limits individual interpretations or preferences. By explicitly outlining reporting methods, identifying appropriate units of measure, and specifying alternative test procedures, these forms provide a measure of quality control in the data collection process.

The standard data collection forms associated with the Mound Plan ER Program SOPs group data and information according to problem-solving needs. They are a means of preventing the collection of invalid or redundant data and eliminating critical data gaps. Each data collection form precisely defines what data are necessary to accurately characterize a particular property or relationship. This reduces the likelihood of initiating field sampling or laboratory analyses only to discover that key pieces of information have not been collected and that further sampling is required.

Each Mound Plant ER Program SOP for data collection activity provides examples of all of the forms required for the accurate recording of the procedure. A blank form will be used for each new location or sample, as specified by the SOP. During the field activity, each form will be completed as accurately and completely as possible, as indicated by the example contained in the SOP. All entries on the data collection form will be made using indelible black ink, with incorrect entries crossed out with a single line and initialed. Each form must be signed or initialed and dated by the person completing the form on the day of information entry. Any additional information not recorded on the form will be recorded in the field notebook. After the field activity is completed, all data collection forms will be reviewed by a technical reviewer other than the person recording the data prior to any use of the data and sufficiently soon to take any necessary corrective action. This review will ensure that forms are completed fully and accurately and will verify the integrity of the data. After the review, the reviewer will sign and date each form.

3.1.4 Sample Tracking

Samples collected in the field should be tracked by the EG&G subcontractor using a PC-based spreadsheet system or equivalent database. The following information will be recorded:

- Laboratory batch number
- Field batch number

- Sample matrix
- Sample identification
- Sample type (investigative vs. Quality control)
- Date of collection
- Date of receipt for laboratory data
- Date of completion for data validation
- Requested analysis

Completed chains of custody should be submitted at the end of each week to the data administrator for entry into the sample tracking system. The sample tracking data are then checked for accuracy and completeness by another individual. When hardcopy data packages are received from the laboratory they will be inventoried and the laboratory batch number and analysis/extraction date will be recorded in the tracking system. When the electronic files are received from the laboratory, the date of receipt will be recorded in the tracking system if the data are successfully loaded into the database. When the data validators submit validated results, the date of completion will also be recorded in the sample tracking system.

3.2 Corrections to Documentation

All measurements made, and samples collected, will be recorded as described above in Subsections 3.1.2 and 3.1.3. If an incorrect entry is made to the original of the data document, the incorrect data will be crossed out with a single strike mark, the correct information entered, and the correction initialed and dated by the person making the correction. There will be no erasures or deletions from any type of data document record.

3.3 Final Evidence File Documentation

Records will be kept by the ER Program EG&G subcontractor to document the quality assurance/quality control activities and to provide support for possible evidentiary proceedings. The following is an outline of project file requirements:

Communications
- Internal
- External

Quality assurance/quality control
- Procedures
- Chain of custody
- Audit reports
- Laboratory quality control reports
- Deviation notification forms
- Nonconformance/corrective action reports

Technical information
- Analytical data
- Field data
- Field logbooks

- Graphic resources
- Data quality acceptance
- Calculations/evaluations
- Data review reports
- Regulatory compliance

Management
- Schedule
- Budget
- Release site data base

Health and Safety
- Plans/procedures
- Audit reports

Documents
- Plans
- ports

All evidence file documentation will be maintained by the DOE or its subcontractor under the ER Program document control system. Upon termination of the project, all records (e.g., chromatograms, spectra, and calibration records) will be archived indefinitely by the DOE. If at any time the DOE chooses to purge its files, the EPA will be advised and offered possession. The ER Program EG&G subcontractor quality assurance manager will ensure that the quality assurance/quality control records are properly stored and retrievable.

DATA VALIDATION METHODS

FIELD STANDARD OPERATING PROCEDURES

FIELD STANDARD OPERATING PROCEDURES

The field procedures were included in the compendium to provide continuity between the Potential Release Site Investigations and the OU9 Site-Wide Investigation. Many of the field procedures, as identified in the table of contents for this section. were copied from Appendix A of the RI/FS Operable Unit 9, Site-Wide Quality Assurance Project Plan. Unlike the preceding sections of the compendium, the ER procedures have not been identified with a section prefix. The ER procedures will continue to be named following the convention introduced in the OU9 QAPP.

As new procedures are required, the procedures will be introduced into the compendium. Each new procedure will clearly reference the first approved sampling plan and date of the approved sampling plan. When a new procedure is added the revised table of contents and the procedure will be distributed to the copy holders of the compendium.

FIELD STANDARD OPERATING PROCEDURES — TABLE OF CONTENTS