

QUALITY ASSURANCE PROJECT PLAN  
SHORT-TERM LIMITED SITE INVESTIGATION  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE

PROVIDENCE, RHODE ISLAND

DECEMBER 2005

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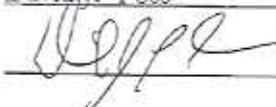
1.0 TITLE AND APPROVAL PAGE

Document Title      QUALITY ASSURANCE PROJECT PLAN  
SHORT-TERM LIMITED SITE INVESTIGATION  
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REVISION 1.2

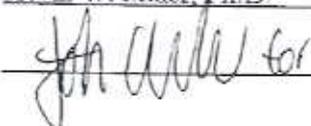
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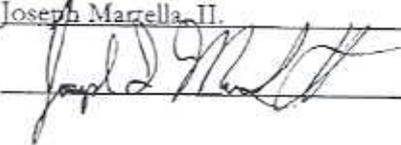
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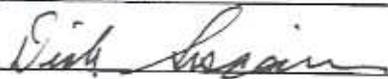
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QUALITY ASSURANCE PROJECT PLAN  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
PROVIDENCE, RHODE ISLAND

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 TITLE AND APPROVAL PAGE .....	Front of Document
2.0 PROJECT ORGANIZATION AND RESPONSIBILITY .....	1
3.0 INTRODUCTION .....	2
3.1 Site History and Description.....	2
3.2 Regulatory Background .....	3
3.3 Project History and Description .....	3
3.4 Plan Objective and Data Quality Objectives.....	4
4.0 SAMPLING DESIGN .....	5
4.1 Sampling Design Rationale .....	5
4.2 Number of Samples .....	5
4.3 Sampling Location.....	6
4.4 Project Schedule .....	6
5.0 SAMPLING AND ANALYTICAL PROCEDURES AND REQUIREMENTS.....	6
5.1 Sampling Procedures.....	6
5.1.1 Preventive Maintenance –Field Equipment.....	6
5.1.2 Calibration –Field Equipment .....	7
5.2 Analytical Procedures.....	8
5.2.1 Preventive Maintenance –Laboratory Equipment and Calibration.....	8
5.3 Laboratory Data Package Deliverables .....	8
6.0 SAMPLE HANDLING AND CUSTODY REQUIREMENTS.....	8
6.1 Documentation of Field Activities.....	8
6.2 Sample Identification .....	9
6.3 Sample Location Identification .....	9
6.4 Sample Labels.....	10
6.5 Field Data Sheets.....	10
6.6 Chain-of-Custody Records.....	10
6.7 Fuss & O'Neill Sample Logbook .....	11
6.8 Sample Containers and Preservation .....	11
6.9 Sample Custody at the Laboratory.....	12
7.0 QUALITY CONTROL REQUIREMENTS .....	12
7.1 Equipment Blanks.....	12
7.2 Trip Blanks .....	13
7.3 Duplicate Samples.....	13
7.4 Field Sample Control Limits.....	13

QUALITY ASSURANCE PROJECT PLAN  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
PROVIDENCE, RHODE ISLAND

TABLE OF CONTENTS (continued)

<u>SECTION</u>	<u>PAGE</u>
7.5 Laboratory Internal Quality Control .....	14
8.0 DATA MANAGEMENT AND DOCUMENTATION .....	14
8.1 Introduction .....	14
8.2 Data Classes.....	14
8.2.1 Field Data.....	14
8.2.2 Analytical Data .....	15
8.2.3 AutoCAD Drawings.....	15
8.2.4 Graphical Outputs.....	15
8.3 Data Administration.....	15
8.3.1 Field Documentation .....	15
8.3.1.1 Chain-of-Custody .....	15
8.3.1.2 Field Data Sheets .....	15
8.3.1.3 Field Logs .....	16
8.3.2 Internal Data Management.....	16
8.3.2.1 Data Management System and AutoCAD Basemap.....	16
8.3.2.2 Field Sampling Data .....	16
8.3.2.3 Original Laboratory Data .....	16
8.3.2.4 Data Management System (DMS) Data Entry.....	16
8.3.2.5 Query Requests .....	17
8.3.2.6 Database Verification.....	17
8.3.2.7 DMS Output for QA Interpretation.....	17
8.4 Data Presentation .....	17
9.0 ASSESSMENTS AND RESPONSE ACTIONS.....	18
10.0 PROJECT REPORTS .....	18
10.1 Introduction .....	18
10.2 Project Status Reports.....	19
10.3 Internal Assessments and Response Actions .....	19
10.3.1 Field Performance and Audits .....	19
10.3.2 Laboratory Performance and Audits.....	19
10.4 Project Results .....	19
11.0 DATA VERIFICATION AND VALIDATION .....	20
11.1 Levels of Data Validation Review.....	20
11.2 Evaluation Criteria.....	22
11.2.1 Precision .....	22
11.2.2 Accuracy.....	23
11.2.3 Completeness .....	24
11.2.4 Representativeness.....	24

QUALITY ASSURANCE PROJECT PLAN  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
PROVIDENCE, RHODE ISLAND

TABLE OF CONTENTS (continued)

<u>SECTION</u>	<u>PAGE</u>
11.2.5 Comparability .....	25
12.0 DATA USABILITY .....	25
13.0 REFERENCES.....	25
 TABLES	 END OF TEXT
Table 3-1 Summary of Previous Environmental Investigation Activities	
Table 3-2 Summary of Action Levels and Reporting Limits	
Table 3-3 Summary of Sediment Ecological Screening Criteria and Laboratory Reporting Limits	
Table 5-1 Fuss & O'Neill, Inc., Standard Operating Procedures, Sample Collection	
Table 5-2 Fuss & O'Neill, Inc., Standard Operating Procedures, Field Equipment Calibration and Preventive Maintenance	
Table 5-3 Fuss & O'Neill, Inc., Field Equipment Preventive Maintenance	
Table 5-4 Fuss & O'Neill, Inc., Field Equipment Calibration and Corrective Action	
Table 5-5 Method and SOP Reference Table, Premier Analytical Laboratory and Severn Trent Laboratories	
Table 5-6 Sampling and Analytical Methods Requirements	
Table 5-7 Preventive Maintenance Schedule, Premier Analytical Laboratory	
Table 5-8 Preventive Maintenance Schedule, Severn Trent Laboratories	
 FIGURES	 END OF TEXT
Figure 2-1 Project Management Organizational Chart	
Figure 3-1 Site Location Map	
Figure 4-1 Proposed Sampling Locations	
Figure 8-1 Field Documentation Flow Chart	
Figure 8-2 Environmental Data Management Flow Chart	
 APPENDICES	 END OF TEXT
Appendix A Quality Assurance Project Plan Writer/Reviewer Checklist	
Appendix B Data Validation Completeness Checklist	
Appendix C Fuss & O'Neill, Inc. Standard Operating Procedures for Sample Collection	
Appendix D Fuss & O'Neill, Inc. Standard Operating Procedures for Field Equipment Operation	
Appendix E Premier Analytical Laboratory Standard Operating Procedures for Analytical Methods	

QUALITY ASSURANCE PROJECT PLAN  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
PROVIDENCE, RHODE ISLAND

TABLE OF CONTENTS (continued)

APPENDICES

END OF TEXT

- Appendix F Severn Trent Laboratories, Inc. Standard Operating Procedures for Analytical Methods
- Appendix G Sample Label Format, Field Data Sheets and Chain-of-Custody Forms

## 2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The following is a list of key personnel for the sampling program to be implemented in this study. Any questions regarding project status should be directed to the principal contact. A project management organization chart is provided as Figure 2-1.

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### 3.0 INTRODUCTION

The following Quality Assurance Project Plan (QAPP) provides a framework for assessing the quality of data obtained from the environmental sampling programs at the Former Gorham Property and Mashapaug Cove in Providence, Rhode Island. The format for this document was developed from the United States Environmental Protection Agency (US EPA) Brownfields Quality Assurance Project Plan Guidance Document. This document defines the field and laboratory procedures to be used for this project and contains several attachments including field sampling Standard Operating Procedures (SOPs), laboratory SOPs, and field sampling forms. A Quality Assurance Project Plan Writer/Reviewer Checklist, is included in Appendix A.

#### 3.1 Site History and Description

The former Gorham Manufacturing Facility is situated on 37 acres (see Figure 3-1). The portion of the site that is involved in this investigation ("the subject site") is approximately 10 acres, including Mashapaug Cove. It is bordered by Stop & Shop to the east and Adelaide Avenue and a residential neighborhood to the south. The site slopes toward Mashapaug Pond and Mashapaug Cove to the north and west with an elevation change of approximately 30 feet.

The subject site is currently vacant, and surrounded by a chain link fence. Buildings that once stood on the subject site have been razed and demolition debris has been removed. A portion of the subject site in the southwest is paved and is referred to as the Western Parking Area. The area adjacent to the cove is referred to as the North Bank Area. The western peninsula of the subject site has been referred to as the Park Parcel.

The Western Parking and North Bank Areas are underlain by heterogeneous fill consisting of granular, reworked soils with lesser amounts of casting sands, coal, coal ash, slag, asphalt, bricks, pipes, wood, cloth, glass, canisters, and occasional crushed, empty drums. Thickness of the fill is approximately one foot in the Western Parking Area and increases to approximately 20 feet in the North Bank Area.

The Park Parcel is approximately eight acres and follows the shoreline of Mashapaug Pond on the north and west sides of the property. A large part of this parcel is wooded and vegetated with poorly defined trails parallel to the pond shoreline. No active industrial or commercial activities are documented to have occurred within the Park Parcel; however, the north bank of the Cove is an area of exposed fill material. VOCs, PAHs, metals and TPH have been associated with this fill material.

Historically, fuel oil was stored in several underground storage tanks (USTs) in the central portion of the property. Many of the tanks have been removed or decommissioned. The closure and assessment of the USTs have been documented in previous submittals to the Rhode Island Department of Environmental Management (RIDEM).



Between 1890 and 1986 sterling silver and plated silverware as well as bronze castings were manufactured on-site. Operations included casting, rolling, polishing, lacquering, forging, plating, annealing, soldering, degreasing, machining and melting. Trichloroethene (TCE), tetrachloroethene (PCE), and 1,1,1-trichloroethane (TCA) were reportedly used as vapor degreasers.

Ownership of the property has changed several times since 1967. It is currently owned by the City of Providence.

Groundwater beneath the subject site is classified by RIDEM as GB. There are no public or private wells within a four-mile radius. The nearest public water supply is the Scituate Reservoir located approximately nine miles to the west. Mashapaug Pond has been classified as Class C.

### 3.2 Regulatory Background

The site is identified on the CERCLIS List as the Gorham Manufacturing Site, Number RID982542318. A summary of the actions taken by RIDEM follows.

November 1987	EPA Potential Hazardous Waste Site Identification Form completed by RIDEM in response to a complaint from the Providence Police Department
June 1989	Preliminary Assessment completed by RIDEM designated site as Medium Priority for Site Inspection
February 1993	Site Investigation Report prepared by Camp, Dresser and McKee under contract to RIDEM

### 3.3 Project History and Description

Environmental investigations and remedial activities at the site began in 1985. A summary of these activities is provided in Table 3-1.

Environmental conditions at the site recognized in the "Site Investigation Summary Report and Risk Assessment" prepared by Harding Lawson Associates in July 1999 include:

- Two contiguous groundwater plumes: one extending to the north and one extending to the east from the vicinity of the former Building W. Constituents of concern in the plumes primarily include TCE, PCE, and TCA
- Fill beneath the West Parking and North Bank Areas contains variable concentrations of polycyclic aromatic hydrocarbons (PAHs), VOCs, metals (arsenic, beryllium, copper, chromium, lead, silver, and zinc), and total petroleum hydrocarbons (TPH)
- Concentrations of arsenic, beryllium, copper, lead, and benzo(a)pyrene in the fill have exceeded RIDEM Method 1 Industrial Direct Exposure Criteria



- Locally, low concentrations of PCE, TCE, and lead were detected in shallow groundwater suggesting sporadic, localized sources in the fill
- PAHs and metals reported in surficial soils have been attributed to the fill material
- TPH-contaminated soil and non-aqueous phase liquid (NAPL) were identified near former oil tanks on the Former Gorham Manufacturing site. This contamination was outside of the subject site, and is currently being monitored and remediated by Textron

The City of Providence has proposed to develop the subject site with a public school and a YMCA facility. The scope of work for this investigation includes:

- Collecting sediment samples at up to five locations within the Mashapaug Cove
- Collecting shallow soil samples (0-2 feet) at up to five locations in areas of apparently undisturbed soil across the portion of the subject site proximal to Mashapaug Cove
- Submitting samples to analytical laboratories for analysis of TPH, VOCs, SVOCs, metals, cyanide, PCBs, and/or dioxins/furans

Upon receipt of the analytical laboratory reports, the data will be imported into a relational database as described in Section 8.3. Data will be validated for completeness, precision, accuracy and representativeness. In addition, data will be compared to applicable published benchmarks and standards.

### 3.4 Plan Objective and Data Quality Objectives

This QAPP is designed to guide quality management of surface soil and sediment sampling at the subject site. The objective of this project is to evaluate the nature of potential contaminants present in sediment within the Mashapaug Pond cove and shallow soil on the former Gorham Facility property.

Soil data will be compared to the RIDEM Residential Direct Exposure and GB Leachability Criteria as published in the RIDEM Remediation Regulations. Sediment data will be compared to published ecological screening values. These values represent concentrations at or below which ecological effects are not expected to occur. The primary source of ecological screening criteria was MacDonald et al. (2000). Because MacDonald et al (2000) did not include some of the constituents of potential concern, other published sources were used to identify conservative screening values for those compounds. Tables 3-2 and 3-3 list the criteria, published sources, and the typical reporting limits provided by Premier and Severn Trent Laboratories.

The generation and use of quality data is important in the assessment of constituent impact on the site and, if necessary, in the selection of adequate responses to concentrations in soil and sediment. These data quality objectives will be documented following each sampling event on a Data Validation Completeness Checklist, included in Appendix B. The checklist will be an



internal document which will be used to record key aspects of data quality thus ensuring that the data quality objectives are met.

#### 4.0 SAMPLING DESIGN

##### 4.1 Sampling Design Rationale

The goal of this project is to identify and characterize any releases of hazardous substances at the site. Tasks required for the completion of this project may include:

- Prepare a project-specific Health and Safety Plan (HASP) in accordance with applicable OSHA requirements and Fuss & O'Neill's standard procedures for the range of compounds of concern and anticipated safety issues at the site
- Complete a limited site investigations in the area of Mashapaug Cove to evaluate the nature of the site where contaminated or polluted soils or sediments may be present
- Validate laboratory analytical data according to EPA Region I Data Validation Protocols (Modified Tier II)
- Evaluate data relative to published sediment benchmarks, RIDEM Residential Direct Exposure Criteria, and RIDEM GB Leachability Criteria
- Prepare a summary report documenting results
- Use results to help determine if the presence of contaminated or polluted soils and sediment may impact human health or pose ecological risk
- If applicable, summarize the options available to remediate the site in accordance with the Remediation Regulations

##### 4.2 Number of Samples

Soil samples will be submitted to Premier Laboratory for analysis of priority pollutant 13 metals by EPA Methods 6010/7470, cyanide by EPA Method 9012A, and PCBs by EPA Method 8082. Sediment samples will be submitted to Premier for analysis of TPH by Standard Method 8100M, VOCs by EPA Method 8260, SVOCs by EPA Method 8270, priority pollutant 13 metals by EPA Methods 6010/7470, cyanide by EPA Method 9012A, pesticides by EPA Method 8081A, and PCBs by EPA Method 8082. Sediment samples will also be submitted to Severn Trent Laboratory for analysis of dioxins and furans by EPA Method 8290.

A minimum of five but no more than ten soil samples will be collected and submitted from five sample locations.

A minimum of five but no more than ten sediment samples will be collected and submitted from five locations within Mashapaug Cove.

### 4.3 Sampling Location

Proposed sampling locations are depicted on Figure 4-1.

### 4.4 Project Schedule

The following schedule for the proposed scope of work will be implemented upon receiving US EPA and RIDEM approval for this workplan. The dates used in this schedule are based on receipt of approval on or before December 23, 2005. The schedule is subject to change if approval is received after that date, or based on the results of sampling activities or unanticipated field conditions.

Former Gorham Property and Mashapaug Cove Quality Assurance Project Plan Proposed Schedule		
Task / Description	Approximate Start Date	Approximate Duration
Sediment sampling	December 28, 2005	1 day
Shallow soil sampling	December 29, 2005	1 day
Laboratory Analysis	December 30, 2005	2 to 4 weeks
Data Management & Reporting	Upon receipt of final laboratory report	4 weeks
Total		8 weeks

## 5.0 SAMPLING AND ANALYTICAL PROCEDURES AND REQUIREMENTS

### 5.1 Sampling Procedures

The Standard Operating Procedures (SOPs) pertaining to sample collection (soil and sediment) are referenced in Table 5-1 and included in Appendix C.

#### 5.1.1 Preventive Maintenance – Field Equipment

The SOPs pertaining to preventive maintenance of field equipment are referenced in Tables 5-2 and 5-3 and included in Appendix D.

Field measurement equipment will be maintained in accordance with Fuss & O'Neill SOPs and manufacturer instructions. The field personnel will be responsible for confirming that equipment is operating properly during use by inspecting the instrument for physical damage and conducting instrument performance checks. Problems encountered in the field with field measurement equipment will be documented in the field logbook. An attempt will be made to correct problems with the instrument while on site. If the instrument's problem is not remedied in the field, it will be taken out of service and replaced, if feasible.

The field measurement equipment is inspected, cleaned, and calibrated on a daily basis prior to leaving Fuss & O'Neill. The calibration information is recorded on calibration forms which are kept at Fuss & O'Neill. The daily preventive maintenance checklist includes the following:

- Visual battery check of battery low indicator
- Replacement of batteries as necessary (spare batteries are kept on hand at Fuss & O'Neill)
- pH electrode is cleaned and calibrated on a daily basis and cleaned and re-calibrated after a noticeably dirty sample (full decontamination)
- Temperature is checked using a certified calibrated thermometer, and, if necessary, the sensor is replaced
- If the specific conductance electrodes are determined to be losing their platinum coating, the electrodes are replatinized according to manufacturer's instructions with 3 percent chloroplatinic acid and the instrument is then re-calibrated
- The conductivity electrode of the SC meter will be cleaned on a daily basis and cleaned and re-calibrated after a noticeably dirty sample (full decontamination)

#### 5.1.2 Calibration – Field Equipment

SOPs for the calibration of field instruments to be used during this project are referenced in [Table 5-2](#) and included in [Appendix D](#). Calibration frequency, acceptance criteria and corrective action are included in [Table 5-4](#).

Field instrument calibration is performed consistent with EPA Region I Draft Calibration of Field Instruments (EPA, June 1998) and in accordance with manufacturer's instructions. Soil samples will be screened using an Organic Vapor Monitor. Surface water sampling will include the measurement of pH, conductivity, dissolved oxygen and temperature by portable field instruments for field screening purposes only. Such measurements are conducted in the field because immediate or in-situ measurement provide results that are more representative of site conditions.

Field instruments will be calibrated before initial use. The instruments will be re-calibrated mid-day and at the end of a daily sampling event. Calibration will be recorded in field logbooks or the calibration log maintained in the Field Operations area at Fuss & O'Neill as appropriate.

Calibration acceptance criteria for field instrumentation have been established by the respective instrument manufacturer and are listed in [Table 5-4](#). If a calibration check determines that any field instrument is outside of the criteria, the instrument will be re-calibrated. If a calibration cannot occur within the acceptance criteria a back-up instrument will be used, if feasible.



## 5.2 Analytical Procedures

Two contract laboratories will be used for this project: Premier Laboratory in Dayville, Connecticut and Severn Trent Laboratory in Sacramento, California. The laboratory SOPs for analytical methods for this project and the laboratory Quality Systems Manual are included in Appendices E and F. Analytical method SOPs are also referenced in Table 5-5.

Where applicable, data collection and laboratory samples for this project will be analyzed in accordance with SW-846 (U.S. EPA, 1986) or EPA (U.S. EPA, 1983b) analytical methods. Sampling and analytical method requirements including container types, preservation and hold times are listed in Table 5-6.

### 5.2.1 Preventive Maintenance – Laboratory Equipment and Calibration

Preventive maintenance, calibration, measurement systems, and corrective action procedures for laboratory equipment are provided in Tables 5-7 and 5-8. Laboratory SOPs are provided in Appendices E and F.

## 5.3 Laboratory Data Package Deliverables

Data package deliverables from the contract laboratory will consist of the following elements:

- Cover letter/letter of transmittal signed by project manager of designee
- SDG narrative signed by project manager or designee
- Field and internal laboratory chain-of-custody records
- Data results sheets
- Laboratory matrix spike and matrix spike duplicate results
- Laboratory control samples and control duplicate results
- Method blank results
- Surrogate recoveries
- Serial dilutions
- Modified Tier II data validation checklists

## 6.0 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

### 6.1 Documentation of Field Activities

All field personnel will carry a bound, water-resistant field notebook in which to record observations during field activities, regardless of whether or not those activities involve sample collection. The field notebook will document site-specific information such as:

- Project name and location
- Time and date of arrival at the site
- Sampling locations and corresponding sample numbers
- Conversations with individuals on site



- Any unusual events or observations
- All information not recorded on field data sheets, including field calibration of equipment
- Time of departure from the site

For field investigations that involve the collection of samples, additional forms of documentation are required. These additional forms are discussed in the following sections.

At the end of a sampling event, or at intervals requested by the project manager, all documentation will be provided to the Project Manager or his/her designee for comprehensive review to ensure that all intended samples were collected and to verify that proper sampling methodologies were employed.

## 6.2 Sample Identification

Each sampling location will be assigned a unique number by which samples can be identified. An example of a sample identification number is as follows:

402 040401-01

This 11-digit code contains three types of information about the collection of the sample. The first three numbers in the code represent the identification number of the individual who collected the sample or supervised the sampling event. This is followed by the year, month and day of sample collection. The last two numbers refer to the site sampling order. If necessary, the number can be expanded to three digits; this should be determined before sampling begins in order to assure that all samples have the same format.

Samples collected at one time from the same sample location will have the same sample identification number. Some common situations are listed below:

- Multiple containers required for various analytical parameters
- Sampling events which require the sample to be submitted to two or more laboratories, as in the case of a split sample discussed below
- Sampling events that require sampling at the same location over two or more days due to insufficient sample volume available per day

## 6.3 Sample Location Identification

Each sampling location will be assigned a unique identifier (site ID). The Site ID will be recorded on the field data sheet in addition to the sample number and will primarily be used for graphical display of sample results as well as internal data handling.

In general, the Site ID will consist of a one- or two-letter prefix referring to the type of media sampled and a serial number indexing the sampling location. The following prefixes will be used for the indicated sampling media:



SD Sediment  
SS Surficial Soil

Sample locations within each class of sampling media will be assigned a two- or three-digit serial number in the order in which they are sampled. Examples of sample location IDs are as follows:

SD-12 Sediment sampling location 12  
SS-01, SS-02, SS-03 Surficial soil locations 01, 02, and 03

Samples collected at multiple depths at the same location will have the same sample location ID. The depth or range of depths at which samples are collected will be recorded on the appropriate field data sheets and chain-of-custody forms. Sample depth information will also be included on data tables.

#### 6.4 Sample Labels

A sample label will be affixed to each sample container at the time of collection. The following information will be recorded on each label with waterproof ink:

- Sample identification number
- Client/project name
- Project location
- Project number
- Date of sample collection
- Time of sample collection
- Name of sample collector
- Type of preservation

An example of a sample label is included in Appendix G.

#### 6.5 Field Data Sheets

Samples will have a field data sheet documenting their collection. This record will be completed by field personnel at the time of sample collection. Examples of sampling field data sheets are provided in Appendix G.

#### 6.6 Chain-of-Custody Records

Control of samples shall be maintained at all times. The chain-of-custody will be used to document all transfers of the sample between the sample collector and the laboratory. The form, consisting of four copies, will include the following information:

- Chain-of-custody identification number



- Project/client name
- Project location
- Project number
- Laboratory conducting analysis
- Name/location of party to receive laboratory report
- Name/location of party to receive laboratory invoice
- Sample identification number
- Analysis to be performed
- Sample type
- Number and type of sample containers
- Type of preservatives
- Signature and affiliation of sampler
- Date and time of collection
- Signatures of people involved in chain of possession
- Dates and times of sample transfers

The samples will remain in the custody of the sample collector until that person relinquishes them to the laboratory or sample delivery person. To reduce the potential for sample tampering, coolers will be shipped to the laboratory with chain-of-custody seals on the edges of the cooler between the lid and the sides. One copy of the chain-of-custody record will be retained by Fuss & O'Neill personnel, while the original and two copies will be relinquished with the samples. A completed record will be returned to Fuss & O'Neill with the sample analysis report. An example chain-of-custody form is presented in [Appendix G](#). Copies of all shipping papers will be retained by the sampler.

#### 6.7 Fuss & O'Neill Sample Logbook

The Fuss & O'Neill sample logbook serves as a central repository of general information on all environmental samples collected by Fuss & O'Neill personnel. The sample logbook is located in Environmental Field Services office area. Samples collected in the field which are relinquished to a fixed laboratory will be recorded in the sample logbook. Samples collected in the field and given directly to the mobile lab for analysis will be recorded in the mobile laboratory sample logbook. Information recorded in the logbook includes sample number, job number, job name, sample location ID, laboratory, date relinquished, and date received.

#### 6.8 Sample Containers and Preservation

Samples will be collected in lab-supplied containers. The type of container utilized will depend upon the analysis to be conducted as indicated in the Quality Assurance Plans for the laboratories found in Appendices E and F and referenced in [Table 5-6](#). Containers will be pre-cleaned and certified clean (I-Chem 300 or equivalent).

Once the samples have been collected and labeled, they will be placed in a cooler to reduce the sample temperature to less than 4°C. Sample temperature upon reaching the laboratory is dependent on how much time has passed since the sample was collected and placed in the

cooler. It is possible that the time between sample collection and delivery to the laboratory may not be long enough for the sample to reach 4°C. If samples are not relinquished to the laboratory at the end of the day, they are to be transferred and stored by Fuss & O'Neill in a refrigerated environment (maintained nominally at 4°C) until they are relinquished to the contract laboratory.

#### 6.9 Sample Custody at the Laboratory

Samples for analytical chemistry will be delivered to the contract laboratory for this project. An internal chain-of-custody form will be generated as the samples are assigned locations within the specific laboratory. Procedures for sample log-in, internal sample tracking and sample disposal at the contract laboratory are included in Appendices E and F.

### 7.0 QUALITY CONTROL REQUIREMENTS

The number and type of Quality Assurance and Quality Control (QA/QC) samples submitted to the laboratory will be project specific.

QA/QC samples submitted to the laboratory will include equipment blanks, trip blanks and field duplicates. The purpose of these samples is to confirm that laboratory results reflect the condition of the various media in the environment and are not the result of poor sampling or laboratory technique. Additionally, duplicate samples will be collected in order to check the precision of the laboratory, also detailed below.

Each QA/QC sample will be given its own sample code. The identity of these samples will be withheld from the laboratory conducting the analysis. When more than one QA/QC sample is submitted with a set of samples, they will be interspersed within those samples so that they are not easily identifiable by the laboratory.

#### 7.1 Equipment Blanks

Equipment blanks will be collected for sampling events in which non-dedicated sampling equipment is used. Equipment blanks may be obtained from equipment that has a potential to come into direct contact with samples. Equipment blanks are generally obtained from sampling equipment which is decontaminated between sample locations.

Blanks are prepared in the field during the sampling event. Laboratory-supplied deionized water is run through the decontaminated equipment which has been utilized during sampling. This water will then be transferred from that piece of equipment to the sample container.

Equipment blanks will be analyzed for the same parameters as samples collected with the piece of equipment used. For most parameters, the sampling device is the only piece of equipment that comes into contact with the sample.

The frequency of equipment blank collection will be a minimum of one per 20 samples by piece of equipment.

## 7.2 Trip Blanks

Trip blanks for VOCs will be obtained for each day of sampling to determine whether samples have been exposed to contamination as a result of sample container handling or transport. Trip blanks will be submitted and analyzed only if VOCs make up a portion of the analyte list on the day of sampling. The blank will be prepared by the laboratory and will accompany the sample containers from the time they leave the lab until the time it will be returned to the lab as a sample. The trip blank sample is labeled as a sample and submitted blind to the laboratory for analysis.

The frequency of trip blank submission will be one per day when VOCs will be analyzed. If more than one cooler contains VOC samples then one trip blank will be submitted in each cooler.

## 7.3 Duplicate Samples

Duplicate samples for analysis will be collected for soil and sediment samples to check the precision of the laboratory analysis. Duplicate samples will be collected at the same time as the original sample and will be analyzed for the same parameters. The duplicate sample will be assigned a different sample number than the original set so that the sample identity is blind to the laboratory.

The frequency of duplicate sample collection and submission will be a minimum of one per 20 samples per matrix.

## 7.4 Field Sample Control Limits

The standard Fuss & O'Neill field sample control limits for quality control are specified below. If the control limits are not met, the Quality Assurance Officer will investigate the cause of the exceedance and determine the validity of the associated data.

Quality Control Sample	Control Limit
Trip Blank	Less than detection limit*
Equipment Blank	Less than detection limit*
Field Duplicates	± 30% Percent Difference for Water; Acceptable Percent Difference will be equal to Matrix Spike Recovery Values for Matrices other than Water **
*	With the exception of common laboratory contaminants of acetone, 2-butanone, methylene chloride, phthalates and toluene which will have a control limit of 5X detection limit.
**	Discrepancies will be addressed on a case-by-case basis.



## 7.5 Laboratory Internal Quality Control

The laboratory Quality Assurance Plan explains the type of quality control checks which are routinely followed. This includes such items as analysis of client reference standards, matrix spikes, blanks, the use of internal standards and surrogate spikes. All calibrations are checked before sample analysis can begin. If the analytical system does not pass the initial QC limits, then the system is determined to be out of control and the cause of the problem must be determined and corrected before measurements can continue. Once the problem is corrected, QC measurements are repeated to verify the calibration. If the system still does not meet control limits, the system is re-examined until the problem is corrected. The QA/QC procedures and analytical precision and accuracy of the methods to be used for this project are provided in Appendices E and F.

## 8.0 DATA MANAGEMENT AND DOCUMENTATION

### 8.1 Introduction

This section defines the specific policies, organization, and procedures related to data management. The Data Management System that is being used for the electronic data management is the GIS/Key™ system. GIS/Key™ is a comprehensive environmental data management and visualization system that satisfies State and Federal reporting requirements by integrating database information, graphics, and analysis tools.

### 8.2 Data Classes

Many different classes of data may be generated during site investigations. Data generated include documentation of field activities, paperwork associated with environmental samples and investigations, and analytical data. The data classes include:

- Field Logbooks
- Sample Identification Numbers
- Field Data Sheets
- Fuss & O'Neill Sample Logbook
- Refrigerator Logbook
- Chain-of-Custody
- Analytical Data
- ArcView™ Drawings
- AutoCAD Drawings

#### 8.2.1 Field Data

Field data collected will be entered into a data management system. Further discussions of field data management are provided in Section 8.3.1 - Field Documentation.



## 8.2.2 Analytical Data

Analytical data generated by the contract laboratories will be entered into a data management system. Data generated for field screening may be entered into the data management system at the discretion of the Project Manager. Further discussions of analytical data management are provided in Section 8.3.2 - Internal Data Management.

## 8.2.3 AutoCAD Drawings

AutoCAD drawings produced for the project are electronically filed on two separate network servers. An example directory structure is for an AutoCAD drawing is J:\DWG\2005\1057\A10 and all associated subdirectories.

## 8.2.4 Graphical Outputs

An original copy of all graphical outputs, i.e. line graphs, bar graphs, three-dimensional graphs will be stored in the appropriate correspondence file.

## 8.3 Data Administration

Data administration as outlined below describes the flow of data, the plan for handling and presenting data and the internal data quality measures that will be implemented prior to final data output to tables, graphics and electronic output to other programs.

### 8.3.1 Field Documentation

Figure 8-1 describes the management of documentation generated in the field when collecting an environmental sample. The three sources of field data are the Chain-of-Custody, Field Data Sheets, and the Field Log.

#### 8.3.1.1 Chain-of-Custody

The COC will be generated in the field and travels with any environmental sample from the point of collection. When the samples leave the custody of field personnel, the field personnel will relinquish them (to refrigerator, cooler, other personnel or the laboratory). The orange copy of the COC will be retained by the personnel relinquishing the sample(s). The yellow copy will then be returned to the project hydrogeologist or engineer. A copy will be attached to the field data sheets and forwarded to the project staff.

#### 8.3.1.2 Field Data Sheets

Field data sheets are typically generated in the field and completed by field personnel. Field data sheets will be relinquished on a daily basis to the project staff who will perform a completeness review of the forms.

### 8.3.1.3 Field Logs

The field log is utilized throughout the day by field personnel. The field log will then be photocopied and the copy will be attached to the field data sheets and forwarded to the project staff. Field logs, when full, will be archived in the Environmental Field Operations area.

### 8.3.2 Internal Data Management

The following section describes data management, once field sampling has occurred, and follows the flowchart outlined in [Figure 8-2](#).

#### 8.3.2.1 Data Management System and AutoCAD Basemap

The Data Management System (DMS) will utilize AutoCAD drawing files as a geographic reference. Sample locations will be assigned X and Y coordinates. AutoCADTM base mapping of the site will be referenced into the site location drawing. Site locations will be designated by a symbol which describes the type of sample location, i.e., sampling well, soil boring, etc. These symbols are American Digital Cartography standard symbols. Once site location mapping is prepared, it can be utilized for other functions in the DMS, i.e., isopleth mapping, groundwater contour mapping, etc. The various types of available outputs are described in subsequent sections of this QAPP. Base mapping may be updated at any time with new sample locations as they are added to the program.

#### 8.3.2.2 Field Sampling Data

As indicated in [Figure 8-2](#), once field sampling has occurred, all field data sheets (including field staff logbook copies) will be forwarded to the Project Hydrogeologist or Engineer, who will review the field data for accuracy and completeness. If any field data sheets are incorrect or incomplete, they will be returned to field personnel for completion.

#### 8.3.2.3 Original Laboratory Data

As indicated in [Figure 8-2](#), when original laboratory data is received by the Project Hydrogeologist or Engineer, it will be reviewed for accuracy and completeness. Accuracy and completeness, as defined herein, means that the requested parameters were appropriately analyzed and that the Fuss & O'Neill sample numbers, date collected, date received, etc. are correctly identified on the laboratory report. Fuss & O'Neill may request that the laboratory deliver its report electronically by email or CD ROM, along with a hardcopy of the report.

#### 8.3.2.4 Data Management System (DMS) Data Entry

Once the field and laboratory data have been reviewed by project staff, the data will be submitted to the DMS operator to be entered into the DMS. Field data and laboratory data can be submitted and entered into the DMS independently of each other or together. For instance, it may be desirable to view groundwater contours immediately after a round of water levels

prior to the receipt of laboratory data. In this case, field measurements will be entered into the DMS and a groundwater contour map produced. Once analytical data are received, data entry will be independently scheduled for entry into the DMS.

#### 8.3.2.5 Query Requests

Query requests (questions asked of the database with resultant output) can be made at the time the data is submitted to the DMS operator for data entry into the DMS. Typical queries are described in Section 8.4.

#### 8.3.2.6 Database Verification

Once data entry is complete, a verification of the data will take place. This verification process will involve an individual visually comparing a hard copy of field data sheets and the laboratory analytical report with a hard copy printout of data from the data management system. This process will ensure that data has been accurately keyed/imported into the data management system prior to utilizing the data for analysis/interpretation.

#### 8.3.2.7 DMS Output for QA Interpretation

Once data entry and verification have been completed, a series of reports is available to evaluate data quality and to aid in data interpretation. The type of reports to be developed would be a function of the nature of the field investigation. These reports could include:

- Analytical data summaries
- Exceedances of applicable Maximum Contaminant Levels (MCLs) and Action Levels.
- Values that are outside of historical ranges.

Project staff, in conjunction with data management staff, will evaluate these reports to identify any anomalous data, outliers and QA/QC sample integrity. Once this data review has occurred, any necessary corrective action steps will be taken.

Once the data review process has been completed, the original field data sheets and analytical laboratory reports will be stamped with a "DMS" stamp and initialed by the person who conducted the verification of the data. The original data will then be filed in the job correspondence file.

### 8.4 Data Presentation

Data presentation must occur in a clear and logical format to accurately interpret and evaluate field and analytical data collected. Types of data that will be provided from the data management system include tabular data presentations, graphical data presentations, contour maps, geological cross sections, section view isopleths and three dimensional graphics.



## 9.0 ASSESSMENTS AND RESPONSE ACTIONS

The Project Manager is responsible for determining the need for and implementation of any corrective action measures to the sampling or analytical procedures. Corrective action will be implemented upon the identification of problems discovered through system audits by analytical data review. If a problem is identified, the QA Officer will:

- Report the problem to the Project Manager
- Evaluate the problem in accordance with data quality objectives
- Determine whether implementation of corrective action is required
- Assign and implement a corrective action
- Evaluate the effectiveness of the corrective action

The following is a list of possible occurrences that may require corrective action and the corresponding action that would likely take place.

- If any sample bottles break during transit such that insufficient sample is available to complete the analysis, that location will be re-sampled, if feasible, to replace the bottles that were broken
- If meters or other sampling equipment break or malfunction during sampling, efforts will be made to repair, re-calibrate, or replace them with back-up equipment
- If the analysis of trip or equipment blanks indicates the presence of target analytes above acceptable concentrations, re-sampling and reanalysis of samples taken that day may be required
- If there are unusual changes in detection limits, re-sampling and reanalysis may be recommended

Assessment and Response Actions for the contract laboratories are defined in Appendices E and F.

## 10.0 PROJECT REPORTS

### 10.1 Introduction

Project reports will be submitted to agencies specified by the client at a frequency specified by the client. At a minimum, project status reports will be submitted to RIDEM on a bi-weekly basis in the form of a \*.PDF via e-mail. In the event that field or project conditions are significantly different from those anticipated and outlined herein, such conditions will be verbally communicated to RIDEM as soon as is practical. RIDEM will communicate the change in conditions to EPA. Based on the relative scale of change in site conditions, the QAPP will be revised at the request of RIDEM and EPA.



The reports will detail project status, results of internal assessments, corrective actions implemented and project results are described as follows.

## 10.2 Project Status Reports

Due to logistical and weather-related problems, deviation from the project schedule may occur. Status reports may be necessary to ensure that all parties are informed. Status reports may include but will not be limited to:

- Progress of the project to date
- Revised project schedule
- Deviations from the work plan
- Analytical data

## 10.3 Internal Assessments and Response Actions

Performance and system audits are a qualitative evaluation of all of the components of the sampling program to ensure proper implementation and usage of the intended data gathering and associated QA/QC procedures. Internal assessments and response actions may be presented in letter reports or attached as an appendix in the final report. The content for these evaluations is outlined separately for Field and Laboratory Audits.

### 10.3.1 Field Performance and Audits

The person designated as the Quality Assurance Officer will check that the QAPP is being implemented. The Project Manager may designate the Project Hydrogeologist/Engineer as the QAO or the role may be filled by another individual who has experience in that capacity. Performance evaluations and audits are conducted routinely as part of the Data Management Plan explained in Section 8. The QAO examines sample log books, field instrument calibration logs, and field data sheets for accuracy and completeness.

### 10.3.2 Laboratory Performance and Audits

Laboratory performance and system audits are addressed in Appendices E and F.

## 10.4 Project Results

The reports describing project results will include information for developing a conceptual model of the site. The content of the report will include but will not be limited to:

- Summary of Investigation Tasks
- Summary of Internal Assessments and Corrective Actions
- Summary of Site Geology and Hydrogeology
- Summary of Analytical Information
- Exceedances of Numerical Criteria (will be highlighted in boldface)



- Recommendations

## 11.0 DATA VERIFICATION AND VALIDATION

The objectives of data validation are to:

- Assess and summarize the analytical quality and defensibility of data for the end user
- Document factors contributing to analytical error that may affect data usability, such as: data discrepancies, poor laboratory practices that impact data quality, site locations for which samples were difficult to analyze
- Document any "sampling error" that may be identified by the data validation process, such as contaminated trip or equipment blanks, incorrect storage or preservation techniques, improper sampling containers, and improper sampling techniques

For EPA Contract Laboratory Protocol (CLP) work, data validation must also accomplish the following additional objectives:

- Assist Regional EPA in monitoring CLP performance for contract administration purposes
- Assist EPA in monitoring any laboratory's performance of non-CLP methods in generating data for submittal to EPA
- Identify contractually non-compliant data that are unusable by EPA
- Provide information concerning the effectiveness of analytical methods
- Identify problems requiring method revision and/or resolution

### 11.1 Levels of Data Validation Review

To provide data of known quality, EPA has established three levels of data validation:

#### *Tier I*

- Review the data package to ensure that it contains all the required documents and forms
- Check that data are complete and accurate to ensure legal defensibility of the data
- Evaluate Performance Evaluation sample results to assess potential usability issues

#### *Tier II*

- Complete Tier I requirements and assess the results of all QC checks and analytical procedures
- Assess sample holding times, sample preservation techniques, matrix effects
- Identify potential problems originating from field sampling work by reviewing field duplicates, trip, and equipment blanks

- Discuss impact of laboratory or field problems on analytical data

#### *Tier III*

- Raw data are examined in detail to check for calculation, compound identification, and/or transcription errors

The details for each tier are provided in EPA's "Tiered Organic and Inorganic Data Validation Guidelines" (1993) and the "Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analysis" (1996) which contain definitions for the acronyms and data validation terminology.

Modifications of these levels of data validation are appropriate since the EPA Region I data validation process was developed in part to satisfy CLP deliverables. Sampling efforts to be conducted under this QAPP do not require CLP deliverables; therefore, modification of these levels of review may be appropriate for this project based on task specific DQOs.

Modified Tier II data validation is anticipated for this project.

In a Modified Tier II data validation, data collected during field operations and data deliverables provided by the analytical laboratory from standard EPA SW-846 method deliverables are reviewed by the end user.

The laboratory provides a "Modified Tier II Data Validation Checklist" with the data deliverables which certifies that SW-846 protocols were followed and that results were within the limits specified in the laboratory's Quality Assurance Plan. In addition, the laboratory certifies that all supporting documentation will be maintained at the laboratory for seven years and will be made available upon request of the end user. By certifying the data in this manner, the laboratory is not required to provide to the data validator all the data regarding analysis of standards and raw data but the validator is still able to complete Tier II Data Validation.

The data validator generates a Data Validation Checklist, applicable to a Modified Tier II data validation. A copy of the Modified Tier II Data Validation checklist is included in [Appendix B](#). The data validator will implement all corrective actions required when data is found to be inadequate.

Quality control checks and analytical procedures will be evaluated through an assessment of the precision, accuracy, completeness, representativeness, and comparability of the data set as defined in Section 11.2.

As part of a Modified Tier II validation, the laboratory will complete checklists to certify that data not provided are available and provide a project narrative summarizing QA/QC and other issues noted by the laboratory.

Data validation will be performed on fixed based laboratory results only; however, as a due diligence check, samples analyzed by Fuss & O'Neill using screening procedures will also be subjected to the completeness checklists presented in [Appendix B](#).

In general, validation should be completed within 21 days of receipt of the final data package from the laboratory. This enables the end user to assess contractual compliance and data usability in order to make timely site decisions. Accelerated site schedules may necessitate shorter turnaround times for validation. In general, the completion of a Data Validation Report should not be delayed because the laboratory failed to forward a resubmittal. In most cases, the Data Validation Report should be completed, the laboratory omission noted, and the data qualified using professional judgment. If a resubmittal is received, an amendment to the original Data Validation Report should be forwarded.

In some cases, the validator must wait for critical information before the validation can be completed. In these cases, the end user will be notified of the delay. If validation reports are time-critical, the end user may request that a partially completed Data Validation Report be generated. Subsequently, an amendment should be written to incorporate all late resubmittals.

## 11.2 Evaluation Criteria

### 11.2.1 Precision

Precision, which is defined as a measure of mutual agreement among individual measurements of the same property, can be described as reproducibility. In the case of laboratory analytical data, precision will be used to describe the reproducibility of the analytical data.

#### *Field Measurement Systems*

To assess precision in the field, a duplicate sample will be collected nominally for every 20 samples per matrix for all parameters. The collection of field duplicates measures a combination of field and laboratory precision, thereby exhibiting more variability than a laboratory duplicate. Calculation to determine Relative Percent Difference (RPD) between the two sample results is performed. RPD is used as a measure of precision. The laboratory will analyze duplicates on a one per 20 frequency, per matrix. Recovery limits are matrix and compound dependent.

RPD is defined as follows:

$$\frac{|\text{Conc}(p) - \text{Conc}(d)|}{(\frac{1}{2})(\text{Conc}(p) + \text{Conc}(d))} \times 100 = \text{RPD}$$

where,

Conc(p) = Primary Sample Concentration, the first sample collected at that location  
Conc(d) = Duplicate Sample Concentration, the second sample collected at that location

For water sample analysis, the percent difference goal for the field will be 30 percent. For matrices other than water, however, there are no established percent difference goals; therefore, as a guide for sample analysis of other matrices, the percent difference goal for the field will be



equal to the matrix spike percent recovery values. If a percent difference result falls outside the guidance range, the discrepancy will be addressed on a case-by-case basis since the results are laboratory, parameter, and matrix dependent.

*Laboratory Measurement Systems*

The objective concerning precision is to equal or exceed the precision demonstrated in the analytical methods on samples of similar matrix. RPD is used as a measure of precision. The laboratory will analyze matrix spikes/matrix spike duplicates on a one per 20 frequency, per matrix. Recovery limits are matrix and compound dependent.

RPD is defined as follows:

$$\frac{|MSR - MSDR|}{(\frac{1}{2})(MSR + MSDR)} \times 100 = RPD$$

where,

MSR = matrix spike recovery  
MSDR = matrix spike duplicate recovery

The absolute value of the recovery difference is used in the above equation.

For organic analysis, the laboratory will be spiking with the matrix spike compound list and utilizing the recovery limits defined in the analytical SOPs. Inorganics utilize the recovery limits defined in the analytical SOPs. If necessary, corrective action by the laboratory will be performed according to the provisions of their Quality Assurance Plan (Appendices E and F).

11.2.2 Accuracy

Accuracy can be defined as the degree of agreement of a measurement with an accepted reference or true value. Accuracy is generally expressed as the ratio of the measured value to the true value, which gives a measure of bias inherent in the system. Accuracy can be assessed both in the field and in the laboratory. The data validation completeness checklist in Appendix B will be used to document the field calibration and laboratory calibration for each sampling event.

*Field Measurement Systems*

Accuracy will be measured for field activities to assess the correct performance of the project measurement systems. Before initial use, the instruments will be calibrated to a known standard. Additionally, at the end of a daily sampling event, the instruments will be calibrated with a known control to assess the accuracy of field measurement systems. The calibration acceptance criteria are defined in Table 5-4.

### *Laboratory Measurement Systems*

The laboratory accuracy will be determined from spiked sample recoveries, published historical data, method validation studies, experience with similar samples, and project specific requirements. The goal for spiked sample recoveries will be the concentrations published in SW-846 Guidance Document. These concentrations vary from one compound to another.

#### 11.2.3 Completeness

Completeness is a measure of the amount of valid data obtained from each sampling event, compared with the amount which was expected to be obtained under correct conditions. A goal of 95 percent completeness of valid analytical results will typically be set for all samples collected. The completeness of the sampling data set can be evaluated as a percentage of the number of valid analytes to the total number of expected analytes.

The equation for determining the completeness of analytical results will be:

$$\frac{(\text{Total Number of Valid Results})}{(\text{Total Number of Results})} \times 100 = \text{Completeness}$$

Completeness of analytical results will be evaluated by analytical method.

If analytical data completeness is below 95 percent due to laboratory or field error, corrective action will consist of evaluation of the results in question as they pertain to the sampling program and possible re-sampling.

Data completeness for field measurement systems will be set at 100 percent. If an instrument fails, a back-up instrument will be used.

#### 11.2.4 Representativeness

Representativeness is defined as the degree to which data accurately and precisely represent a characteristic of a population. This can be stated as how well the chosen sampling points represent the actual conditions and variations within the study area. For example, if a batch of samples was broken in transit to the laboratory or the samples were compromised in some fashion at the laboratory, a representative sampling event may not have occurred. If this were the case, the need for a resampling event would be considered.

Data representativeness of both field and laboratory systems will be addressed through field and analytical procedures. Trip blanks will be evaluated for detectable contaminant concentrations. If contamination is present in the blank samples, the representativeness of the concentration in the sample media may be affected. Representativeness in laboratory measurement systems will be addressed by the Project Hydrogeologist or other qualified personnel who will record that the laboratory is using the proper analytical procedures and meeting holding times. The data



validation completeness checklist in Appendix B will be used to record that the proper procedures were followed in the field and in the laboratory for each sampling event.

#### 11.2.5 Comparability

Comparability is an expression of the confidence with which one data set can be compared to another. The comparability objective is to collect and analyze samples in a method which will address that the data are comparable to data collected in previous and future investigations for this study area. The comparability of data is addressed by using standard protocols for the collection of field samples and by using standard methodologies for analytical procedures which were used in past investigations. If, for instance, it is determined that the laboratory used a different method than one specified, an evaluation will occur and document whether this compromised the comparability of data. The data validation completeness checklist in Appendix B will be used to document the comparability of each sampling event.

### 12.0 DATA USABILITY

The purpose of this QAPP is to outline a systematic process and structure for data quality such that the data will support decisions. The generation and use of quality data is important in the assessment of constituent impact on the site and, if necessary, in the selection of adequate responses to concentrations in soil, groundwater, sediment, or surface water. The function of the data verification process is to identify sampling and "analytical error" and not to make final determinations about the overall usability of the data for the project (U.S. EPA, 1996). The usability assessment will be conducted by the QAO and the results of the assessment will be reported to the Project Hydrogeologist. The usability assessment will report how validated project data is reconciled with the project quality objectives and limitations, if any, of the data. Reconciliation may require resampling or recommending the use of selected data even through it did not meet the data quality objectives (DQOs). It is anticipated that laboratory reporting limits must be below applicable action levels to facilitate data usability.

For example, assume that a sample is analyzed for SVOCs by Method 8270. The QC for benzo(a)pyrene (BAP) is not acceptable, while the QC for all other SVOCs is acceptable. It is possible that the results may be usable for SVOCs excluding BAP.

Quality control issues will be discussed in the usability assessment and the QA Officer will recommend the use or rejection of the data. Ultimately, the end user will determine the usability of the data based on an understanding of the project data quality objectives and the results of the data verification process. The results of the usability assessment will be summarized in periodic reports developed annually.

### 13.0 REFERENCES

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# TABLES

Table 3-1  
Summary of Selected Previous Environmental Investigations  
Former Gorham Property and Mashapaug Cove

Title and Date	Author	Investigation Activities	RIDEM Submittal Date
<u>Environmental Assessment</u> , Adelaide Avenue Plant, Providence RI, May 17, 1988.	Goldberg Zoino & Associates	<ul style="list-style-type: none"> <li>• Installation of 6 monitoring wells (GZA-1 to 6).</li> <li>• Collection of Groundwater samples and surface water samples.</li> </ul>	Not submitted
<u>Soil and Groundwater Contamination Site Assessment of the Gorham Manufacturing Facility</u> , Providence, Rhode Island, May 25, 1989.	Hunter, Inc.	<ul style="list-style-type: none"> <li>• Excavation of 47 test pits (TP-1 through TP-47)</li> <li>• Drilling of 24 soil borings (B-1 to B-24)</li> <li>• Soil sampling and laboratory analysis</li> <li>• Installation of monitoring wells (MW-A to MW-R)</li> <li>• Collection of groundwater samples</li> </ul>	Not submitted
<u>Remedial Investigation Report</u> , Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, May 1995.	ABB Environmental Services Inc.	<ul style="list-style-type: none"> <li>• Ground penetrating radar survey</li> <li>• Wetlands delineation</li> <li>• Storm water runoff sampling (SR-001 and SR-002)</li> <li>• Permeability testing</li> <li>• Building N UST removal</li> <li>• Soil borings (TP-1 to TP-61 and SB-1 to SB-10)</li> <li>• Sampling of Building F crawl space soils (IN-t to IN-8)</li> <li>• Collection of north bank are fill drainage swale surface soil samples (SD-1 to SD-8)</li> <li>• Redevelopment and resampling of existing wells (September 1994)</li> <li>• Installation of 12 monitoring wells (MW-101 to MW-111)</li> <li>• Sampling of 12 RI wells (December 1994)</li> </ul>	May 1995

Table 3-1  
Summary of Selected Previous Environmental Investigations  
Former Gorham Property and Mashapaug Cove

Title and Date	Author	Investigation Activities	RIDEM Submittal Date
<u>Supplemental Remedial Investigation Report</u> Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, December, 1995.	ABB Environmental Services Inc.	<ul style="list-style-type: none"> <li>• Installation of monitoring wells MW-112, 113, and 114</li> <li>• Building W soil gas survey</li> <li>• Free product bail-down test (MW-K)</li> <li>• Groundwater sampling (September 1995)</li> <li>• Building W floor vault and trench inspection and sampling</li> </ul>	December 29, 1995
<u>Limited Design Investigation Report</u> Former Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, August 1996.	ABB Environmental Services Inc.	<ul style="list-style-type: none"> <li>• Building W vault and sump decontamination</li> <li>• Building W soil borings</li> <li>• Monitoring well installations (MW-115, 116, and 117)</li> <li>• Groundwater sampling (May 1996)</li> </ul>	August 20, 1996
<u>Short-Term Response Action</u> Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, July 1997.	ABB Environmental Services Inc.	<ul style="list-style-type: none"> <li>• Excavation and removal of oily soils from Building F crawl space.</li> </ul>	July 1997
<u>Groundwater Assessment and Bioremediation Screening Report</u> , Former Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, June 1998.	Harding Lawson Associates	<ul style="list-style-type: none"> <li>• Groundwater sampling</li> <li>• AFCEE bioremediation screening</li> </ul>	June 25, 1998
<u>Supplemental Investigation Report</u> Former Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, December 1998.	Harding Lawson Associates	<ul style="list-style-type: none"> <li>• Surface soil sampling on undeveloped parcel (SS-100 to 110)</li> <li>• North Bank area toe of fill GPS delineation.</li> </ul>	December 1, 1998.

Table 3-1  
Summary of Selected Previous Environmental Investigations  
Former Gorham Property and Mashapaug Cove

Title and Date	Author	Investigation Activities	RIDEM Submittal Date
<u>Site Investigation Summary Report and Risk Assessment</u> , Former Gorham Manufacturing Site, 333 Adelaide Avenue, Providence, Rhode Island, July 1999	Harding Lawson Associates	<ul style="list-style-type: none"> <li>• Excavation of exploratory test pits in the suspected groundwater VOC source area</li> <li>• Installation of additional monitoring wells for the delineation of the groundwater source area and eastern VOC plume</li> <li>• Collection of groundwater and surficial soil samples</li> <li>• Oversight of removal of two 30,000 gallon #6 fuel oil tanks</li> </ul>	August 1999
<u>Method 3 Human Health Risk Assessment – Park Parcel</u> , Former Gorham Manufacturing Facility, 333 Adelaide Avenue, Providence, Rhode Island, August 2004	MACTEC Engineering and Consulting, Inc.	<ul style="list-style-type: none"> <li>• Use historical data from the site to complete a human health risk assessment</li> </ul>	August 2004

NOTE: This table is not intended to be a comprehensive listing of all environmental investigations conducted on the site. Most of the information in this table was obtained from the July 1999 Site Investigation Summary Report and Risk Assessment prepared by Harding Lawson Associates.



Table 5-1  
Fuss & O'Neill, Inc.  
Standard Operating Procedures  
Sample Collection  
Former Gorham Property and Mashapaug Cove

SOP # and Appendix Reference	Project Sampling SOPs
010000	Site Etiquette
020000	Field Activity Documentation
020100	Sample Identification Numbers
020200	Sample Labels
020300	Field Data Sheets
020400	Chain of Custody Forms
020500	Analytical Parameter Request Forms
020600	Sample Logbooks
030000	Sample Handling
030100	Relinquishing Samples
040000	Decontamination Procedures
070000	Sediment Sampling
080000	Soil Sampling
080100	Soil Sampling – Scoop
080200	Soil Sampling – Hand Auger



Table 5-2  
Fuss & O'Neill, Inc.  
Standard Operating Procedures  
Field Equipment Calibration and Preventive Maintenance  
Former Gorham Property and Mashapaug Cove

SOP # and Appendix Reference	Project Equipment SOPs
150100	Fuss & O'Neill, Inc. SOP for Calibration and Maintenance of: YSI Model 63 SCT Meter
150200	Fuss & O'Neill, Inc. SOP for Calibration and Maintenance of: YSI Model 85 DO + SCT Meter
50300	Fuss & O'Neill, Inc. SOP for Calibration and Maintenance of: YSI-600 Series Water Analyzer

Table 5-3  
 Fuss & O'Neill, Inc.  
 Standard Operating Procedures  
 Field Equipment Preventive Maintenance  
 Former Gorham Property and Mashapaug Cove

Instrument	Activity	Frequency	SOP Ref.
YSI Model 63 SCT Meter	Battery replacement	As needed	150100
	pH probe cleaning	As Needed	150100
	Specific conductivity and temperature sensor cleaning	Daily	15100
YSI Model 85 DO + SCT Meter	Battery replacement	As Needed	150200
	Sensor cleaning	Daily	150200
YSI-600 Series Water Analyzer	Probe or probe port replacement	As needed	150300
	Battery replacement	As needed	150300
	pH probe and ORP sensor cleaning	As needed	150300
	Specific conductivity and temperature sensor cleaning	Daily	150300
	DO sensor cleaning and membrane replacement	As needed	150300

Table 5-4  
Fuss & O'Neill, Inc.  
Field Equipment Calibration and Corrective Action  
Former Gorham Property and Mashapaug Cove

Instrument	Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Ref.
YSI Model 63 SCT Meter	pH calibration	Every use*	±0.15 pH units	Recalibrate	150100
	SC calibration	Every use*	±25 micromhos/cm	Recalibrate	150100
YSI Model 85 DO + SCT Meter	DO calibration	Every use*	8 - 10 mg/L	Recalibrate	150200
	SC calibration	Every use*	±25 micromhos/cm	Recalibrate	150200
YSI-600 Series Water Analyzer	pH calibration	Every use*	±0.15 pH units	Recalibrate	150300
	SC calibration	Every use*	±25 micromhos/cm	Recalibrate	150300
	DO calibration	Every use*	8 - 10 mg/L	Recalibrate	150300
	ORP calibration	Every use*	±10 mV	Recalibrate	150300
* Field instrumentation is calibrated at the beginning, middle, and end of the day.					

Table 5-5  
Method and SOP Reference Table  
Premier & Severn Trent Laboratories  
Former Gorham Property and Mashapaug Cove

Analytical Method Reference		Project Analytical SOPs			
Appendix Reference	EPA Method Number	Document Title	Laboratory SOP Number	Effective Date	Revision Number
E	3500B	Organic Extraction and Sample Preparation	Doc. 07	3/21/00	1
E	3510C	Separatory Funnel Liquid-Liquid Extraction	Doc. 08	4/13/05	1.21
E	3545	Pressurized Fluid Extraction	Doc. 10	3/11/05	1.1
E	3650B	Acid-Base Partition Cleanup	Doc. 17	3/21/00	1
E	3665A	Sulfuric Acid Cleanup for PCBs	Doc. 19	5/6/05	1.1
E	8082	Polychlorinated Biphenyls by Gas Chromatography	Method 8082	3/12/02	1.1
E	8260B	Volatile Organics by Gas Chromatography	Method 8260B	4/24/05	2.21
E	8270C	Semi-Volatile Organics Analysis	Method 8270C	12/19/01	1.2
E	7471A	Mercury in Solids (Automated)	Method 7471A	1/24/04	2.2
E	6010B	ICP Metals	Method 6010B	1/21/04	3.1
E	-	Instrument Maintenance –Metals Department	-	8/24/99	1.1
E	-	Maintenance –Organics	-	3/28/01	1.1
E	335.3/SM4500 CN E	Determination of Total Cyanide	Doc. 20	4/2/01	2
E	9012A/SM4500-CN-C+E	Cyanide, Total	Doc. 35	7/19/01	1.1
E	-	Thielsch Engineering, Inc. Construction Materials Testing Lab: Particle Size Analysis of Soils	TEI-110-E	-	-
E	9060	Northeast Generation Services Analytical Lab: SW-846 Method	Method 9060	6/2/99	-

Table 5-5  
 Method and SOP Reference Table  
 Premier & Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Analytical Method Reference		Project Analytical SOPs			
Appendix Reference	EPA Method Number	Document Title	Laboratory SOP Number	Effective Date	Revision Number
		9060 TOC (Solid Samples)			
F	8290	Method 8290 and TO-9A – Polychlorinated Dioxins and Furans by HRGC/HRMS	SAC-ID-0005	12/15/02	6.0
F	8290	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry (HRGC/HRMS)	STL-SW8290	2/04	-

Table 5-6  
 Sampling and Analytical Methods Requirements  
 Former Gorham Property and Mashapaug Cove

Parameter	Matrix	No. of Sample Locations	Sampling SOP	Analytical SOP Ref. Table No.	Container	Preservation	Hold Time
VOCs	Sediment	5	070000	5-5	4 oz Glass/T	4°C	14 days
	Surficial Soil	5	080000	5-5			
TPH	Sediment	5	070000	5-5	4 oz glass/T	4°C	14 days(x)/ 40 days
	Surficial Soil	5	080000	5-5			
SVOCs	Sediment	5	070000	5-5	4 oz glass/T	4°C	14 days(x)/ 40 days
	Surficial Soil	5	080000	5-5			
PCBs	Sediment	5	070000	5-5	4 oz glass/T	4°C	14 days(x)/ 40 days
	Surficial Soil	5	080000	5-5			
Metals (total and dissolved)	Sediment	5	070000	5-5	4 oz Glass	4°C	6 months
	Surficial Soil	5	080000	5-5			
Pesticides	Sediment	5	070000	5-5	4 oz Glass	4°C	14 days(x)/ 40 days
	Surficial Soil	5	080000	5-5			
Dioxin/Furan	Sediment	5	070000	5-5	4 oz Glass	4°C	30 days (x) /15 days
	Surficial Soil	5	080000	5-5			

Notes:

1. All samples are to be stored at 4EC
2. T = Teflon lined cap
3. (x) indicates holding time prior to extraction

Table 5-7  
Preventive Maintenance Schedule  
Premier Laboratory  
Former Gorham Property and Mashapaug Cove

Instrument	Activity	Frequency	SOP Ref. *
VOC – GC/MS	Check gas	Daily	Refer to SOP in Appendix E
	System bake	Daily	
	Replace septa	As needed	
	Change/Replace Liner	As needed	
	Change column	As needed	
	Change Ferrules	As needed	
	Replace trap	As needed	
	Change Vacuum pump oil	2x year	
SVOC – GC/MS	Check gas	Daily	Refer to SOP in Appendix E
	Clean/replace liner	As needed	
	Replace column	As needed	
	Change ferrules	As needed	
	Change Vacuum pump oil	2x year	
	Check/change pump tubing	Daily	
	Change capillary tubing	As needed	
	Clean Nebulizer	Monthly (as needed)	
	Clean spray change	Monthly (as needed)	
	Clean torch	As needed	
ICP	Check/ Change Pump Tubing	Daily	Refer to SOP in Appendix E
	Change capillary tubing	As needed	
	Rinse (between samples)	Per sample	
	Clean torch	As needed	
	Clean spray chamber	As needed	
	Clean nebulizer	As needed	
	20 minute rinse before shut-down	Daily	

Table 5-7  
Preventive Maintenance Schedule  
Premier Laboratory  
Former Gorham Property and Mashapaug Cove

Instrument	Activity	Frequency	SOP Ref. *
GC –PCB	Check gas	Daily	Refer to SOP in Appendix E
	System bake	Daily	
	Replace septa	As needed	
	Change/Replace Liner	As needed	
	Change column	As needed	
	Change Ferrules	As needed	
GC -	Check gas	Daily	Refer to SOP in Appendix E
	System bake	Daily	
	Replace septa	As needed	
	Change/Replace Liner	As needed	
	Change column	As needed	
	Change Ferrules	As needed	
	Replace trap	As needed	
Atomic Absorption Mercury, Cold Vapor	Clean cell	Monthly	Refer to SOP in Appendix E
	Clean windows	Monthly	
	Change tubing	As needed	
Lachat Analyzer	Change tubing	As needed	Refer to SOP in Appendix E

Table 5-8  
Preventive Maintenance Schedule  
Severn Trent Laboratories  
Former Gorham Property and Mashapaug Cove

Instrument	Activity	Frequency	SOP Ref. *
VOC –GC/MS	Check gas	Daily	Refer to SOP in Appendix F
	System bake	Daily	
	Replace septa	As needed	
	Change/Replace Liner	As needed	
	Change column	As needed	
	Change Ferrules	As needed	
	Replace trap	As needed	
	Change Vacuum pump oil	2x year	
SVOC –GC/MS	Check gas	Daily	Refer to SOP in Appendix F
	Clean/replace liner	As needed	
	Replace column	As needed	
	Change ferrules	As needed	
	Change Vacuum pump oil	2x year	
	Check/change pump tubing	Daily	
	Change capillary tubing	As needed	
	Clean Nebulizer	Monthly (as needed)	
	Clean spray change	Monthly (as needed)	
	Clean torch	As needed	
GC -	Check gas	Daily	Refer to SOP in Appendix F
	System bake	Daily	
	Replace septa	As needed	
	Change/Replace Liner	As needed	
	Change column	As needed	
	Change Ferrules	As needed	
	Replace trap	As needed	

Table 3-2  
 Summary of Action Levels and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Residential Direct Exposure Criteria (mg/Kg)	Industrial/Commercial Direct Exposure Criteria (mg/Kg)	GB Leachability Criteria (mg/Kg)	GB Groundwater Objectives (mg/L)	Soil Reporting Limits (mg/Kg)	Aqueous Reporting Limits (mg/L)
Antimony	10	820	NE	NE	0.50	0.010
Arsenic	7.0	7.0	NE	NE	0.50	0.010
Beryllium	0.4	1.3	NE	NE	0.05	0.001
Cadmium	39	1,000	NE	NE	0.10	0.002
Chromium, Trivalent	1,400	10,000	NE	NE	0.50	0.010
Chromium, Hexavalent	390	10,000	NE	NE	0.50	0.050
Copper	3,100	10,000	NE	NE	0.50	0.010
Lead	150	500	NE	NE	0.20	0.004
Mercury	23	610	NE	NE	0.02	0.0002
Nickel	1,000	10,000	NE	NE	0.50	0.010
Selenium	390	10,000	NE	NE	0.50	0.010
Thallium	5.5	140	NE	NE	0.25	0.005
Zinc	6,000	10,000	NE	NE	0.50	0.010
Cyanide	200	10,000	NE	NE	0.25	0.01
PCBs	10	10	10.0	NE	0.013	0.0002
Acetone	7,800	10,000	NE	NE	0.02	0.02
Benzene	2.5	200	4.3	0.14	0.005	0.005
Bromodichloromethane	10	92	NE	NE	0.005	0.005
Bromoform	81	720	NE	NE	0.005	0.005
Bromomethane	0.8	2,900	NE	NE	0.005	0.005
Carbon Tetrachloride	1.5	44	5.0	0.07	0.005	0.005
Chlorobenzene	210	10,000	100	3.2	0.005	0.005
Chloroform	1.2	940	NE	NE	0.005	0.005
Dibromochloromethane	7.6	68	NE	NE	0.005	0.005
1,2-Dibromo-3-chloropropane	0.5	4.1	NE	0.002	0.005	0.001
1,1-Dichloroethane	920	10,000	NE	NE	0.005	0.005
1,2-Dichloroethane	0.9	63	2.3	0.11	0.005	0.005
1,1-Dichloroethene	0.2	9.5	0.7	0.007	0.005	0.005
cis-1,2-Dichloroethene	630	10,000	60	2.4	0.005	0.005

Table 3-2  
 Summary of Action Levels and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Residential Direct Exposure Criteria (mg/Kg)	Industrial/Commercial Direct Exposure Criteria (mg/Kg)	GB Leachability Criteria (mg/Kg)	GB Groundwater Objectives (mg/L)	Soil Reporting Limits (mg/Kg)	Aqueous Reporting Limits (mg/L)
trans-1,2-Dichloroethene	1,100	10,000	92	2.8	0.005	0.005
1,2-Dichloropropane	1.9	84	70	3.0	0.005	0.005
Ethylbenzene	71	10,000	62	1.6	0.005	0.005
Ethylene dibromide	0.01	0.07	NE	NE	0.005	0.005
Isopropylbenzene	27	10,000	NE	NE	0.005	0.005
Methyl ethyl ketone	10,000	10,000	NE	NE	0.02	0.02
Methyl isobutyl ketone	1,200	10,000	NE	NE	0.02	0.02
Methyl t-butyl ether	390	10,000	100	5.0	0.005	0.005
Methylene chloride	45	760	NE	NE	0.005	0.005
Styrene	13	190	64	2.2	0.005	0.005
1,1,1,2-Tetrachloroethane	2.2	220	NE	NE	0.005	0.005
1,1,2,2-Tetrachloroethane	1.3	29	NE	NE	0.005	0.005
Tetrachloroethene	12	110	4.2	0.15	0.005	0.005
Toluene	190	10,000	54	1.7	0.005	0.005
1,1,1-Trichloroethane	540	10,000	160	3.1	0.005	0.005
1,1,2-Trichloroethane	3.6	100	NE	NE	0.005	0.005
Trichloroethene	13	520	20	0.54	0.005	0.005
Vinyl chloride	0.02	3.0	NE	NE	0.005	0.005
Xylenes (total)	110	10,000	NE	NE	0.005	0.005
Acenaphthene	43	10,000	NE	NE	0.167	0.005
Acenaphthylene	23	10,000	NE	NE	0.167	0.005
Anthracene	35	10,000	NE	NE	0.167	0.005
Benzo(a)anthracene	0.9	7.8	NE	NE	0.167	0.005
Benzo(a)pyrene	0.4	0.8	NE	NE	0.167	0.005
Benzo(b)fluoranthene	0.9	7.8	NE	NE	0.167	0.005
Benzo(g,h,i)perylene	0.8	10,000	NE	NE	0.167	0.005
Benzo(k)fluoranthene	0.9	7.8	NE	NE	0.167	0.005
1,1-Biphenyl	0.8	10,000	NE	NE	0.167	0.005
bis(2-ethylhexyl)phthalate	46	410	NE	NE	0.167	0.005

Table 3-2  
 Summary of Action Levels and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Residential Direct Exposure Criteria (mg/Kg)	Industrial/Commercial Direct Exposure Criteria (mg/Kg)	GB Leachability Criteria (mg/Kg)	GB Groundwater Objectives (mg/L)	Soil Reporting Limits (mg/Kg)	Aqueous Reporting Limits (mg/L)
bis(2-chloroethyl)ether	0.6	5.2	NE	NE	0.167	0.005
bis(2-chloroisopropyl)ether	9.1	82	NE	NE	0.167	0.005
4-chloroaniline	310	8,200	NE	NE	0.167	0.005
2-Chlorophenol	50	10,000	NE	NE	0.167	0.005
Chrysene	0.4	780	NE	NE	0.167	0.005
Dibenzo(a,h)anthracene	0.4	0.8	NE	NE	0.167	0.005
o-Dichlorobenzene	510	10,000	NE	NE	0.167	0.005
m-Dichlorobenzene	430	10,000	NE	NE	0.167	0.005
p-Dichlorobenzene	27	240	NE	NE	0.167	0.005
3,3-Dichlorobenzidine	1.4	13	NE	NE	0.167	0.005
2,4-Dichlorophenol	30	6,100	NE	NE	0.167	0.005
Diethyl phthalate	340	10,000	NE	NE	0.167	0.005
2,4-Dimethyl phenol	1,400	10,000	NE	NE	0.167	0.005
Dimethyl phthalate	1,900	10,000	NE	NE	0.167	0.005
2,4-Dinitrophenol	160	4,100	NE	NE	0.167	0.005
2,4-Dinitrotoluene	0.9	8.4	NE	NE	0.167	0.005
Fluoranthene	20	10,000	NE	NE	0.167	0.005
Fluorene	28	10,000	NE	NE	0.167	0.005
Hexachlorobenzene	0.4	3.6	NE	NE	0.167	0.005
Hexachlorobutadiene	8.2	73	NE	NE	0.167	0.005
Hexachloroethane	46	410	NE	NE	0.167	0.005
Indeno(1,2,3-cd)pyrene	0.9	7.8	NE	NE	0.167	0.005
2-Methylnaphthalene	123	10,000	NE	NE	0.167	0.005
Naphthalene	54	10,000	NE	NE	0.167	0.005
Pentachlorophenol	5.3	48	NE	NE	0.167	0.005
Phenanthrene	40	10,000	NE	NE	0.167	0.005
Phenol	6,000	10,000	NE	NE	0.167	0.005
Pyrene	13	10,000	NE	NE	0.167	0.005
1,2,4-Trichlorobenzene	96	10,000	NE	NE	0.167	0.005

Table 3-2  
 Summary of Action Levels and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Residential Direct Exposure Criteria (mg/Kg)	Industrial/Commercial Direct Exposure Criteria (mg/Kg)	GB Leachability Criteria (mg/Kg)	GB Groundwater Objectives (mg/L)	Soil Reporting Limits (mg/Kg)	Aqueous Reporting Limits (mg/L)
2,4,5-Trichlorophenol	330	10,000	NE	NE	0.167	0.005
2,4,6-Trichlorophenol	58	520	NE	NE	0.167	0.005
Aldrin	NE	NE	NE	NE	0.00067*	0.00002
alpha-BHC	NE	NE	NE	NE	0.00067*	0.00002
beta-BHC	NE	NE	NE	NE	0.00067*	0.00002
delta-BHC	NE	NE	NE	NE	0.00067*	0.00002
gamma-BHC (Lindane)	NE	NE	NE	NE	0.00067*	0.00002
alpha-Chlordane	NE	NE	NE	NE	0.00067*	0.00002
gamma-Chlordane	NE	NE	NE	NE	0.00067*	0.00002
4,4'-DDD	NE	NE	NE	NE	0.00067*	0.00002
4,4'-DDE	NE	NE	NE	NE	0.00067*	0.00002
4,4'-DDT	NE	NE	NE	NE	0.00067*	0.00002
Dieldrin	0.04	0.4	NE	NE	0.00067*	0.00002
Endosulfan I	NE	NE	NE	NE	0.00067*	0.00002
Endosulfan II	NE	NE	NE	NE	0.00067*	0.00002
Endosulfan sulfate	NE	NE	NE	NE	0.00067*	0.00002
Endrin	NE	NE	NE	NE	0.00067*	0.00002
Endrin aldehyde	NE	NE	NE	NE	0.00067*	0.00002
Heptachlor	NE	NE	NE	NE	0.00067*	0.00002
Heptachlor epoxide	NE	NE	NE	NE	0.00067*	0.00002
Methoxychlor	NE	NE	NE	NE	0.00067*	0.00002
Toxaphene	NE	NE	NE	NE	0.00067*	0.00002
Total Petroleum Hydrocarbons	NE	NE	NE	NE	10	0.100
1,2,3,7,8-PeCDD	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,6,7,8-HxCDD	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,7,8-HxCDD	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,7,8,9-HxCDD	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,6,7,8-HpCDD	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,5,6,7,8-OCDD	NE	NE	NE	NE	0.001	0.0000001

Table 3-2  
 Summary of Action Levels and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Residential Direct Exposure Criteria (mg/Kg)	Industrial/Commercial Direct Exposure Criteria (mg/Kg)	GB Leachability Criteria (mg/Kg)	GB Groundwater Objectives (mg/L)	Soil Reporting Limits (mg/Kg)	Aqueous Reporting Limits (mg/L)
2,3,7,8-TCDF	NE	NE	NE	NE	0.0001	0.00000001
1,2,3,7,8-PeCDF	NE	NE	NE	NE	0.0005	0.00000005
2,3,4,7,8-PeCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,6,7,8-HxCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,7,8,9-HxCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,7,8-HxCDF	NE	NE	NE	NE	0.0005	0.00000005
2,3,4,6,7,8-HxCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,6,7,8-HpCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,7,8,9-HpCDF	NE	NE	NE	NE	0.0005	0.00000005
1,2,3,4,5,6,7,8-OCDF	NE	NE	NE	NE	0.001	0.0000001

Table 3-3  
 Summary of Sediment Ecological Criteria and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Units	Ecological Screening Criteria	Final RL (Wet Weight)	Final RL (Dry Weight, assuming 50% solid)
Antimony	mg/Kg	2.0 <sup>b</sup>	0.50	1.00
Arsenic	mg/Kg	9.79 <sup>a</sup>	1.00	2.00
Beryllium	mg/Kg	NE	0.050	0.10
Cadmium	mg/Kg	0.99 <sup>a</sup>	0.20	0.40
Chromium, Trivalent	mg/Kg	43.4 <sup>a</sup>	1.00	2.00
Chromium, Hexavalent	mg/Kg	43.4 <sup>a</sup>	0.50	1.00
Copper	mg/Kg	31.6 <sup>a</sup>	1.00	2.00
Lead	mg/Kg	35.8 <sup>a</sup>	0.40	0.80
Mercury	mg/Kg	0.18 <sup>a</sup>	0.01	0.02
Nickel	mg/Kg	22.7 <sup>a</sup>	1.00	2.00
Selenium	mg/Kg	NE	0.50	1.00
Thallium	mg/Kg	NE	0.25	0.50
Zinc	mg/Kg	121 <sup>a</sup>	1.00	2.00
Cyanide	mg/Kg	0.0001 <sup>c,g</sup>	0.25	0.50
PCBs	µg/Kg	59.8 <sup>b</sup>	13	26
Acetone	µg/Kg	8.7 <sup>d</sup>	20	40
Benzene	µg/Kg	57 <sup>e</sup>	5	10
Bromodichloromethane	µg/Kg	NE	5	10
Bromoform	µg/Kg	650 <sup>d,e</sup>	5	10
Bromomethane	µg/Kg	NE	5	10
Carbon Tetrachloride	µg/Kg	47 <sup>d</sup>	5	10
Chlorobenzene	µg/Kg	410 <sup>d</sup>	5	10
Chloroform	µg/Kg	22 <sup>d</sup>	5	10
Dibromochloromethane	µg/Kg	NE	5	10
1,2-Dibromo-3-chloropropane	µg/Kg	NE	5	10
1,1-Dichloroethane	µg/Kg	27 <sup>d</sup>	5	10
1,2-Dichloroethane	µg/Kg	250 <sup>d</sup>	5	10
1,1-Dichloroethene	µg/Kg	31 <sup>d</sup>	5	10
cis-1,2-Dichloroethene	µg/Kg	400 <sup>d</sup>	5	10
trans-1,2-Dichloroethene	µg/Kg	400 <sup>d</sup>	5	10
1,2-Dichloropropane	µg/Kg	NE	5	10

Table 3-3  
 Summary of Sediment Ecological Criteria and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Units	Ecological Screening Criteria	Final RL (Wet Weight)	Final RL (Dry Weight, assuming 50% solid)
Ethylbenzene	µg/Kg	NE	5	10
Ethylene dibromide	µg/Kg	NE	5	10
Isopropylbenzene	µg/Kg	NE	5	10
Methyl ethyl ketone	µg/Kg	NE	20	40
Methyl isobutyl ketone	µg/Kg	NE	20	40
Methyl t-butyl ether	µg/Kg	NE	5	10
Methylene chloride	µg/Kg	NE	5	10
Styrene	µg/Kg	NE	5	10
1,1,1,2-Tetrachloroethane	µg/Kg	NE	5	10
1,1,2,2-Tetrachloroethane	µg/Kg	940 <sup>e</sup>	5	10
Tetrachloroethene	µg/Kg	410 <sup>d</sup>	5	10
Toluene	µg/Kg	50 <sup>d</sup>	5	10
1,1,1-Trichloroethane	µg/Kg	30 <sup>d</sup>	5	10
1,1,2-Trichloroethane	µg/Kg	1200 <sup>d</sup>	5	10
Trichloroethene	µg/Kg	220 <sup>d</sup>	5	10
Vinyl chloride	µg/Kg	NE	5	10
Xylenes (total)	µg/Kg	160 <sup>d</sup>	5	10
Acenaphthene	µg/Kg	NE	6.7	13.4
Acenaphthylene	µg/Kg	NE	6.7	13.4
Anthracene	µg/Kg	57.2 <sup>b</sup>	6.7	13.4
Benzo(a)anthracene	µg/Kg	31.7 <sup>f</sup>	6.7	13.4
Benzo(a)pyrene	µg/Kg	31.9 <sup>f</sup>	6.7	13.4
Benzo(b)fluoranthene	µg/Kg	NE	6.7	13.4
Benzo(g,h,i)perylene	µg/Kg	170 <sup>g</sup>	6.7	13.4
Benzo(k)fluoranthene	µg/Kg	240 <sup>g</sup>	6.7	13.4
1,1-Biphenyl	µg/Kg	1100 <sup>d,e</sup>	167	334
bis(2-ethylhexyl)phthalate	µg/Kg	89,000 <sup>d</sup>	167	334
bis(2-chloroethyl)ether	µg/Kg	NE	167	334
bis(2-chloroisopropyl)ether	µg/Kg	NE	167	334
4-chloroaniline	µg/Kg	NE	167	334
2-Chlorophenol	µg/Kg	NE	167	334

Table 3-3  
 Summary of Sediment Ecological Criteria and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Units	Ecological Screening Criteria	Final RL (Wet Weight)	Final RL (Dry Weight, assuming 50% solid)
Chrysene	µg/Kg	166 <sup>a</sup>	6.7	13.4
Dibenzo(a,h)anthracene	µg/Kg	6.22 <sup>h</sup>	6.7	13.4
o-Dichlorobenzene	µg/Kg	330 <sup>d</sup>	167	334
m-Dichlorobenzene	µg/Kg	1700 <sup>d</sup>	167	334
p-Dichlorobenzene	µg/Kg	340 <sup>d</sup>	167	334
3,3-Dichlorobenzidine	µg/Kg	NE	167	334
2,4-Dichlorophenol	µg/Kg	NE	167	334
Diethyl phthalate	µg/Kg	600 <sup>d</sup>	167	334
2,4-Dimethyl phenol	µg/Kg	NE	167	334
Dimethyl phthalate	µg/Kg	NE	167	334
2,4-Dinitrophenol	µg/Kg	NE	167	334
2,4-Dinitrotoluene	µg/Kg	NE	167	334
Fluoranthene	µg/Kg	423 <sup>a</sup>	6.7	13.4
Fluorene	µg/Kg	77.4 <sup>a</sup>	6.7	13.4
Hexachlorobenzene	µg/Kg	NE	167	334
Hexachlorobutadiene	µg/Kg	1000 <sup>d</sup>	167	334
Hexachloroethane	µg/Kg	NE	167	334
Indeno(1,2,3-cd)pyrene	µg/Kg	200 <sup>g</sup>	6.7	13.4
2-Methylnaphthalene	µg/Kg	130 <sup>d</sup>	167	334
Naphthalene	µg/Kg	176 <sup>a</sup>	6.7	13.4
Pentachlorophenol	µg/Kg	NE	167	334
Phenanthrene	µg/Kg	204 <sup>a</sup>	6.7	13.4
Phenol	µg/Kg	NE	167	334
Pyrene	µg/Kg	53 <sup>h</sup>	6.7	13.4
1,2,4-Trichlorobenzene	µg/Kg	9200 <sup>d</sup>	167	334
2,4,5-Trichlorophenol	µg/Kg	NE	167	334
2,4,6-Trichlorophenol	µg/Kg	NE	167	334
Total Petroleum Hydrocarbons	µg/Kg	NE	10	20
2,3,7,8-TCDD	ng/Kg	NE	1.0	2.0
1,2,3,7,8-PeCDD	µg/Kg	NE	5.0	10
1,2,3,6,7,8-HxCDD	µg/Kg	NE	5.0	10

Table 3-3  
 Summary of Sediment Ecological Criteria and Laboratory Reporting Limits  
 Premier laboratory and Severn Trent Laboratories  
 Former Gorham Property and Mashapaug Cove

Compound	Units	Ecological Screening Criteria	Final RL (Wet Weight)	Final RL (Dry Weight, assuming 50% solid)
1,2,3,4,7,8-HxCDD	µg/Kg	NE	5.0	10
1,2,3,7,8,9-HxCDD	µg/Kg	NE	5.0	10
1,2,3,4,6,7,8-HpCDD	µg/Kg	NE	5.0	10
1,2,3,4,5,6,7,8-OCDD	µg/Kg	NE	10	20
2,3,7,8-TCDF	µg/Kg	NE	1.0	2.0
1,2,3,7,8-PeCDF	µg/Kg	NE	5.0	10
2,3,4,7,8-PeCDF	µg/Kg	NE	5.0	10
1,2,3,6,7,8-HxCDF	µg/Kg	NE	5.0	10
1,2,3,7,8,9-HxCDF	µg/Kg	NE	5.0	10
1,2,3,4,7,8-HxCDF	µg/Kg	NE	5.0	10
2,3,4,6,7,8-HxCDF	µg/Kg	NE	5.0	10
1,2,3,4,6,7,8-HpCDF	µg/Kg	NE	5.0	10
1,2,3,4,7,8,9-HpCDF	µg/Kg	NE	5.0	10
1,2,3,4,5,6,7,8-OCDF	µg/Kg	NE	10	20

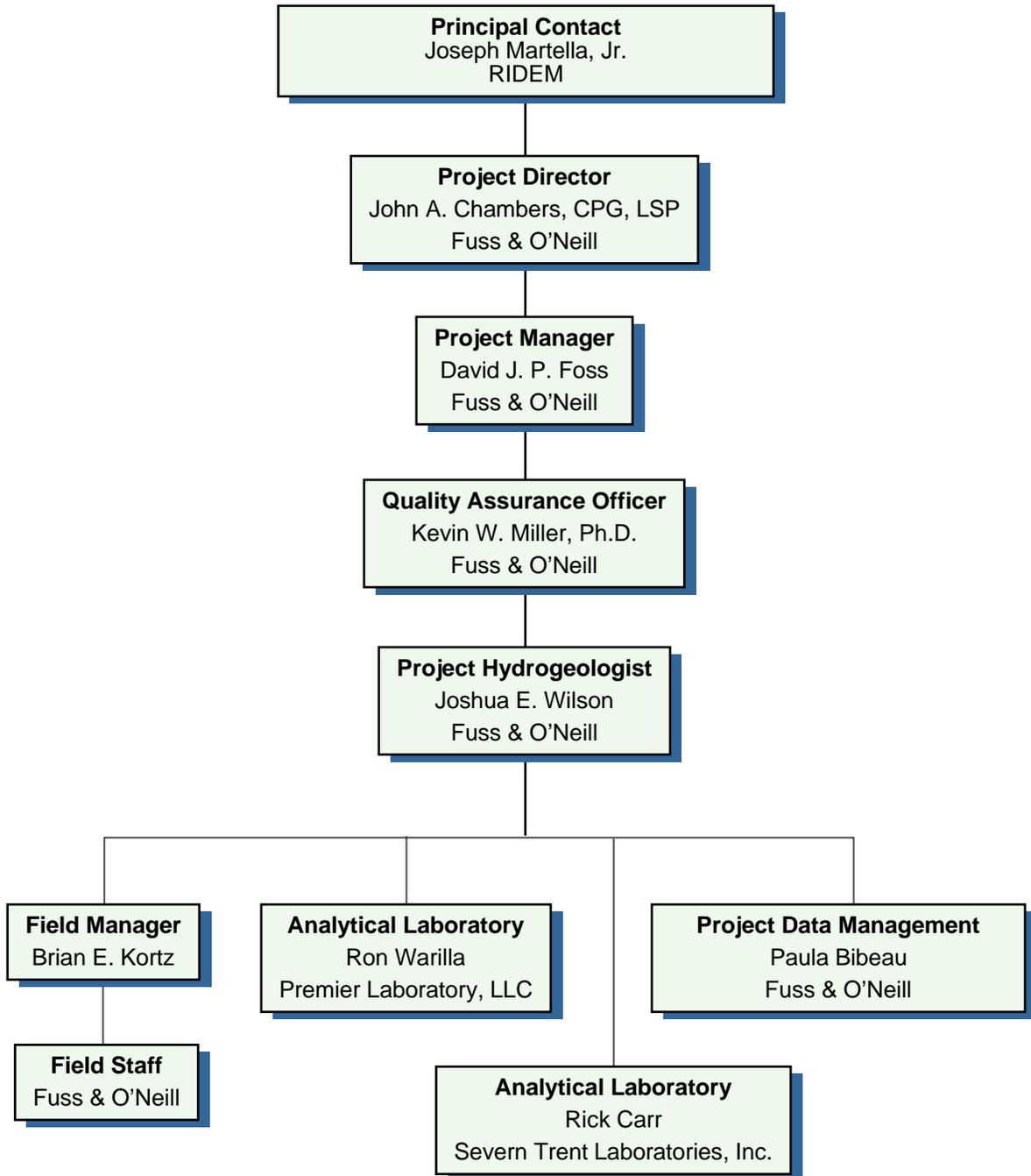
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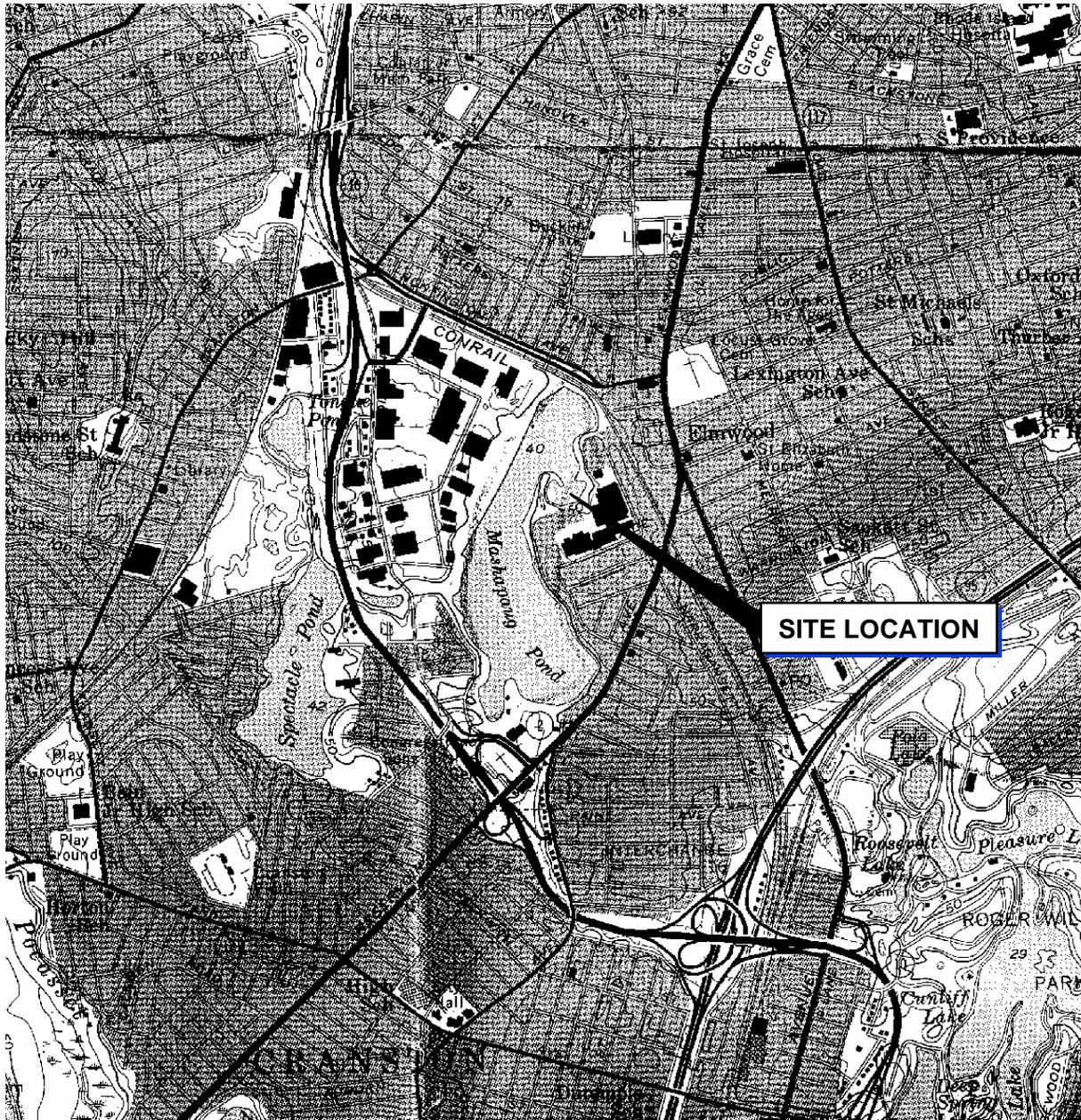
- a. MacDonald, D.D., C.G. Ingersoll, and T.A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater ecosystems. Archives of Environmental Contamination and Toxicology 39:20-31.
- b. Long, E. R. and L. G. Morgan. 1991. The Potential for Biological Effects of Sediment-Sorbed Contaminants Tested in the National Status and Trends Program, National Oceanographic and Atmospheric Administration, Tech. Memorandum NOS OMA 52, August 1991. Seattle, Washington.
- c. USEPA Region 5. 2003. Ecological Screening Levels (ESLs) for RCRA Appendix IX hazardous constituents. August 2003 Available Online: <http://www.epa.gov/reg5rcra/ca/ESL.pdf>
- d. Jones, D.S., G.W. Suter, and R.N. Hull. 1997. Toxicological benchmarks for screening contaminants of potential concern for effects on sediment-associated biota: 1997 revision. Oak Ridge National Laboratories. ES/ER/TM-93/R4
- e. United States Environmental Protection Agency (USEPA). 1996. Office of Solid Waste and Emergency Response (OSWER) ECO Update, "Ecotox Thresholds," Vol. 3, No. 2, January 1996. EPA/540/F-95/038
- f. Smith, S.I., D.D. MacDonald, K.A. Keenleyside, C.G. Ingersoll, and L.J. Field. 1996. A preliminary evaluation of Sediment Quality Assessment Values for Freshwater Ecosystems J. Great Lakes Res. 22(3):624-638.
- g. Persaud, D., R. Jaagumag, and A. Hayton. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Queen's Printer for Ontario. Reprinted 1994
- h. Canadian Council of Ministers of the Environment (CCME). 2002. Summary of existing Canadian Environmental quality guidelines. Updated 2002.

# FIGURES

FIGURE 2—1

Project Organization Chart  
Former Gorham Site and Mashapaug Cove  
Short-Term Limited Site Investigation  
Providence, Rhode Island  
December 2005





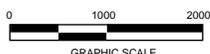
**MAP REFERENCE:**  
 THIS MAP WAS PREPARED FROM THE FOLLOWING  
 7.5 MINUTE SERIES TOPOGRAPHIC MAP:  
 PROVIDENCE, RHODE ISLAND 1957  
 PHOTOREVISED 1970 AND 1975

Rhode Island



Quadrangle Location

FILE PATH AND NAME



SCALE: 1"=2000'



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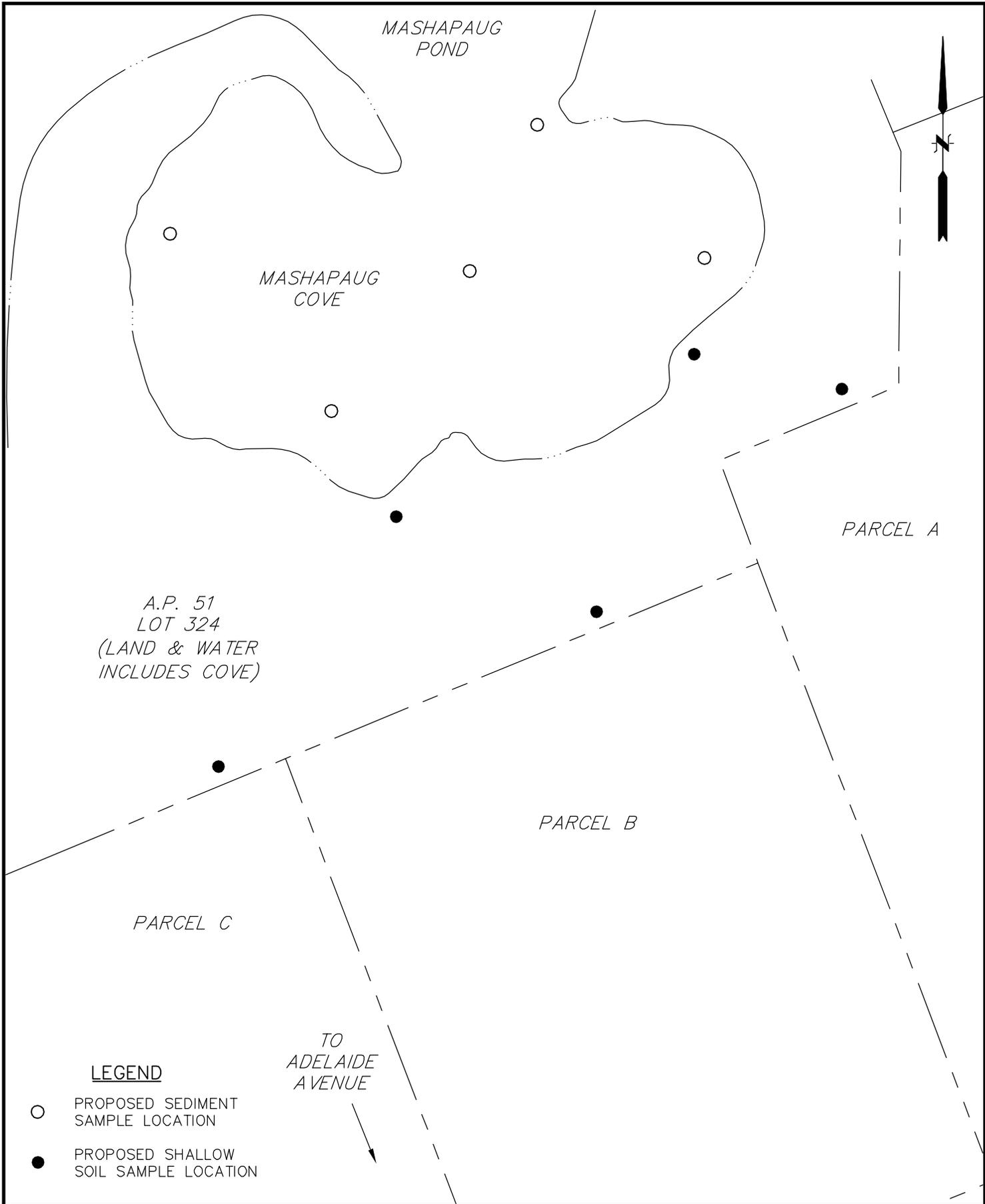
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**SITE LOCATION MAP**  
 FORMER GORHAM PROPERTY &  
 MASHAPAUG COVE

PROVIDENCE RHODE ISLAND

PROJ. No: 20051057A10  
 DATE: December 2005

**FIGURE 3- 1**



SCALE:	
HORZ.:	1" = 1'
VERT.:	
DATUM:	
HORZ.:	
VERT.:	


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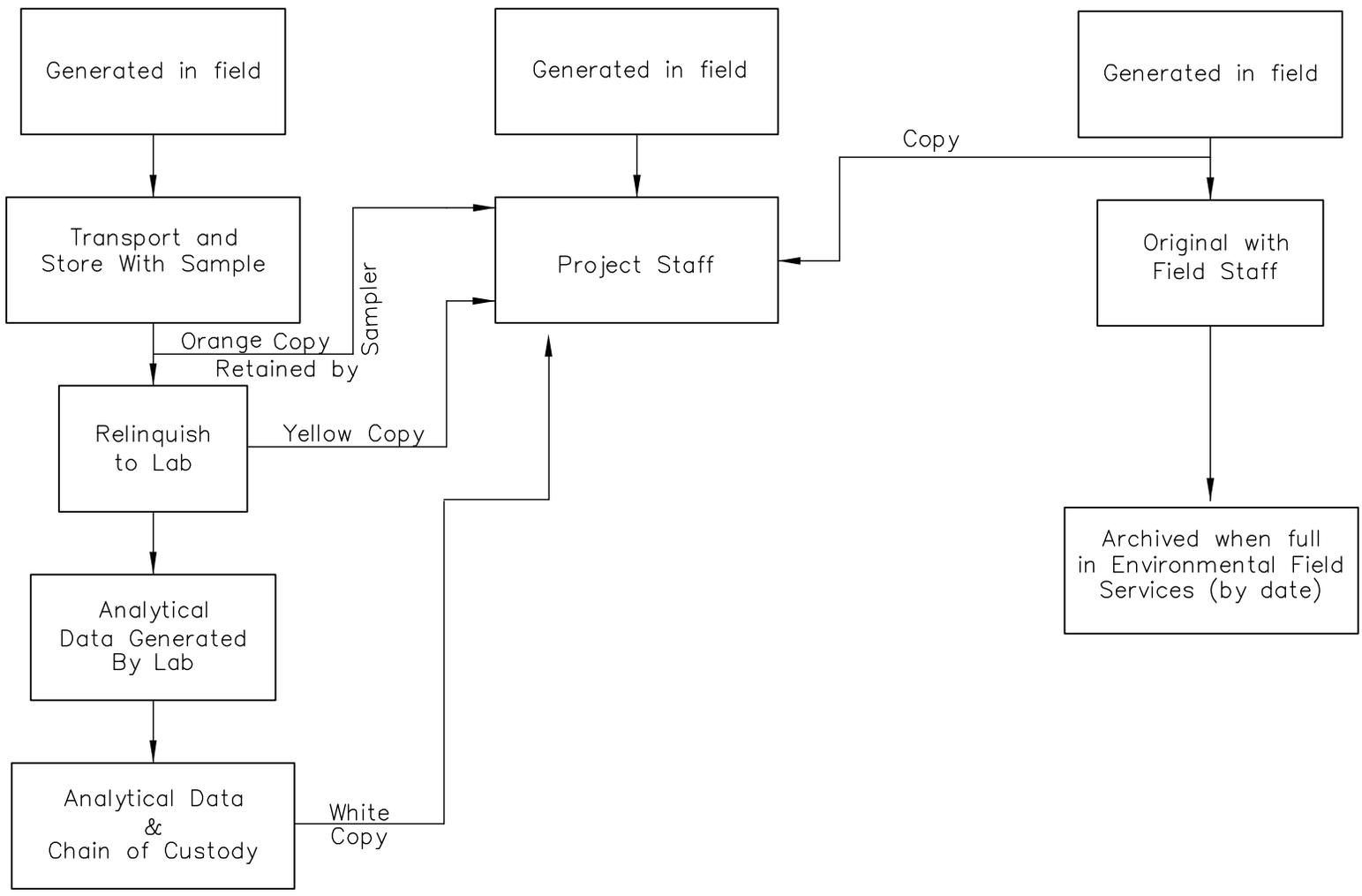
**PROPOSED SAMPLING LOCATIONS**  
**FORMER GORHAM SITE AND**  
**MASHAPAUG COVE**  
**SHORT-TERM LIMITED SITE INVESTIGATION**  
 PROVIDENCE RHODE ISLAND

PROJ. No.: 20051057.A10  
 DATE: DECEMBER 2005  
**FIGURE**  
**4-1**

CHAIN OF CUSTODY  
(C.O.C.)

FIELD DATA FORMS

FIELD LOGBOOKS



SCALE:	
HORZ.: NTS	
VERT.:	
DATUM:	
HORZ.:	
VERT.:	



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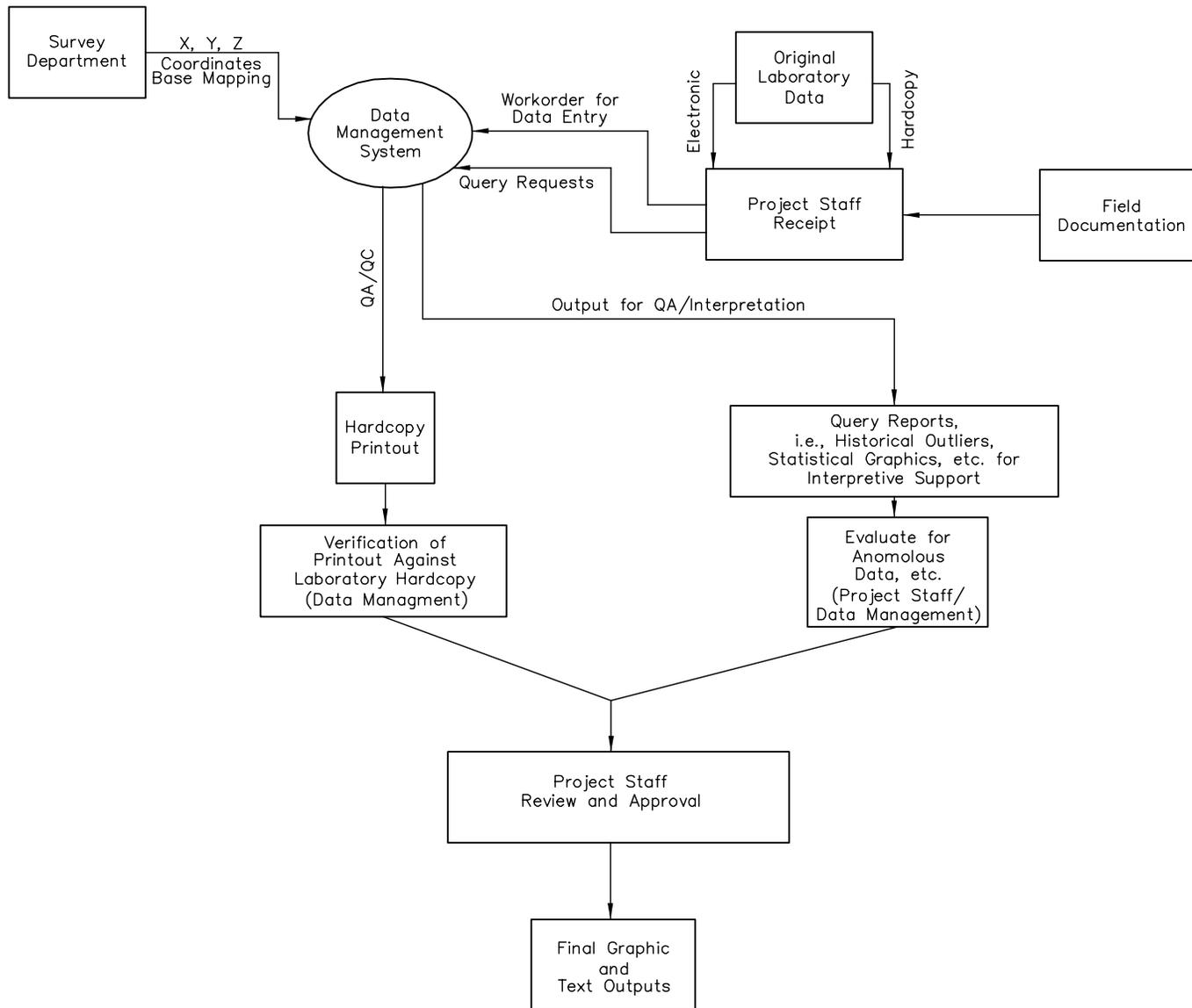
FIELD DOCUMENTATION FLOW CHART  
FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
QUALITY ASSURANCE PROJECT PLAN

PROVIDENCE

RHODE ISLAND

PROJ. No.: 20051057.A10  
DATE: DECEMBER 2005

FIGURE 8-1



UCS: LMS VIEW: CHART LMAN: FO: STANDARD CTB:

SCALE:	
HORZ.:	NTS
VERT.:	
DATUM:	
HORZ.:	
VERT.:	



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ENVIRONMENTAL DATA MANAGEMENT FLOW CHART  
 FORMER GORHAM PROPERTY AND MASHAUG COVE  
 QUALITY ASSURANCE PROJECT PLAN

PROVIDENCE

RHODE ISLAND

PROJ. No.: 20051057.A10  
 DATE: DECEMBER 2005

FIGURE 8-2



## APPENDIX A

### QUALITY ASSURANCE PROJECT PLAN WRITER/REVIEWER CHECKLIST



Quality Assurance Project Plans  
 Writer/Reviewer Checklist

Region 1, New England

(Based on Quality Assurance Guidance for Conducting Brownfields Site Assessments)

QAPP title and date: Former Gorham Property and Mashapaug Cove, December 2005

QAPP prepared by: Fuss & O'Neill, Inc.

Reviewed by and date: Lynne Matteson, December 8, 2005

PROJECT MANAGEMENT		Section
Form A	Title and Approval Page	1.0
Form B	Project Organization and Responsibility	2.0
Form C	Problem Definition	3.0
Form D	Project Description/Project Timeline	3.4, 4.1, 4.4
MEASUREMENT DATA ACQUISITION		
Form E	Sampling Design	4.0
Form F-1	Sampling and Analytical Methods Requirements	5.0
Form F-2	Method and SOP Reference Table	Tables 5-1, 5-2, 5-5
Form G	Preventative Maintenance – Field Equipment	5.1.1, Tables 5-2, 5-3
Form H	Calibration and Corrective Action – Field Equipment	5.1.2, Table 5-4
Form I	Preventative Maintenance – Laboratory Equipment	5.2.1, Tables 5-7, 5-8
Form J	Calibration and Corrective Action – Laboratory Equipment	5.2.1, Tables 5-7, 5-8
Form K	Sample Handling and Custody Requirements	6.0
Form L	Analytical Precision and Accuracy	11.2
Form M	Field Quality Control Requirements/Laboratory Quality Control Requirements	7.0
Form N	Data Management and Documentation	8.0
ASSESSMENT/OVERSIGHT		
Form O	Assessments and Response Actions	9.0
Form P	Project Reports	10.0
DATA VALIDATION AND USABILITY		
Form Q-1	Verification of Sampling Procedures	6.1
Form Q-1	Data Verification and Validation	11.0
Form R	Data Usability	12.0

QAPP Elements	Comments
<b>PROJECT MANAGEMENT</b>	
Form A – Title and Approval Page	Completed
<ul style="list-style-type: none"> <li>U Project Title</li> <li>U Prepared for, prepared by</li> <li>U Plan data and revision number</li> <li>U Approving Officials (names, titles, signatures, and data signed) Examples:               <div style="text-align: center; margin-left: 100px;">                 Project Manager                  Project QA Officer                  Facility Project Manager Approval                  CT LEP Project Manager Approval                  Other signatures as appropriate               </div> </li> </ul>	Section 1.0
Form B – Project Organization and Responsibility	Completed
<p>Identify <u>key</u> individuals in all organizations participating in the project, including: data users, decision makers, QA managers, leaders responsible of implementing the project, subcontractor leads, etc. (Chart, table, or text format acceptable, or any combination)</p> <ul style="list-style-type: none"> <li>U Identify the title/position, name of key individual, organization they work for, and contact information (telephone number and email).</li> <li>U Indicate lines of communication and authority between key individuals.</li> <li>U For complex projects, consider defining roles and responsibilities of key individuals.</li> </ul>	Section 2.0 Figure 2-1
Form C – Problem Definition	Completed
<ul style="list-style-type: none"> <li>U Provide sufficient background information from a historic, scientific, and/or regulatory perspective.</li> <li>U Identify specific primary contaminants of concern (i.e., lead as opposed to just metals), and indicate magnitude of contamination present.</li> <li>U Provide historic maps and summary data, as appropriate to illustrate problem.</li> <li>U Present the current understanding of the conceptual site model (CSM) for the project, and indicate how contamination may be acting in the environment. (Note, reference any important documents/reports used in development of the CSM and sampling design for the project.)</li> <li>i Clearly state the problem(s) to be solved, decision(s) to be made, and outcome(s) to be achieved.</li> </ul>	Section 3.0

QAPP Elements	Comments
Form D – Project Description	Completed
<ul style="list-style-type: none"> <li>U Provide an <u>outline</u> for the Phase II or Phase III tasks to be performed, including:</li> <li>U State the principle use of the data.</li> <li>U Identify the media and parameters being sampled.</li> <li>U Identify field measurements, field analytical and off-site laboratory testing.</li> <li>U Distinguish between the critical parameters on which decisions will be based (i.e., specific analytes or compounds of concern), and non-critical data used for supporting purposes.</li> <li>U Identify action levels to be met for <u>known</u> primary contaminants of concern that will drive decisions.</li> <li>U Define important conditions under which data should be collected (i.e., storm event, season, flow rate, etc.).</li> <li>U Cite relevant regulatory standards or criteria that data will be compared against.</li> <li>i Provide clear discussion on how project tasks relate to resolving problems/issues stated in background section.</li> </ul>	Section 3.4, 4.0 Table 3-1
Form D (Cont.) – Project Timeline	Completed
<ul style="list-style-type: none"> <li>U Provide a projected timeline for field, laboratory and reporting activities to be completed. (Note, please allow for QAPP review and approval in timeline planning. A minimum of 3 weeks is recommended.)</li> </ul>	Section 4.4
<b>MEASUREMENT DATA ACQUISITION</b>	
Form E – Sampling Design	Completed
<p>Provide the details and design elements to the various project tasks to be performed, including the anticipated samples to be collected and/or the experiment to be conducted. (Note, a thorough site reconnaissance should be performed and results incorporated into the plan.)</p> <ul style="list-style-type: none"> <li>i Explain the thought process behind the logic and rationale for the layout of the sample locations (discuss in terms of what individual locations are meant to represent, and globally in terms of what a set or series of samples are meant to represent).</li> <li>U Explain any design assumptions being made, and the importance of any particular compounds/analytes to the project (e.g., an indicator for other contaminants).</li> <li>U When sampling locations and/or choice of analytical parameters cannot be predetermined, document the decision logic or input that will be used in the field to make those decisions.</li> <li>U Include detailed sampling maps and sample summary tables (form F-1) that clarify and <u>reflect</u> the design text.</li> </ul>	Section 4.0

QAPP Elements	Comments
Form F-1 – Sampling and Analytical Methods Requirements	Completed
<p>Include detailed sample summary table that clarifies and reflects the design text. Note, the table (or tables) can be of your own design, but they need to include:</p> <ul style="list-style-type: none"> <li>U Sample Matrix</li> <li>U Parameter</li> <li>U Number of field samples to be collected for each parameter and matrix.</li> <li>U Number of each type of field QC sample to be collected with each parameter and matrix.</li> <li>U Analytical method reference, <u>including preparation and analysis methods</u> (e.g., VOCs in soil –SW846 5035A/8260B)</li> <li>U Sampling Method reference (refer to Form F-2)</li> <li>U Sample containers (number per sample, size, and type)</li> <li>U Preservation (temperature, light, chemical)</li> <li>U Maximum holding times (preparation and analysis)</li> </ul>	<p>Section 4.2 Figure 3-2 Table 5-6</p>
Form F-2 – Method and SOP Reference Table	Completed
<p>Provide a table of references for the standardized procedures used to collect, prepare, and analyze the samples. Include:</p> <ul style="list-style-type: none"> <li>U The analytical method references for the preparation and analysis of the samples. Include document title, method name and number, revision number and date.</li> <li>U The corresponding laboratory SOP reference for each analytical procedure listed. Include document title, revision number, date and author (i.e., organization).</li> <li>U The field SOPs for sample collection, sample preservation, equipment decontamination, etc. Include document title, revision number, date and author (i.e., organization).</li> <li>U Provide appendices to the QAPP which includes copies of all field sampling and laboratory SOPs.</li> <li>U Note, if any project-specific modifications are to be incorporated into the standard procedures, then please document those changes in the QAPP or, better yet, modify the SOP to reflect the actual procedures being used.</li> <li>U For non-standard methods, attach the individual analytical SOPs.</li> </ul>	<p>See Section 5.0 Tables 5-1, 5-2, and 5-5 Appendices C, D, E, F, and G</p>
Form G – Preventative Maintenance – Field Equipment	Completed
<p>List the inspection, acceptance testing, and preventative maintenance program for the various field equipment being used on the project.</p> <ul style="list-style-type: none"> <li>U Describes how problems are resolved and results documented.</li> </ul>	<p>See Section 5.1.1 Table 5-3 Appendix D</p>

QAPP Elements	Comments
Form H – Calibration and Corrective Action – Field Equipment	Completed
<p>Provide a field equipment calibration table for the various field equipment being used on the project that requires calibration. (Note, calibration records should be traceable to equipment.) Include:</p> <ul style="list-style-type: none"> <li>U Type of calibration check (e.g., initial calibration with number of standards, independent calibration checks, continuing calibration checks, blank checks, etc.)</li> <li>U Frequency</li> <li>U Acceptance criteria (control limits)</li> <li>U Corrective Actions when control limits are exceeded</li> </ul>	<p>See Section 5.1.2 Table 5-4 Appendix D</p> <p>Section 7.4, Table 5-4</p>
Form I – Preventative Maintenance – Laboratory Equipment	Completed
<p>Describe the inspection, acceptance testing and preventative maintenance program for the various laboratory instrumentation being used on the project.</p> <ul style="list-style-type: none"> <li>U Describes how problems are resolved and results documented.</li> </ul>	<p>See Appendices E, F See laboratory SOPs for individual analytical methods Appendices E and F and QC limits and Tables 5-7, 5-8</p>
Form J – Calibration and Corrective Action – Laboratory Equipment	Completed
<p>Provide a laboratory instrument calibration table for the various laboratory instrumentation being used on the project that requires calibration. (Note, calibration records should be traceable to laboratory instrument.) Include:</p> <ul style="list-style-type: none"> <li>U Type of calibration check (e.g., initial calibration with number of standards, independent calibration check, continuing calibration checks, blank checks, internal standard checks, interference checks, etc.).</li> <li>U Frequency</li> <li>U Acceptance criteria (control limits)</li> <li>U Corrective actions when control limits are exceeded</li> </ul>	<p>See Section 5.2.1</p> <p>See Appendices E&amp;F</p> <p>See laboratory SOPs (Appendices E and F) for individual analytical methods and QC limits</p>
Form K – Sample Handling and Custody Requirements	Completed
<p>Describe chain-of-custody (COC) procedures between field preparation, transport, and laboratory receipt (can simply reference SOP, if available, and attach).</p> <ul style="list-style-type: none"> <li>U Describe the sample numbering scheme that will be used throughout the project (i.e., sample number code that identifies location, matrix, depth, etc.).</li> <li>U Provide example sample labels, COC forms, and custody seals.</li> </ul>	<p>Section 6.0 Section 6.2 Appendix G</p>

QAPP Elements	Comments
Form L – Analytical Precision and Accuracy	Completed
<p>Complete an analytical precision and accuracy table for the various analyses being performed on the project. Include:</p> <ul style="list-style-type: none"> <li>U Analyte or parameter</li> <li>U Method reference (preparation and analysis)</li> <li>U Detection Limit (aqueous and/or soil)</li> <li>U Quantitation Limit (aqueous and/or soil)</li> <li>U Laboratory Precision (aqueous and/or soil)</li> <li>U Laboratory Accuracy (aqueous and/or soil)</li> </ul> <p>i Note 1: primary contaminants of concern for the site should be completed individually. Non-critical analytes may be listed by broad parameter, their detection limits can be referenced to an attachment, and precision and accuracy values can represent the broad parameter.</p> <p>i Note 2: the Quantitation Limit, often called Reporting Limit, is the minimum concentration the laboratory feels confident in reporting at (as opposed to the detection limit which is determined statistically).</p>	See laboratory SOPs (Appendices E and F) for individual analytical methods and QC limits
Form M – Field Quality Control	Completed
<p>Provide a field sampling QC table. Break down by matrix and parameter as necessary to reflect actual project work. Include:</p> <ul style="list-style-type: none"> <li>U Type of QC sample: field duplicates, split samples (sent to different labs), trip blanks, equipment blanks, cooler temperature blanks, etc. (Note, for soil samples, document field duplicates as either collocated (adjacent to each other), or as split of a single homogenized sample. Collocated duplicates are used to evaluate variability within an area of soil.)</li> <li>U Frequency</li> <li>U Acceptance criteria (control limits)</li> <li>U Corrective Actions when control limits are exceeded</li> </ul>	Section 7.0
Form M (Cont.) – Laboratory Quality Control	Completed
<p>Provide an analytical QC table. Break down by method and matrix as necessary to reflect actual project work. Include:</p> <ul style="list-style-type: none"> <li>U Type of QC sample (lab duplicates, matrix spikes, matrix spike duplicates, surrogates, lab control samples, method blanks, etc.)</li> <li>U Frequency</li> <li>U Acceptance criteria (control limits)</li> <li>U Corrective actions when control limits are exceeded</li> </ul>	<p>Section 7.5</p> <p>See laboratory SOPs for individual analytical methods (Appendices E and F) and QC limits</p>

QAPP Elements	Comments
Form N – Data Management and Documentation	Completed
<p>Describe the documentation and management of the data that is to be generated for the project, including field, laboratory, and final data manipulation.</p> <p>Field Documents and Records</p> <ul style="list-style-type: none"> <li>U Describe the routine procedures used for taking field notes.</li> <li>U List the remaining field documents and records that will be generated, collected, and managed in the project file.</li> <li>U Briefly describe the process for collection and review of the field notes and other records as they are generated (include any QA checks for completeness, consistency, accuracy, etc.).</li> <li>U Specify where the records will be stored and for how long.</li> <li>U Provide an appendix to the QAPP which includes copies of all field forms and checklists to be used with the project.</li> </ul> <p>Laboratory Documents and Records</p> <p>Specify the contents of the laboratory data package deliverable that the laboratory is responsible for providing (note, establish report expectations for both client and customer). Types of information to request from the laboratory:</p> <ul style="list-style-type: none"> <li>U Data Results Sheets</li> <li>U Method Blank Results</li> <li>U Surrogate Recoveries and Acceptance Limits</li> <li>U Matrix Spike/Matrix Spike Duplicate Results and Acceptance Limits</li> <li>U Spike/Duplicate Results and Acceptance Limits</li> <li>U Laboratory Control Sample Results and Acceptance Limits</li> <li>U Initial and Continuing Calibration Results and Acceptance Limits</li> <li>U All Raw Data and Logbook Sheets</li> <li>U Project Narrative which contains an explanation of any qualified data, and any observations or deviations encountered during analysis.</li> </ul> <p>Post Data Manipulation</p> <p>Describe the handling of electronic data (i.e., the flow of where it goes, and any data manipulation to transfer the data into tables, graphics, databases, models, etc.). Indicate where the data will be stored and identify applicable software as appropriate. Include:</p> <ul style="list-style-type: none"> <li>U Field Data</li> <li>U Lab Data</li> <li>U Office Data (models, databases, etc.)</li> <li>U Describe any checks that will be performed to detect and correct errors, and to prevent loss of data during data reduction, data reporting and data entry into forms/reports/databases.</li> </ul>	<p>Section 8.0</p> <p>Figures 8-1, 8-2</p> <p>Appendix G</p> <p>Section 5.3</p> <p>Sections 8.3</p>

QAPP Elements	Comments
<b>ASSESSMENT/OVERSIGHT</b>	
Form O – Assessments and Response Actions	Completed
Describe the assessment/oversight plan for the project, including: <ul style="list-style-type: none"> <li>U Types of assessments and oversight that will be performed,</li> <li>U Frequency (approximate dates and/or timing),</li> <li>U Individuals responsible for performing the assessments and oversight (e.g., field leader, QA officer, LEP),</li> <li>U Individuals who will receive assessment/oversight report (indicate written or verbal)</li> <li>U Identify individuals responsible for dealing with corrective actions, and follow up on assessments and oversight.               <ul style="list-style-type: none"> <li>i Note, small one-time projects should include, as a minimum, field oversight (early on in the project) by an experienced field leader knowledgeable in the project objectives. More complex projects, with multiple concerns, should increase the level of oversight and target key areas for examination. Also consider laboratory audits for important analytical parameters.</li> </ul> </li> </ul>	Sections 9.0, 10.3
Form P – Project Reports	Completed
Identify the types of reports (written and verbal) that will be provided to management for the project (e.g., status reports, interim reports, final reports, Performance Evaluation sample results, assessment/oversight findings, data quality assessments). Include: <ul style="list-style-type: none"> <li>U Type of report</li> <li>U Frequency</li> <li>U Approximate due dates</li> <li>U Individuals responsible for preparing reports</li> <li>U Individuals and organizations receiving reports</li> <li>i Note, detail the specific contents of the final project report to establish appropriate expectations for both client and customer</li> </ul>	Section 10.0
<b>DATA VALIDATION AND USABILITY</b>	
Form Q-1 – Verification of Sampling Procedures	Completed
Describe the final data evaluation process that will be performed on the data collected in the field (field notes, boring logs, field screening and field analytical data, etc.) <ul style="list-style-type: none"> <li>U Indicate that all the field data will be collected and organized for the project file and reviewed to insure it is complete and compliant with the requirements specified in the QAPP.</li> <li>U Indicate who will perform this task and how it will be documented (e.g., cover letter plus field data evaluation report), including any observations, trends, conclusions and limitations concerning the field data.</li> </ul>	Section 6.1

QAPP Elements	Comments
Form Q-2 – Data Verification and Validation	Completed
<p>Describe the final data evaluation process that will be performed on the laboratory data.</p> <ul style="list-style-type: none"> <li>U Indicate that all the laboratory data will be collected and organized for the project file and reviewed to insure it is complete and compliant with the requirements specified in the QAPP (Form N).</li> <li>U Indicate that the data will be evaluated to determine if the data met the precision, accuracy, bias, and sensitivity criteria supplied in Forms J, L, and M. (Note, data validation may also apply to field analytical data that is used in making critical decisions.)</li> <li>U Define any data qualifiers that will be applied to the data, should acceptance criteria (or control limits) not be met (i.e., U, J, UJ, R, B, etc.).</li> <li>U Indicate who will perform this task and how it will be documented (e.g., cover letter plus validation report), including any observations, trends, conclusions and limitations concerning the laboratory data.</li> </ul>	Section 11.0 Appendix B
Form R – Data Usability	Completed
<p>Describe the project evaluation process that will be performed, using the complete data set as a whole, to determine if the objectives and criteria established for the project have been met. Including:</p> <ul style="list-style-type: none"> <li>U Describe the data tables, graphs and statistical evaluations that will be prepared/performed on the project data to illustrate trends, anomalies, and gaps in the data.</li> <li>U Indicate that important limitations on the use of the data, found during the data evaluation steps in Forms Q-1 and QC-2, will be documented and explained in the final report.</li> <li>U Indicate that the representativeness of the data will be evaluated and the results documented in the final report (i.e., does the data characterize the environment well (at locations, to areas, to the site as a whole), or are data gaps evident and more data needed).</li> <li>U Indicate the results of any comparability studies (i.e., data against data, data against regulatory standards, etc.) will be documented and explained in the final report.</li> <li>U Indicate that the data will be reviewed to determine if the sampling design met the project's objectives and the results documented in the final report.</li> <li>U Indicate that the overall usability of the data will be evaluated and the results documented in the final report.</li> <li>U Indicate that the final report will analyze the project data and present observations, draw conclusions, identify data gaps, and describe any limitations in the way the data should be used or interpreted.</li> <li>U Indicate who will be responsible for performing the project evaluation and reconciliation.</li> </ul>	Section 12.0



## APPENDIX B

### DATA VALIDATION COMPLETENESS CHECKLIST

**FORMER GORHAM PROPERTY AND MASHAPAUG COVE PROJECT SAMPLING  
MODIFIED TIER I COMPLETENESS CHECKLIST**

	<u>YES</u>	<u>NO</u>
<b>1. SAMPLING AND FIELD MEASUREMENTS:</b>		
Field measurement calibration records	G	G
Groundwater field measurements (if applicable)	G	G
Soil sampling field measurements (if applicable)	G	G
Sediment sampling field measurements (if applicable)	G	G
Surface water sampling field measurements (if applicable)	G	G
Low-flow sampling field measurements (if applicable)	G	G
Documentation of field activities	G	G
Sample numbering and labeling	G	G
Chain-of-Custody records	G	G
Trip blanks	G	G
Duplicate samples	G	G
Equipment blanks	G	G
Split samples (if any)	G	G
 <b>2. LABORATORY MEASUREMENTS:</b>		
Trip blanks	G	G
Instrument blanks	G	G
Laboratory control samples	G	G
Duplicates samples	G	G
Equipment blanks	G	G
Matrix spike/matrix spike duplicates	G	G
Analysis type	G	G
Chain-of-Custody records	G	G
Surrogate recoveries	G	G
Sample Project Narratives	G	G
Split samples (if any)	G	G

TOTAL: \_\_\_\_\_

PERCENT COMPLETE: \_\_\_\_\_ %

FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
 FUSS & O'NEILL MODIFIED TIER II DATA VALIDATION CHECKLIST

PERFORMED AND, WHERE APPLICABLE,  
 WITHIN ACCEPTABLE LIMITS?

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
1. SAMPLING AND FIELD MEASUREMENTS:			
Field measurement calibration records			
pH - ± 0.3 pH units	G	G	_____
S.C. - ± 5% of calibration solution, within calibration range?	G	G	_____
Temperature - ± 0.5 °C	G	G	_____
D.O. - ± 5% of calibration solution	G	G	_____
Groundwater field measurements (if applicable)			
Water depth measured to within 0.01 ft.?	G	G	_____
Soil sampling field measurements (if applicable)			
OVM - ± 2 ppm	G	G	_____
OVA - ± 2 ppm	G	G	_____
Sediment sampling field measurements (if applicable)			
Descriptive information recorded?	G	G	_____
Surface water sampling field measurements (if applicable)			
Water depth measured to within 0.01 ft.?	G	G	_____
Low-flow sampling field measurements (if applicable)			
S.C. - ± 10%	G	G	_____
pH - ± 0.2 pH units	G	G	_____
Temperature - ± 10%	G	G	_____
Turbidity - ±5 NTU	G	G	_____
Documentation of field activities			
Site-specific information documented in field notebook?	G	G	_____
Field data sheets completed?	G	G	_____
Sample numbering and labeling			
Sample numbering conforms to sample I.D. system identified in QAPP?	G	G	_____
Chain-of-Custody records			
Chain-of-Custody forms completed?	G	G	_____



FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
FUSS & O'NEILL MODIFIED TIER II DATA VALIDATION CHECKLIST  
(Continued)

PERFORMED AND, WHERE APPLICABLE,  
WITHIN ACCEPTABLE LIMITS?

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
Trip blanks			
Trip blanks submitted, one per day?	G	G	_____
Any compounds detected in trip blanks?	G	G	_____
Duplicate samples			
Field duplicates performed, 1/20 samples?	G	G	_____
Duplicates performed on 10% of samples screened for explosives?	G	G	_____
Is percent difference within 30% for all field parameters?	G	G	_____
Equipment blanks			
Equipment blanks submitted, one per sampling day?	G	G	_____
Any compounds detected in equipment blank?	G	G	_____
Split samples (if any)			
Split samples collected?	G	G	_____
Is percent difference within 30% for split samples?	G	G	_____

2. LABORATORY MEASUREMENTS:

Trip blanks			
Trip blanks submitted, one per day?	G	G	_____
Any compounds detected in trip blanks?	G	G	_____
Instrument blanks**	G	G	_____
Laboratory control samples**	G	G	_____
Duplicates samples**	G	G	_____
Equipment blanks**	G	G	_____
Matrix spike/matrix spike duplicates**	G	G	_____
Analysis type	G	G	_____
Chain-of-Custody records	G	G	_____
Surrogate recoveries**	G	G	_____
Sample Project Narratives	G	G	_____
Split samples (if any)**	G	G	_____
Most recent EPA WP-PE sample results**	G	G	_____



FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
LABORATORY MODIFIED TIER II DATA VALIDATION CHECKLIST  
ORGANIC COMPOUNDS

PERFORMED AND, WHERE APPLICABLE, WITHIN  
ACCEPTABLE LIMITS?\*

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
1. SDG Project Narratives	G	G	_____
2. Traffic Report	G	G	_____
3. Volatiles Data	G	G	_____
a. Sample Data			
Target Compound List (TCL) Results	G	G	_____
Reconstructed total ion chromatograms (RIC) for each sample	G	G	_____
For each sample:			
Raw spectra and background-subtracted			
mass spectra of target compounds identified	G	G	_____
Mass spectra of all reported TICs with three best library matches	G	G	_____
Percent solids calculations	G	G	_____
b. Standards Data (all instruments)			
Initial Calibration Data	G	G	_____
RICs and Quan Reports for all Standards	G	G	_____
Continuing Calibration	G	G	_____
RICs and Quan Reports for all Standards	G	G	_____
Internal Standard Area Summary	G	G	_____
c. Raw QC Data			
Blank Data	G	G	_____
Matrix Spike Data	G	G	_____
Matrix Spike Duplicate Data	G	G	_____
4. Semivolatiles Data			
a. QC Summary			
Surrogate Percent Recovery Summary	G	G	_____
MS/MSD Summary	G	G	_____
Method Blank Summary	G	G	_____
Tuning and Mass Calibration	G	G	_____

FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
LABORATORY MODIFIED TIER II DATA VALIDATION CHECKLIST  
ORGANIC COMPOUNDS

PERFORMED AND, WHERE APPLICABLE, WITHIN  
ACCEPTABLE LIMITS?\*

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
b. Sample Data			
TCL Results	G	G	_____
Tentatively Identified Compounds	G	G	_____
Reconstructed total ion chromatograms (RIC) for each sample	G	G	_____
For each sample:			
Raw spectra and background-subtracted mass spectra of TCL compounds	G	G	_____
Mass spectra of TICs with 3 best library matches	G	G	_____
GPC chromatograms (if GPC performed)	G	G	_____
c. Standards Data (all instruments)			
Initial Calibration Data	G	G	_____
RICs and Quan Reports for all Standards	G	G	_____
Continuing Calibration	G	G	_____
RICs and Quan Reports for all Standards	G	G	_____
Internal Standard Areas Summary	G	G	_____
Internal Standard Areas Summary	G	G	_____
d. Raw QC Data			
Decafluorotriphenylphosphine (DFTPP)	G	G	_____
Blank Data	G	G	_____
Matrix Spike Data	G	G	_____
Matrix Spike Duplicate Data	G	G	_____
5. Miscellaneous Data			
Original preparation and analysis forms or copies of preparation and analysis log book pages	G	G	_____
Internal sample & sample extract transfer chain-of custody records	G	G	_____
Screening Records	G	G	_____
All instrument output, including strip charts from screening activities (describe or list)	G	G	_____



FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
LABORATORY MODIFIED TIER II DATA VALIDATION CHECKLIST  
ORGANIC COMPOUNDS

PERFORMED AND, WHERE APPLICABLE, WITHIN  
ACCEPTABLE LIMITS?\*

	YES	NO	COMMENTS
6. Chain-of-Custody Records	G	G	_____
Sample Log-in Sheet (Lab & DC1)	G	G	_____
Miscellaneous Shipping/Receiving Records (describe or list)	G	G	_____
_____			
_____			
7. Internal Lab Sample Transfer Records and Tracking Sheets (describe or list)	G	G	_____
_____			
_____			
8. Other Records (describe or list)	G	G	_____
_____			
_____			
9. Comments:			_____
_____			
_____			
_____			

\*\* See laboratory Quality Assurance Plan for limits.

Completed by: \_\_\_\_\_  
(Lab) (Signature) (Printed Name/Title) Date

I certify that the above information is true and accurate. I further certify that all laboratory results associated with the above analyses will be made available for review for seven (7) years following certification of this document.

Certified by: \_\_\_\_\_  
(Lab) (Signature) (Printed Name/Title) Date



FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
LABORATORY MODIFIED TIER II DATA VALIDATION CHECKLIST  
ORGANIC COMPOUNDS

PERFORMED AND, WHERE APPLICABLE, WITHIN  
ACCEPTABLE LIMITS?\*

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
1. SDG Project Narratives	G	G	_____
2. Inorganic Analysis Data Sheet	G	G	_____
3. Initial and Continuing Calibration Verification	G	G	_____
4. CRDL Standard for AA and ICP	G	G	_____
5. Blanks	G	G	_____
6. ICP Interference Check Sample	G	G	_____
7. Spike Sample Recovery	G	G	_____
8. Post Digest Spike Sample Recovery	G	G	_____
9. Duplicates	G	G	_____
10. Laboratory Control Sample	G	G	_____
11. Standard Addition Results	G	G	_____
12. ICP Serial Dilutions	G	G	_____
13. Instrument Detection Limits, Quarterly	G	G	_____
14. ICP Interelement Correction Factors, Annually	G	G	_____
15. ICP Linear Ranges Quarterly	G	G	_____
16. Preparation Log	G	G	_____
17. Analysis Run Log	G	G	_____
18. ICP Raw Data	G	G	_____
19. Furnace AA Raw Data	G	G	_____
20. Mercury Raw Data	G	G	_____
21. Percent Solids Calculations	G	G	_____
22. Digestion Logs	G	G	_____
23. EPA Shipping/Receiving Records (List all individual records)	G	G	_____
Chain-of Custody Records	G	G	_____
Sample Log-In sheet	G	G	_____
24. Miscellaneous Shipping/Receiving Records (List all individual records)	G	G	_____
			_____
			_____
			_____



FORMER GORHAM PROPERTY AND MASHAPAUG COVE  
LABORATORY MODIFIED TIER II DATA VALIDATION CHECKLIST  
ORGANIC COMPOUNDS

PERFORMED AND, WHERE APPLICABLE,  
WITHIN ACCEPTABLE LIMITS?\*

	<u>YES</u>	<u>NO</u>	<u>COMMENTS</u>
25. Internal Lab Sample Transfer Records and Tracking Sheets (Describe or List)			
_____	G	G	_____
_____	G	G	_____
26. Internal Original Sample Preparation and analysis Records (Describe or List			
Preparation Records	G	G	_____
Analysis Records	G	G	_____
Description	G	G	_____
27. Other Records (Describe or List)			
_____	G	G	_____
_____	G	G	_____
28. Comments:			
_____			
_____			

\*\* See laboratory Quality Assurance Plan for limits.

Completed by: \_\_\_\_\_  
(Lab) (Signature) (Printed Name/Title) Date

I certify that the above information is true and accurate. I further certify that all laboratory results associated with the above analyses will be made available for review for seven (7) years following certification of this document.

Certified by: \_\_\_\_\_  
(Lab) (Signature) (Printed Name/Title) Date



## APPENDIX C

### FUSS & O'NEILL, INC. STANDARD OPERATING PROCEDURES FOR SAMPLE COLLECTION

Upon arrival on-site, all Fuss & O'Neill field personnel will follow the following guidelines:

1. The client/owner will be notified of site visits.
2. Field personnel will always carry their business cards for identification purposes.
3. Field personnel will strictly adhere to policies in effect at the client's facilities. An example of such a policy is signing in and out of buildings or offices and wearing facility specified safety gear (hard hats, eyeglasses).
4. The client/owner's property will be respected at all times.
5. Field personnel will not discuss specifics of sampling or contaminants with any site employees or passers-by without authorization from project management and the client.
6. Field personnel will not be permitted to smoke in the client's presence or while in indoor facilities. **In addition, no smoking will be permitted in the vicinity of sample collection.**
7. All field activities will be conducted following the established sampling plan and the site health and safety plan for the site.
8. Wells will be locked and maintained in good condition between sampling events.
9. The homeowner will be notified prior to any domestic well sampling. If no one is home and a sample cannot be obtained, field personnel will leave a note to inform the resident of the sampling attempt and the name of a contact person with whom to reschedule. A business card should always accompany this note.
10. When domestic wells are purged from an outside tap, a hose will be attached whenever possible to direct the water away from the building.
11. Contaminated and/or dirty protective gear will be properly decontaminated and removed prior to entering on-site buildings and offices.
12. No discarded materials will be left at sample locations. All trash, which has accumulated at a site as a result of field activities, will be collected and discarded back at Fuss & O'Neill offices.
13. Field Staff will keep company vehicles clean and in presentable condition while conducting field activities.
14. Fuss & O'Neill staff is not to sign a manifest for hazardous waste transport under any circumstances.

## **FIELD NOTEBOOKS**

All field personnel will carry a bound field notebook. All field activities will be documented in the field notebook, regardless of whether or not those activities involve sample collection. Each employee's book will be numbered sequentially with the format of the employee number followed by the book number (i.e. **156-01**) and will be labeled on the cover as such with the range of dates covered by the book (i.e. 10/23/03 to 8/17/04). Each page of the field notebook book will be numbered with the employee number, the book number, and the page number (i.e. **156-01-01, 156-01-02, 156-01-03**, etc.). The field notebook will document site-specific information such as:

- Project name and location
- Names of other Fuss & O'Neill personnel involved in field activities
- Time and date of arrival at the site
- Weather conditions
- Sampling locations and corresponding sample numbers
- Documentation of field calibration of instruments
- Conversations with individuals on site
- Any unusual events or observations
- All information not recorded on field data sheets
- Time of departure from the site

For field investigations that involve the collection of samples, additional forms of documentation are required. See SOPs 020100, 020200, 020300, 020400, 020500, and 020600.

## INTRODUCTION

All samples collected must have unique identifiers (Sample IDs). Since sampling locations may be sampled many times (e.g., quarterly monitoring programs), there must be a means of distinguishing samples from each other, whether from one sampling event or multiple events.

Fuss & O'Neill has adopted a protocol for assigning sample identification numbers. It is necessary to follow this protocol to ensure that:

- Sample locations are blind to the laboratory
- Analytical requests can be easily communicated with the laboratory
- Analytical data provided by the laboratory can be assigned to the correct sample location for reporting purposes

The only time sample IDs are to deviate from this protocol is when the client has specified an alternative sample numbering scheme.

## SAMPLE IDENTIFICATION NUMBER

Each sampling location will be assigned a number by which samples can be identified. An example of a sample identification number is as follows:

XXXYYMMDD-## (e.g., 156050608-01)

This 11-digit code contains three types of information about the collection of the sample.

XXX	The employee number of the individual who collected the sample or supervised the sampling event
YY	Sample year
MM	Sample month
DD	Sample day
##	Assigned chronologically. Generally, the first sample collected during a sampling event is -01 and the numbers increase until the sampling event is completed. If the event continues over several days, the numbering sequence continues without returning to -01.

For sampling events that will involve the collection of over 100 samples, it is important to use a three-digit identifier (e.g., 156050608-001).

When multiple sample containers are filled from one sample location, all such samples are assigned the same sample identification number. Common situations where this will happen include, but are not limited to:

- Multiple containers required for various analytical parameters

- Split samples that are being submitted to different laboratories
- Sampling events that require sampling at the same location over two or more days due to insufficient sample volume available (e.g., low yield monitoring wells)

An exception to this rule is duplicate samples which should be assigned unique sample identification numbers to keep the duplicates blind to the laboratory.

## INTRODUCTION

All samples must be labeled in order to provide pertinent information to everyone who will be handling the sample. It is imperative that labels be applied to each sample container to ensure that all samples get transferred together.

## SAMPLE LABELS

A sample label will be affixed to each sample container at the time of collection. Labels must be completed legibly with waterproof ink to prevent obliteration of the label. An example of a sample label is provided on Figure 020200.

The following information will be recorded on each label with waterproof ink:

- Sample identification number
- Project name
- Project location
- Project number
- Date of sample collection
- Time of sample collection
- Name/Initials of sampler
- Type of preservation

Labels are created using *Microsoft Word*, File, New..., Field Services Tab, Labels\_Bottles.dot.

**Figure 020200**

FUSS & O'NEILL, INC., ENVIRONMENTAL SERVICES  
146 HARTFORD ROAD, MANCHESTER, CT 06040  
(860) 646-2469

---

Date:

Project:

Location:

Project #:

Preservative: ICE / Hcl / HNO3 /  
H2SO4 / FHN03

Sampler:

Time:

Sample ID:

## **INTRODUCTION**

Many different types of environmental samples may be collected. These include, but are not limited to: groundwater, surface water, soil, sediment, concrete chips, wipes, indoor air, soil gas, and test pits. While some of the necessary documentation will be standard regardless of the type of sample (e.g., sample ID, date, time, sampler identification), each type also has information that is unique.

## **FIELD DATA SHEETS**

Field data sheets have been developed for most types of samples encountered during field activities so that pertinent information is recorded at the time of sampling. Field personnel will thoroughly complete a field data sheet for each sample collected at the time of sampling.

At the conclusion of each sampling event, the field data sheets will be given to the project manager for review. This review should be conducted as soon as possible to ensure that, if edits are required, they can be done efficiently.

Examples of each of the existing field data sheets are provided as attachments to this Standard Operating Procedure.

# Air Sampling Field Data

Client/Project Name:		
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

## Sample Location Info

--

## Sample Data

Sample Data	Container	Quantity	Preservative
Date: _____ Sampler: _____ Start Time: _____ Finish Time: _____ Sample Time: _____			

Comments:

# Concrete Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

## Sample Location Info

## Sample Data

	Container	Quantity	Preservative
Date: _____ Start Time: _____ Sampler: _____ Finish Time: _____  Sampling Method: Chisel / Core / Drill Bit / Other _____			

## Description Data

Organic Vapor Reading: \_\_\_\_\_ Instrument: \_\_\_\_\_  
  
 Appearance:

## Comments:

# Concrete Sampling Field Data

Client/Project Name: _____	Project # _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____		
Date: _____	Weather: _____	

Sample #:	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Core Sampler / Shovel / Hammer Drill / Trowel / Other _____ Field Decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Description: _____ Sample Depth: _____ PetroFLAG/OVM _____			

Sample #:	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Core Sampler / Shovel / Hammer Drill / Trowel / Other _____ Field Decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Description: _____ Sample Depth: _____ PetroFLAG/OVM _____			

# Direct Push Groundwater Sample Field Data Sheet

Client/Project Name:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:		
<b>Sample#:</b>	<u><b>Sample Location</b></u>	

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Approx. sample depth: _____ (ft) Sampling Method: Bailer / Peristaltic / Watera / Other _____ Filtered in Field: No / @ Sample Location			
Method of Filtration: Pressure / Vacuum / Syringe Pump ID #: <input style="width: 50px;" type="text"/> Filter: Disposable / Other _____			
Appearance:			
Comments:			

\* - Organic-free DI water used in these containers.

Comments:

# Sediment Sampling Field Data

Client/Project Name: _____ Project Location: _____ Date: _____ Weather: _____ Sampler(s): _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
---	----------------------------	--

Sample #:	Container	Quantity	Preservative
<b>Site ID:</b>			
GPS Coord. _____ N _____ W   Photo #: _____			
Water Quality Parameters      Surface      Bottom			
Temperature (C)      _____      _____			
Conductivity (uS/cm)      _____      _____			
pH / ORP (mV)      _____ / _____      _____ / _____			
DO (mg/L / %)      _____ / _____      _____ / _____			
Sampling Device: Core Sampler – Type: _____			
Dredge – Type: Ponar / Ekman / Other: _____			
Field decon: Yes / No / Dedicated			
Type of Sample: Grab / Composite / Other _____			
Sample Depth: _____ Feet			

# Sediment Sampling Field Data

Client/Project Name: _____ Project Location: _____ Date: _____ Weather: _____ Sampler(s): _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
---	----------------------------	--

<b>SAMPLE #:</b>	Container	Quantity	Preservative
<b>SITE ID:</b>			
GPS Coord. _____ N _____ W   Photo #: _____			
Water Quality Parameters                      Surface                      Bottom			
Temperature (C)                      _____                      _____			
Conductivity (uS/cm)                      _____                      _____			
pH / ORP (mV)                      _____ / _____                      _____ / _____			
DO (mg/L / %)                      _____ / _____                      _____ / _____			
Sampling Device:    Core Sampler – Type: _____			
Dredge – Type: Ponar / Ekman / Other: _____			
Field Decon:                      Yes / No / Dedicated			
Type of Sample:                      Grab / Composite / Other _____			
Sample Depth: _____ Feet			
<b>NOTES &amp; COMMENTS</b>			

<b>SAMPLE #:</b>	Container	Quantity	Preservative
<b>SITE ID:</b>			
GPS Coord. _____ N _____ W   Photo #: _____			
Water Quality Parameters                      Surface                      Bottom			
Temperature (C)                      _____                      _____			
Conductivity (uS/cm)                      _____                      _____			
pH / ORP (mV)                      _____ / _____                      _____ / _____			
DO (mg/L / %)                      _____ / _____                      _____ / _____			
Sampling Device:    Core Sampler – Type: _____			
Dredge – Type: Ponar / Ekman / Other: _____			
Field Decon:                      Yes / No / Dedicated			
Type of Sample:                      Grab / Composite / Other _____			
Sample Depth: _____ Feet			
<b>NOTES &amp; COMMENTS</b>			

# Equipment Blank Field Data

Client/Project Name:  Project Location:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
<b>Sample#:</b>	<u>Well ID</u> <b>Equip Blank</b>	

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Blank Water Supplied By: Lab / F&O / Other _____ Equipment Used: Bailer / Filter / Pump / Other _____ Filtered in Field? No / @ Vehicle Method of Filtration: Pressure / Vacuum / Syringe Pump ID #: <input style="width: 50px;" type="text"/> Filter: Disposable / Other _____			
Appearance:			
Comments:			

\* - Organic-free DI water used in these containers.

Comments:

# Field Blank Field Data

Client/Project Name:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:		
<b>Sample#:</b>	<u>Well ID</u> <b>Field Blank</b>	

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Blank Water Supplied By:    Lab / F&O / Other _____			
Comments:			

\* - Organic-free DI water used in these containers.

Comments:

# Flow Meter Field Data

Client/Project Name: Project Location:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
	<i>Sample Location</i>	

## Sample Location Info

## Sample Data

Date: _____	Time: _____
Sampler: _____	Flow: _____
Weather: _____	
Water Temperature: _____	
Pipe Outside Diameter: _____	
Pipe Material: _____	
Pipe Circumference: _____	

Comments:



# Methane Field Data Sheet

Client/Project Name:	PROJECT #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	Date:	

Sampler: \_\_\_\_\_ Weather: \_\_\_\_\_  
 Start Time: \_\_\_\_\_ Finish Time: \_\_\_\_\_  
 Meter Used: \_\_\_\_\_ Calibrated date/time: \_\_\_\_\_ Calibration Gas Used: \_\_\_\_\_

Location                      % Methane                      %LEL                      Comments

# Monitoring Well Field Data Sheet

Client/Project Name: _____ Project Location: _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
<b>Sample#:</b> _____	<u>Well ID</u> _____	

## Elevation Data

Date: _____	Time: _____	Well Diameter (inches): _____
Depth (feet)	+ Correction	= True Depth
Water Level PVC	+	=
Water Level TPS	+	=
Bottom of Well	+	=
Measuring Device ID#: _____		Water Column Height: _____ (feet) Gal/foot x 3 factor: x _____
Comments: _____		Volume to be Purged: _____ (gal)
Sampler: _____		Weather: _____

## Well Condition Checklist

[circle appropriate item(s); cross out if not applicable]

<i>General Condition:</i> Good / Needs Repair <i>Protective Steel:</i> OK / Cracked / Leaking / Bent / Loose / None <i>Well # Visible?:</i> Y / N <i>Well Cap:</i> Good / Broken / None <i>Evidence of rain water between steel and PVC?:</i> Y / N <i>Evidence of ponding around well?:</i> Y / N <i>Gopher-type holes around collar?:</i> Y / N Comments: _____	<i>Is well plumb?:</i> Y / N <i>Lock:</i> Good / Broken / None <i>Rust around cap?:</i> Y / N <i>PVC Riser:</i> Good / Damaged / None <i>Concrete collar:</i> OK / Cracked / Leaking / None <i>Other Evidence:</i> Rodents / Insects / None <i>Curb Box:</i> Y / N (key is: Hex/Pent/Other) <i>Curb Box Gasket:</i> OK / Replace / Other _____	
--	---	--

## Purge Data

Start Time: _____ Stop Time: _____ Total Time Purged: _____ Pump Rate: _____ (gpm) Volume Purged: _____ (gallons)	Purge Device: Dedicated / Nondedicated Device type: Bailer / Peristaltic / Submersible / Bladder Comments: _____ Well Yield: High / Moderate / Low / Dry
---	---

## Sample Data

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Approx. sample depth: _____ (ft) Weather: _____ Sampling method: Bailer / Peristaltic / Bladder Bailer type: SS 2" / SS 1.25" / SS Short / PVC 2" / Other _____ Bailer Cord: Dedicated / Nondedicated Filtered in Field?: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID # _____ Filter: Disposable / Other _____ Field Decon: Bailer / Tubing / Other _____ Appearance: _____			

## Field Parameters

Note: SC calculation based on (temp) at time of SC measurement.

Instrument ID#		Instrument ID#	
pH	Temp		Spec. Cond.

# Monitoring Well Abandonment Log

Client/Project Name:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<i>Well ID</i>	

## Water Column Data

Date:	Time:	Well Diameter (inches): _____												
	<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 30%;">Depth (feet)</th> <th style="width: 10%;">+ Correction</th> <th style="width: 60%;">= True Depth</th> </tr> </thead> <tbody> <tr> <td style="padding: 2px;">Water Level PVC</td> <td style="text-align: center; padding: 2px;">+</td> <td style="text-align: center; padding: 2px;">=</td> </tr> <tr> <td style="padding: 2px;">Water Level TPS</td> <td style="text-align: center; padding: 2px;">+</td> <td style="text-align: center; padding: 2px;">=</td> </tr> <tr> <td style="padding: 2px;">Bottom of Well</td> <td style="text-align: center; padding: 2px;">+</td> <td style="text-align: center; padding: 2px;">=</td> </tr> </tbody> </table>	Depth (feet)	+ Correction	= True Depth	Water Level PVC	+	=	Water Level TPS	+	=	Bottom of Well	+	=	Water Column Height: _____ (feet) gal/foot: _____ Volume: _____ (gal) Gallons x 13 = _____ (ml bleach)
Depth (feet)	+ Correction	= True Depth												
Water Level PVC	+	=												
Water Level TPS	+	=												
Bottom of Well	+	=												
Measuring Device ID: _____		Time Bleach Added to Well: _____												
Comments: _____		Sampler: _____												
		Weather: _____												

## Description of Abandonment Procedures:

## Backfill Info

Interval: _____ - _____ (ft)	(Approximate volumes if available)
Description: Bentonite Grout / Bentonite Chips	Volume: _____ cu. ft.
Other: _____	

## Surface Completion

Interval: _____ - _____ (ft)	(Approximate volumes if available)
Description: Concrete / Other: _____	Volume: _____ cu. ft.

\_\_\_\_\_  
Licensed Non-Water Well Driller Name

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date



# Monitoring Inspection Datasheet

<b>Client/Project Name:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
<b>Project Location:</b>	
<b>PROJECT #:</b>	
<b>WELL ID#:</b>	

## Elevation Data

Date: 5/19/05	Time:	Well Diameter (inches): _____
	Depth (feet)	+ Correction = True Depth
Water Level PVC		+ =
Water Level TPS		+ =
Bottom of Well		+ =
Sampler: _____		
Measuring Device ID#: _____		
Weather: _____		
Comments:		

## Well Condition Checklist

[circle appropriate item(s); cross out if not applicable]

Curb Box: Y / N (key is: Hex/Pent/Other)
Stand Pipe: Y / N
General Condition: Good / Needs Repair
Is well plumb?: Y / N
Protective Steel: OK / Cracked / Leaking / Bent / Loose / None
Locking Mechanism: Y / N / Broken
Lock: Good / Broken / None
Well # Visible?: Y / N
Rust around cap?: Y / N
Well Cap: Good / Broken / None
PVC Riser: Good / Damaged / None
PVC Cap: Y / N
Evidence of rain water between steel and PVC?: Y / N
Concrete collar: OK / Cracked / Leaking / Pitted / None
Evidence of ponding around well?: Y / N
Other Evidence: Rodents / Insects / None
Gopher-type holes around collar?: Y / N
Curb Box Gasket: OK / Replace / Other
Comments:

# Potable Water Field Data

Client/Project Name:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:		
<b>Sample#:</b>	<u>Well ID</u>	

## Sample Location Data

Document all sample attempts

Name: _____	Date	Time	Reason for not sampling
Street Address: _____			
City, State, Zip: _____			
Special instructions:			
Sample taken on →			XXXXXXXXXXXXXXXXXXXXXXXXXXXX

## 5 minute purge

Sample taken from:	Outside tap / Inside sink / Pre-filter / Basement Tap/ Other _____
Filter bypassed by:	Sample point / Not Bypassed / No Filter / Other _____
Start time:	_____
Stop time:	_____
Total Time Purged:	_____
Comments:	

## Sample Data

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____			
Sampler: _____ Weather: _____			
Appearance:			
Comments:			

## Field Parameters

Note: SC calculation based on (temp) at time of SC measurement.

Instrument ID#	Instrument ID#		
pH	Temp	(Temp)Corr Factor x Calib Factor x Conductivity =	Spec. Cond.
	( )	x x =	

Comments:

# Pumping Well Field Data

Client/Project Name:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<i>Sample Location</i>	
Sample #:		

## Sample Location Info (sketch map including location of sample)

Special Instructions;

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Method: Bailer / Peristaltic / Grab / Other _____ Sampling Device: SS 2" / SS 1.25" / SS Short / PVC 2" / Other _____			
Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: <input style="width: 50px;" type="text"/> Filter: Disposable / Other _____ Field Decon: Filter / Tubing / Other _____ Appearance:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

## Pumping Data

Time	Water Level	Q (rate in gpm)	Totalizer Reading

## Type of Well

- Public Supply
- Interceptor
- Hydrocarbon Recovery
- Other \_\_\_\_\_

## Comments:

# Soil Gas Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

## Sample Location Info

## Sample Data

	Container	Quantity	Preservative
Date: _____      Start Time: _____ Sampler: _____      Finish Time: _____  Time Interval: _____			

Comments:

# Soil Sampling Field Data

Client/Project Name: _____		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____	<b>PROJECT #:</b> _____	
<b>Sample#:</b> _____	Sampling Location _____	

Sample Location Info (sketch map including location of stockpile)

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Geoprobe / Shovel / Split Spoon / Trowel / Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____  Sample Depth: _____ PetroFLAG / OVM _____			

Comments:



Sampler: _____ Time: _____ Sampling Device: Auger / Geoprobe / Core Sampler / Shovel / Split Spoon / Scoop/ Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Soil Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			
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## Soil Stockpile Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

### Sample Location Info (sketch map including location of stockpile)

### Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Core Sampler / Shovel / Trowel / Other _____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____			

### Description Data

Generic Sample Description: Sand, Gravel, etc.  Source of Contamination:   Analytical parameters:	<b>Previous stockpile sampling</b> <b>Y / N</b>
--	--

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Comments:



## Stormwater Field Data Sheet

Client/Project Name:	Project #:
Project Location:	<i>Location ID</i>
Sample #:	

### Sample Location Info

Special Instructions:
-----------------------

### Sample Data

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____	32 oz. Amber	1	H2SO4
Sampler: _____ Weather: _____	250 mL Plastic	1	HNO3
	500 mL Plastic	1	H2SO4
Estimated Flow Rate(GPM) _____	250 mL Plastic	1	As Is
Stagnant / Dry / Other _____	1 L Plastic	1	As Is
Appearance:			
Comments:			

### Field Parameters

Parameter	Instrument	Value
pH		
Temperature		
rain pH		

Comments:

# Surface Water Field Data

Client/Project Name:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b><i>Monitoring Location</i></b>	
Sample #:		

## Sample Location Info

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____			
Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: <input style="width: 50px;" type="text"/> Filter: Disposable / Other _____ Field Decon: Tubing / Other _____			
Appearance:			
Comments:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

Comments:

# Surface Water Field Data

Client/Project Name: _____ Project Location: _____ Date: _____ Weather: _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
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<b>Sample #:</b> _____ <b>Sample Location</b> Date: _____ Time: _____ Sampler: _____ Weather: _____ Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____ Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: _____ Filter: Disposable / Other _____ Field Decon: Tubing / Other _____ Appearance: _____ Comments: _____	Container	Quantity	Preservative
---	-----------	----------	--------------

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

<b>Sample #:</b> _____ <b>Sample Location</b> Date: _____ Time: _____ Sampler: _____ Weather: _____ Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____ Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: _____ Filter: Disposable / Other _____ Field Decon: Tubing / Other _____ Appearance: _____ Comments: _____	Container	Quantity	Preservative
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## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

# Tank Grave Soil Sampling Field Data

Client/Project Name: _____	Project #: _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____	Site ID: _____	
Date: _____ Weather: _____	Tank ID: _____	

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Date: _____ Time: _____ Sampler: _____ Weather: _____ Sampling Device: Auger / Core Sampler / Shovel / Split Spoon / Trowel Other _____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Sol Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Date: _____ Time: _____ Sampler: _____ Weather: _____ Sampling Device: Auger / Core Sampler / Shovel / Split Spoon / Trowel Other _____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Sol Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Date: _____ Time: _____ Sampler: _____ Weather: _____ Sampling Device: Auger / Core Sampler / Shovel / Split Spoon / Trowel Other _____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Sol Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Date: _____ Time: _____ Sampler: _____ Weather: _____ Sampling Device: Auger / Core Sampler / Shovel / Split Spoon / Trowel Other _____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Sol Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

# Trip Blank Field Data

Client/Project Name:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:		
<b>Sample#:</b>	<u>Well ID</u> <b>Trip Blank</b>	

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____ Blank Water Supplied By: Lab / F&O / Other _____  Comments:	Voa	2	HCl

Comments:

# Waste Sampling Field Data

Client/Project Name: _____	Project #: _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____	<i>Sample Location</i>	
Sample #: _____		

## Sample Location Info

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Core Sampler / Shovel / Split Spoon/ Plastic Scoop / Trowel / Other _____  Field Decon: Yes / No / Dedicated  Type of Sample: Grab / Composite / Other _____			

## Description Data

pH Reading: \_\_\_\_\_ Instrument: \_\_\_\_\_  
Temp: \_\_\_\_\_  
OVM (ppm): \_\_\_\_\_  
  
Sample Description: (odor/color/consistency)  
  
  
  
Type of Container Sampled: Drum / Tank / Tote / Other: \_\_\_\_\_

## Comments:

# Wastewater Field Data

Client/Project Name:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: Willimantic CT	<b><i>Monitoring Location</i></b>	
Sample #:		

## Sample Location Info

Permit: \_\_\_\_\_

Discharge Location: \_\_\_\_\_

Special Instructions: \_\_\_\_\_

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Estimated Flow Rate (GPM) <u>see comments</u> Stagnant / Dry / Other _____			
Filtered in Field: No / @ Well / @ Vehicle Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: <input style="width: 50px;" type="text"/> Filter: Disposable / Other____ Field Decon: Tubing / Other _____			
Appearance:			
Comments:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

Comments:

# Wipe Sampling Field Data

Client/Project Name: _____	Project #: _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____	<i>Sample Location</i>	
Sample #: _____		

## Sample Location Info

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Wipe / Other  Wipe Area: _____ x _____			

## Description Data

Organic Vapor Reading: \_\_\_\_\_ Instrument: \_\_\_\_\_  
  
  
Appearance: \_\_\_\_\_

## Comments:







### UST Excavation Field Data Sheet

Client/Project Name: \_\_\_\_\_

Project Location: \_\_\_\_\_

**PROJECT #:** \_\_\_\_\_

**SITE ID:** \_\_\_\_\_

**Sampler Name:** \_\_\_\_\_

**Weather:** \_\_\_\_\_

**Tank ID:** \_\_\_\_\_

**Date:** \_\_\_\_\_

**GENERAL CONTRACTOR:**

**TANK DISPOSAL:**

Hauler Name: \_\_\_\_\_

Disposal Site Name: \_\_\_\_\_

**LIQUID DISPOSAL:**

Hauler Name: \_\_\_\_\_

Disposal Site Name: \_\_\_\_\_

Quantity: \_\_\_\_\_ gallons

**SOIL DISPOSAL:** (Separate FDS required)

Disposal Site Name: \_\_\_\_\_

Estimated Quantity: \_\_\_\_\_ cubic yards

Location of Stockpile: \_\_\_\_\_

#### TANK DATA

Data Removed: \_\_\_\_\_

Dimensions: Diameter: \_\_\_\_\_ ft. X Length: \_\_\_\_\_ ft.

Capacity: \_\_\_\_\_ gallons

Tank Material: Steel / Fiberglass / Other (describe) \_\_\_\_\_

**TANK CONDITION**

\_\_\_\_\_ Good (not visibly deteriorated)

\_\_\_\_\_ Fair (some corrosion, no obvious signs of tank failure)

\_\_\_\_\_ Poor (visible holes, obvious failure)

\_\_\_\_\_ Tank Not Inspected

Tank Used For: \_\_\_ Gasoline \_\_\_ Diesel \_\_\_ Waste Oil \_\_\_ #2 Oil \_\_\_ #4 Oil \_\_\_ #6 Oil \_\_\_ Other (describe)

Comments:

#### FIELD OBSERVATIONS

**EXCAVATION DETAILS**

Generic Soil Description: \_\_\_\_\_ (Sand/Gravel/etc.)

Evidence of Bedrock? No \_\_\_\_\_ Yes \_\_\_\_\_ Depth \_\_\_\_\_

Groundwater Present? No \_\_\_\_\_ Yes \_\_\_\_\_ Depth \_\_\_\_\_

Comments:

- Attach sketch showing limits and dimensions of excavation and stockpile(s) and indicate sample locations with sample IDs
- Confirm north arrow
- Describe pipe trench sampling
- Describe condition (staining, discoloration, etc.) of concrete pad



## **INTRODUCTION**

Samples collected during field investigations and other activities are always subject to potential review by regulatory agencies and/or subpoena for litigation purposes. In order to document that control of the samples has been maintained at all times, it is necessary to utilize chain-of-custody forms to document where the sample is at all times from collection in the field to analysis in the laboratory.

## **CHAIN-OF-CUSTODY RECORDS**

Chains-of-custody (COC) forms consist of four copies. One copy is kept by each person who has had custody. At a minimum, COCs will include the following information:

- Chain-of-custody identification number
- Project/client name
- Project location
- Project number
- Laboratory conducting analysis
- Name/location of party to receive laboratory report
- Name/location of party to receive laboratory invoice
- Sample number
- Sample type
- Number and type of sample containers
- Type of preservatives
- Signature and affiliation of sampler
- Date and time of collection
- Signatures of people involved in chain of possession
- Dates and times of sample transfer

A sample of a completed COC is attached.

With each transfer of the samples from one person to another or to a sample refrigerator, the transfer must be documented. All samples listed on the COC must be verified as being present by both the person relinquishing the samples and the one receiving them. This verification is documented by checking the appropriate transfer box for each sample on the right side of the COC.

1. When the sampler relinquishes the samples to someone else or to the sample refrigerator, the first transfer box is checked and the sampler signs the bottom of the COC as “relinquisher”.
2. The person receiving the samples verifies that all samples are present and signs the bottom of the COC to accept the samples and documents the date and time. If the samples are

relinquished to the Sample Refrigerator in one of the Fuss & O'Neill offices, the "relinquisher" documents the receiver as "F&O Fridge".

3. The "relinquisher" keeps the last copy of the COC.
4. With each subsequent transfer of the samples, the last person to accept custody of the samples completes the transfer by checking the next transfer box for each sample and signing the COC.
5. The person receiving the samples verifies that the transfer boxes have been checked and signs the COC to acknowledge receipt.
6. The "relinquisher" keeps the last copy of the COC and forwards it to the project manager or his/her designee. When samples are relinquished from the F&O Fridge, they must be removed by F&O personnel. A copy of the COC is to be left in the filing basket in the field operations laboratory.
7. If subsequent transfers of the samples are made, steps 4 through 6 are repeated as necessary.

Each Fuss & O'Neill COC allows for samples to be transferred up to four times. If more than four transfers are necessary, additional COCs will be completed until the samples arrive at the laboratory. The original record will be returned to Fuss & O'Neill with the sample analysis report.

## **INTRODUCTION**

All samples collected by Fuss & O'Neill during investigations and other activities are subject to potential review by regulatory agencies and/or subpoena for litigation purposes. The field data sheets and chains-of-custody are used to document sample collection and transfers between collection and arrival at the laboratory. In order to maintain a comprehensive record of samples collected, log books are kept in the Field Operations area in each office.

## **SAMPLE LOGBOOKS**

There are two sample logbooks in every F&O office: a refrigerator logbook and a "master" sample logbook. Every sample collected by F&O employees will be recorded in the "master" logbook in each F&O office. The logbook will contain:

- job name
- job number
- date collected
- sample id
- sample location
- initials of sampler
- where the samples were relinquished on what date

The refrigerator logbook will only be filled out if the samples are relinquished to the F&O refrigerator in each respective office. The refrigerator logbook will contain:

- job name
- job number
- date collected
- chain of custody id
- initials of sampler
- dates of relinquishing

See section 030100 for relinquishing procedures.

## INTRODUCTION

Sample handling procedures are important for preserving the quality of data collected in the field.

## SAMPLE HANDLING

Field samples are to be collected in lab-supplied containers. The type of container used will depend upon the analysis to be conducted. Samples will be preserved in the field with ice packs or ice and lab-supplied chemicals according to the instructions of the laboratory. The pH of the samples will be verified in the field after preservation by pouring a small amount of the sample onto a piece of pH paper.

Liquid samples collected for volatile organic analyses will be preserved with HCl to a pH of <2. Any acid used for preservation will be added to the VOA vial before the sample is collected to ensure that adequate mixing occurs. The vial will not be reopened once it has been properly sealed.

Once any necessary pH adjustments have been made, the sample container will be placed in a cooler to reduce the temperature to approximately 4 degrees Celsius. **The outside of the sample container will be rinsed with tap water before it is placed into the cooler.**

A chain-of-custody form, establishing the party responsible for the samples, will be filled out in the field after sample collection. This form will document the entire history of sample custody and all transfers of sample possession. A description of a chain-of-custody form is provided in the section entitled "Documentation of Field Activities." Once the sample information has been entered, field personnel will sign the form in the lower left-hand corner, noting also their affiliation with Fuss & O'Neill, the date and the time. The samples will then remain in the custody of the sample collector until that person relinquishes them to another party as discussed in Standard Operating Procedure No. 030100.

## **RELINQUISHING SAMPLES DIRECTLY TO THE LABORATORY**

Under most circumstances, samples are transported from the site directly to the laboratory. Upon arriving at the laboratory, the sample collector will sign the chain-of-custody, thereby relinquishing the samples to the laboratory. The laboratory technician accepting the samples will make sure that all of the sample containers are present by checking transfer box next to each sample. The technician will then sign the chain-of-custody as well as record the date and time of sample transfer to acknowledge the transaction. The last sheet of the chain-of-custody will be retained by the sample collector, to be returned to Fuss & O'Neill.

## **RELINQUISHING SAMPLES TO THE REFRIGERATOR**

Samples will be transferred to the refrigerator when they cannot be taken to the laboratory directly, or when the laboratory has agreed to pick them up at Fuss & O'Neill. Upon arriving at Fuss & O'Neill, the sample collector will unlock the refrigerator. As samples are transferred from the coolers to the refrigerator, the sample collector will check the box next to each sample to be sure that all samples are present. The sample collector will then sign the chain-of-custody, writing "F&O refrigerator" as the receiving party. The date and time at which the samples were placed into the refrigerator will also be recorded. The chain-of-custody will remain in the refrigerator with the samples. The serial number of each chain-of-custody form will be recorded in the refrigerator log book along with the sample collector's initials, the date and the time. The refrigerator will then be locked.

The samples must be relinquished from the refrigerator to the F&O employee who will transfer them to the laboratory. As samples are removed from the refrigerator, they will be checked against the chain-of-custodies to be certain that all are properly accounted for. The F&O employee will then write "F&O refrigerator" as the relinquishing party and sign to accept the samples. In addition, that person will write the date and the time of the transfer on the chain-of-custody as well as in the sample log book. Transfer of the samples from the F&O employee to the laboratory will then follow the procedure outlined in "Transferring the Samples Directly to the Laboratory," above.

## **INTRODUCTION**

Decontamination of personal protective equipment (PPE) and field equipment is required when conducting activities which involve contact with hazardous materials.

The use of PPE and proper decontamination of PPE reduces the risk of exposure to hazardous materials. Field personnel should consult project personnel and relevant site-specific documents in order to determine the nature of the contamination at the site and the correct PPE to use while conducting site activities.

Field equipment and instruments which encounter hazardous materials or contamination must be decontaminated. If thorough decontamination is to be conducted in-house, all equipment must nevertheless be cleaned of all obvious contamination prior to leaving the site. A description of field cleaning and decontamination requirements for equipment and instruments and in-house decontamination techniques are described below.

## **FIELD DECONTAMINATION OF PERSONAL PROTECTIVE EQUIPMENT**

Personal protective equipment which comes into contact with the sample will be replaced or decontaminated in the field between sampling locations.

In most cases, only gloves will need to be decontaminated or replaced between sampling locations to prevent the inadvertent cross-contamination of samples. With gloves on, field personnel will wash their hands in the following reagent sequence: non-phosphate soap solution scrub, tap water rinse, 20% methanol solution scrub and a final deionized water rinse. This procedure will be upgraded depending on the nature of the contamination.

All contaminated/soiled PPE will be discarded or decontaminated prior to leaving the site. The removal of this equipment will be undertaken outside of the exclusion zone, if one has been established, in the following order.

1. Tyvek suits will be removed and discarded.
2. Boots will at least be rinsed with water prior to being removed. The boot wash procedure will be upgraded depending on the nature of the contamination. Under some circumstances, disposable boots may be used and discarded prior to leaving the site.
3. When a respirator is utilized, it will be worn until all personal protective equipment except the gloves has been removed. The respirator will be washed with soap and water and wiped down with alcohol after use. Respirator cartridges will be discarded.
4. Gloves will be removed after all other personal protective equipment has been properly decontaminated and removed. They will either be decontaminated or discarded prior to leaving the site. Glove decontamination will follow the procedure outlined above.

## FIELD DECONTAMINATION OF SAMPLING EQUIPMENT

Decontamination of field equipment must be conducted in the following two situations:

- When the same piece of sampling equipment is to be used at more than one sampling point
- Prior to removing equipment from a site

Equipment which comes into direct contact with the sample must be thoroughly decontaminated between sampling points in order to prevent cross-contamination. A description of field decontamination procedures for different types of equipment is included in sections pertaining to their use in the field. The decontamination procedure generally consists of the following sequence: non-phosphate detergent scrub, tap water rinse, 20% methanol solution scrub, deionized water rinse, 10% nitric acid solution rinse and a final deionized water rinse. In general, equipment must be disassembled in order to be adequately decontaminated.

In addition, all equipment, instruments and containers must be cleaned of obvious contamination prior to leaving the site. This practice reduces the possibility of transporting contamination off-site. This practice also protects subsequent handlers of these items from inadvertent exposure to chemicals or contamination.

All water-resistant equipment shall at least be cleaned with a non-phosphate detergent scrub and a water rinse. Grossly contaminated items, such as bailers utilized for the sampling of pure product, may require full field decontamination prior to removal from the site. Any field equipment which requires more extensive, in-house decontamination after treatment in the field will be stored in plastic bags or bins for transport to the Fuss & O'Neill facility. A description of in-house decontamination methods is provided below.

Water-sensitive equipment and all field instruments must be wiped down with a moist paper towel to remove debris and contamination prior to leaving a site.

The outside surface of containers is often covered with contamination and/or corrosive preservatives. **Consequently, sealed sample containers must be rinsed with tap water in the field before the samples are placed in the cooler.** This measure will reduce the possibility that subsequent handlers will be exposed to contamination and/or hazardous preservatives.

When the sampling vehicle comes into contact with excessive mud or dirt as a result of field activities, it should be washed as soon as possible. Field personnel often have access to water which may be used to clean the truck before leaving the site. When no such water is available on-site, the truck will be taken to a car wash. Both the outside of the truck and the cab will be cleaned when necessary. When the underside of the truck is washed, special emphasis will be placed on cleaning the mud off the brakes.

## **IN-HOUSE DECONTAMINATION OF SAMPLING EQUIPMENT**

All sampling equipment will be fully disassembled and decontaminated between sampling events as described below. Decontamination procedures may vary slightly depending upon how the equipment will be used.

Equipment will be decontaminated in-house as follows: non-phosphate detergent scrub, tap water rinse, 20% methanol solution scrub, deionized water rinse, 10% hydrochloric acid solution rinse, and deionized water rinse. A double bottom stainless steel pan will be used during the acid rinse to collect all rinsate which must be neutralized to approximately pH 7 before disposal. Equipment will be allowed to air dry before it is wrapped in a protective aluminum foil covering.

## **INTRODUCTION**

Sediments provide essential habitat for many freshwater, estuarine, and marine organisms. Many toxic chemicals and waste materials accumulate in sediments and can pose a hazard to aquatic life through direct toxicity as well as to aquatic life, wildlife and human health through bioaccumulation in the food chain.

Sediment quality assessment typically includes analysis of anthropogenic contaminants, benthic community structure, physico-chemical characteristics, and direct measures of whole sediment and pore water toxicity. Accurate assessment of environmental hazards posed by sediment contamination depends in large part on the accuracy and representativeness of these samples.

The use of consistent sediment collection, manipulation, and storage methods will help provide high quality samples which will, in turn, allow for accurate data to be obtained.

## **TECHNICAL TERMS**

**Sediment:** Particulate material that usually lies below water or formulated particulate material that is intended to lie below water in a test

**Contaminated Sediment:** Sediment containing chemical substances at concentrations that pose a known or suspected threat to environmental or human health

**Pore (Interstitial) Water:** Water occupying space between sediment or soil particles

**Core Sample:** A sediment sample collected to obtain a vertical profile

**Grab:** Any device designed to “bite” or “scoop” into the bottom sediment of a lake, stream, estuary, ocean, and similar habitats to sample sediment. Grabs are samplers with jaws that are forced shut by weights, lever arms, springs, or cables. Scoops are grab samplers that scoop sediment with a rotating container.

**Ecotox Thresholds:** Benchmark values in ecological risk assessments defined as media-specific contaminant concentrations above which there is sufficient concern regarding adverse ecological effects to warrant further site investigation

**Data Quality Objectives:** Qualitative and quantitative statements that clarify the purpose of the monitoring study, define the most appropriate type of data to collect, and determine the most appropriate methods and conditions under which to collect them

**Measurement Quality Objectives:** Statements that describe the amount, type, and quality of data needed to address the overall project objectives

**Bioaccumulation:** The net accumulation of a substance by an organism as a result of uptake from

all environmental sources

Bioavailability: The degree to which a chemical is taken up by aquatic organisms

## **PRE-SAMPLING CHECKLIST**

### Documentation

- General paperwork (maps, sampling and Health & Safety plans, etc.)
- Logbook
- Pen and permanent marker
- Sediment sampling field data sheets
- Sample labels
- Chain-of-custody forms

### Personal Equipment

- Site-specific equipment required by Site Safety Plan
- Munsell Color Chart
- Grain size chart
- Disposable or rubber gloves
- 6' Ruler or small tape measure
- Water level measurement device
- Hand sprayers
- Paper towels
- Plastic garbage bags
- Bucket(s)
- Wooden stakes
- Survey flagging

### Sampling Equipment

- Approved sampling device
- Sediment Core Samplers
  - AMS Core Sampler with Core Catcher (Slide hammer)
  - Wildco® Hand Corer
- Grab Samplers
  - Wildco® Ponar (petite or regular)
  - Wildco® Ekman (standard, tall or large)
- Stainless steel mixing bowl
- Wooden tongue depressors, stainless steel spoon, or disposable plastic scoop
- Zip-lock poly bags

- Turkey baster or Siphon
- Coolers with ice packs or ice
- Water quality multi-meter: temperature, conductivity/specific conductance, dissolved oxygen, oxidation-reduction potential (ORP), and pH

#### Decontamination Equipment

- Non-phosphate detergent
- 10% Nitric acid solution
- 20% Methanol solution
- Deionized water
- Tap water

#### Site-Specific Equipment

- Keys to site facilities (when applicable)
- Sample containers for lab parameters

### **GENERAL SAMPLING INFORMATION**

Descriptive information of the site location and conditions will be documented before collecting sediment samples. Descriptive information includes:

- Date, time and weather conditions
- Explanation of the sample location
- GPS coordinates (NAD83, deg-min-sec)
- Site location photographs

Water quality data will be collected before collecting sediment samples. In waters less than two feet deep, water quality data will be collected just above (less than 6 inches) the sediment water interface. In waters greater than two feet deep, water quality data will be collected just below (6 to 12 inches) the water surface and just above the sediment/water interface. Water quality measurement will include temperature, conductivity/specific conductance, dissolved oxygen, oxidation-reduction potential (ORP), and pH.

Upon collection, sediment samples will be described in the field following ASTM D2488 methods. The color of the sediment will be determined by consulting a Munsell Color Chart. A description of grain size will be based on a particle size chart and in accordance with USDA particle size classification. Odors or sheens associated with the samples will be documented at the time of sampling.

## SHALLOW (WADABLE) STREAMS AND LAKES

Sediments from shallow (wadable) fluvial (stream and river) and lacustrine (lake and pond) settings will be collected as grab samples or hand cores. Samples will be collected in areas of deposition where sediments are finer-grained and water turbulence is minimal. A labeled survey stake will be placed at the sampling location after samples have been collected.

Decontaminated stainless steel tools, disposable wooden tongue depressors, or disposable plastic scoops will be used to homogenize and/or transfer the samples to appropriate sample containers.

The type of sample containers and sampling devices used will depend upon the physical characteristics of the sediment, the depth of overlying water, the depth of sediment to be analyzed, and the proposed lab analyses.. The following table shows the appropriate sampling device based on proposed analysis.

Proposed Analysis	Sample Depth		
	0 – 0.5 ft	0 – 1.0 ft	>1.0 ft
VOCs	C	C	C
SVOCs	C,G	C,G	C
Metals	C,G	C,G	C
Grain Size*	C,G	C,G	C
Total Organic Carbon*	C,G	C,G	C
pH*	C,G	C,G	C
SEM/AVS	C,G	C,G	C
Toxicity	C,G	C,G	C
Benthic Community	G	C,G	C

C = Core sampler; G = Grab or Dredge Sampler

\*These analyses should be conducted for all sediment samples

When two (2) or more sample locations are identified within a water body, sampling will proceed with the downstream samples first. The remainder of the samples will be collected as field personnel move upstream. If the sample collector must enter a stream prior to sampling, sediment samples will be collected upstream of the disturbance. The sampler will not enter a stagnant body of water until such time as a sediment sampling is to be acquired. Samples will be collected from lakes only after all surface water samples and field measurements in the vicinity have been taken.

When relatively undisturbed samples are required, a stainless steel hand corer will be utilized. Once the sample is collected, it will either be capped (such as with Teflon® for VOC analyses) or removed from the tool with a stainless steel spatula or wooden tongue depressor, and placed into an appropriate sample container.

Grab samples will be visually inspected to ensure the following acceptability conditions are satisfied:

- The sample is not over filled such that sediment is touching the top of the sampler
- Overlying water is present (indicating minimal leakage).
- The overlying water is clear and not excessively turbid
- The sediment/water interface is intact and relatively flat, with no sign of channeling or sample washout
- The desired depth has been achieved
- There is no evidence of sediment loss due to incomplete closure of the sampler, penetration at an angle, or tilting upon retrieval

Core samples will be visually inspected to ensure the following acceptability conditions are satisfied:

- The core sampler was not inserted at an angle or tilted upon retrieval
- The desired depth has been achieved
- There is no evidence of sediment loss

Overlying water should be removed prior to processing and storage by siphoning, not decanting. Samples will be collected in such a manner that samples potentially containing volatiles are handled or agitated to the least possible extent. Once samples potentially containing volatiles have been collected, samples will be collected for the remaining constituents.

Sediment samples collected will be greater than 30 percent solids to be considered a valid sample. Therefore, the collected sample will be left undisturbed for a short time to separate into liquid and solid fractions. The water fraction can then be siphoned from the sediment to increase the percentage of solids in the sample. The laboratory performing analyses will be instructed to take corrective measures in the event it finds the solids content to be less than 30 percent. Corrective measures may include not analyzing the sample or analyzing the sample and including percent solids information in the report narrative.

## **DEEP STREAMS AND LAKES**

When sampling sediments by hand (e.g. by core methods) is not convenient or possible, sediments will be collected with a dredge sampler.

The dredge will be lowered in a controlled manner to the sampling point by a length of nylon rope. At the point of contact, the rope will slacken and the sampler will be retrieved from the water. Dredge samples will be visually inspected to ensure the following acceptability conditions are satisfied:

- The sample is not over filled such that sediment is touching the top of the sampler
- Overlying water is present (indicating minimal leakage).
- The overlying water is clear and not excessively turbid
- The sediment/water interface is intact and relatively flat, with no sign of channeling or sample washout
- The desired depth has been achieved

- There is no evidence of sediment loss due to incomplete closure of the sampler, penetration at an angle, or tilting upon retrieval

A sample will be taken from the dredge with a decontaminated stainless steel tool or a wooden tongue depressor to be homogenized and/or placed into the appropriate sample container. Overlying water should be removed prior to processing and storage by siphoning, not decanting. Samples will be collected in such a manner that samples potentially containing volatiles are handled or agitated to the least possible extent. Once samples potentially containing volatiles have been collected, samples will be collected for the remaining constituents.

Sediment samples collected will be greater than 30 percent solids to be considered a valid sample. Therefore, the collected sample will be left undisturbed for a short time to separate into liquid and solid fractions. The water fraction can then be siphoned from the sediment to increase the percentage of solids in the sample. The laboratory performing analyses will be instructed to take corrective measures in the event it finds the solids content to be less than 30 percent. Corrective measures may include not analyzing the sample or analyzing the sample and including percent solids information in the report narrative.

This method of sediment sampling may cause significant bottom disturbance and, as a result, will be conducted only after water samples and field measurements in the vicinity have been conducted.

## **DECONTAMINATION**

All equipment utilized for sediment sampling will be decontaminated or discarded between sampling locations as appropriate. Obvious sediment build-up or contamination will be thoroughly removed with site water paying careful attention to any joints, cavities, or pivot points. Equipment to be decontaminated in the field should be suspended over a tub or bucket. Decontamination procedures are outlined in SOP 040000.

## **SAMPLING PROCEDURE – CORE SAMPLES**

Sediment core samples can be obtained using either a slide hammer, Wildco® sediment core sampler. The protocol for collecting sediment core samples using either core samplers is as follows:

- a. Affix a sample label to each container for that particular location immediately prior to sampling.
- b. If sampling in a stream or flowing body of water, approach the sampling location from downstream, being careful not to disturb any underlying sediment. When two or more sediment samples from the same stream are to be collected, the first sample collected should be at the furthest downstream location, with subsequent samples collected in a sequence progressing upstream.
- c. Lower the sampler through the water, then advance the core barrel to the desired depth.
- d. Remove the core barrel by gently rocking it from side to side, or reverse pounding it from the hole.
- e. For VOC samples, remove the slide hammer from the core barrel and slide the sample sleeves out. Place caps lined with non-stretchable Teflon® (per EPA) over both ends of the sample sleeves. Label the sleeves, and place in an iced cooler. The laboratory performing the analyses will extract a sediment sample from the sleeve.
- f. For grab samples, remove the slide hammer from the core barrel and slide the sample sleeves out. Transfer the sediment from the sample sleeves to the sample containers using a dedicated tongue depressor. For composite samples, transfer the sediment directly into a stainless steel bowl for homogenization. Materials such as surface vegetation (grass, leaves and plant stalks), roots, gravel, and artificial fill materials (e.g., asphalt, wood, brick, glass, ceramic, etc.) should not be included in the samples.
- g. All labeled sample containers will be placed in an iced cooler.

Sediment core samples can also be obtained using a Wildco® hand corer capable of collecting samples to 20-inch depths. The cored sample can be separated into discrete sediment samples, typically corresponding to the top (0-6"), middle (6-13"), and bottom (13"-20") layers.

## **SAMPLING PROCEDURE – GRAB OR DREDGE SAMPLES**

Surficial sediment samples can be obtained using either a simple scoop/dipper or a dredge sampler such as a Wildco® Ponar or Ekman dredge. The protocol for collecting surficial sediment samples using a scoop or dipper is as follows:

- a. Affix a sample label to each container for that particular location immediately prior to sampling.
- b. If sampling in a stream or flowing body of water, approach the sampling location from downstream, being careful not to disturb any underlying sediment. When two or more sediment samples from the same stream are to be collected, the first sample collected should be at the furthest downstream location, with subsequent samples collected in a sequence progressing upstream.
- c. Push the scoop or dipper firmly downward into the sediment, then lift upward. Quickly lift the sampler out of the water.
- d. Transfer the sediment from the scoop/dipper to the sample containers using a dedicated tongue depressor. For composite samples, transfer the sediment directly into a stainless steel bowl for homogenization. Materials such as surface vegetation (grass, leaves and plant stalks), roots, gravel, and artificial fill materials (e.g., asphalt, wood, brick, glass, ceramic, etc.) should not be included in the samples.
- e. All labeled sample containers will be placed in an iced cooler.

A dredge sampler, such as a Wildco® Ponar or Ekman dredge, can be used to collect gross surficial, or bulk sediment samples. Typically, a dredge sampler is used to obtain a sample from the first four inches of surficial sediments, those nearest the sediment-water interface. Collection of surficial sediment samples is typically performed for analysis of bulk sediment parameters such as benthic invertebrates, acid volatile sulfides and simultaneously extracted metals (AVS/SEM), and sediment toxicity.

The general procedure for collecting surficial sediment samples using a dredge sampler is as follows:

- a. Affix a sample label to each container for that particular location immediately prior to sampling.
- b. If sampling in a stream or flowing body of water, approach the sampling location from downstream, being careful not to disturb any underlying sediment. When two or more sediment samples from the same stream are to be collected, the first sample collected should be at the furthest downstream location, with subsequent samples collected in a sequence progressing upstream.

- c. For the Ekman dredge, Hold the sampling pole so the open sampler jaws are positioned several inches above the surface of the sediment, then firmly thrust the sampler downward. Depress the button at the top of the sampling pole to release the spring loaded jaws. For the Ponar dredge, lower the sampler into the water. Once the sampler is approximately one (1) foot from the bottom, allow it to free-fall.
- d. Slowly retrieve the samples and inspect the jaws to ensure proper closer. Inspect the sample within the dredge to ensure no sediments were lost during retrieval.
- e. Transfer the sediment from the dredge sampler to the sample containers using a dedicated tongue depressor. For composite samples, transfer the sediment directly into a stainless steel bowl for homogenization. Materials such as surface vegetation (grass, leaves and plant stalks), roots, gravel, and artificial fill materials (e.g., asphalt, wood, brick, glass, ceramic, etc.) should not be included in the samples.
- f. All labeled sample containers will be placed in an iced cooler.

# Sediment Sampling Field Data

Client/Project Name: _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location: _____		
Date: _____ Weather: _____ Sampler(s): _____		

Sample #:	Container	Quantity	Preservative
Site ID:			
GPS Coord. _____ N _____ W   Photo #: _____			
Water Quality Parameters _____ Surface _____ Bottom _____			
Temperature (C) _____			
Conductivity (uS/cm) _____			
pH / ORP (mV) _____ / _____			
DO (mg/L / %) _____ / _____			
Sampling Device: Core Sampler – Type: _____			
Dredge – Type: Ponar / Ekman / Other: _____			
Field decon: Yes / No / Dedicated			
Type of Sample: Grab / Composite / Other _____			
Sample Depth: _____ Feet			

## INTRODUCTION

Soil samples will be described in the field following ASTM D2488-69 methods. The soil color will be determined by consulting a Munsell Color Chart, while the description of grain size will be based on a particle size chart. This descriptive information, along with a thorough explanation of the sample location and sampling conditions, will be recorded on a field data sheet. Examples of soil sampling field data sheets and boring log are provided at the end of this document.

## SAMPLING EQUIPMENT FOR SOIL SAMPLING

The following list includes the equipment which may be necessary to the sampling of soils:

### 1. Documentation

- General paperwork (maps, sampling and H&S plans, etc.)
- Logbook
- Pen and permanent marker
- Soil sampling field data sheets
- Sample labels
- Chain-of-custody forms

### 2. Personal Equipment

- Equipment required by Site Safety Plan
- Munsell Color Chart
- Grain size chart
- Disposable or rubber gloves
- 6' Ruler or small tape measure
- Water level measurement device
- Hand sprayers
- Paper towels
- Plastic garbage bags
- Bucket
- Wooden stakes

### 3. Sampling Equipment

- Excavation tools (shovels, post-hole diggers, pick, pry-bar and other tools)
- Approved sampling device (stainless steel hand auger, trier, aluminum coring tube, syringe sampler, etc.)
- Stainless steel mixing bowl
- Wooden tongue depressors or stainless steel spoon
- Zip-lock poly bags
- Organic vapor detector
- Coolers with ice packs or ice

**4. Decontamination Equipment**

- Non-phosphate detergent
- Nitric acid solution
- Methanol solution
- Deionized water
- Tap water

**5. Site-Specific Equipment**

- Keys to site facilities (when applicable)
- Sample containers for lab parameters

**6. SS Soil Auger - Available Parts List**

<u>Qty</u>	<u>Description</u>
3	Soil Auger Tool Kits
in each:	<ul style="list-style-type: none"><li>- 1-1/4" or 2-1/2" auger w/bail</li><li>- 7/8" open end combination wrench</li><li>- 15/16" open end combination wrench</li><li>- strap wrench</li><li>- 3-1/4" or 2-1/2" auger brush</li><li>- sampling tube extraction tool</li><li>- container of vegetable lubricant</li></ul>
3	Extension handles
4	6' extensions
1	3' extension

**SURFACE SOIL SAMPLING**

Surface soil sampling will be conducted with a stainless steel hand auger, trier, aluminum coring tube, syringe sampler, scoop, or another approved sampling device. The hand auger and scoops can be used when undisturbed samples are not required. See SOP 080100 for scoop sampling techniques. See SOP 080200 for hand auger sampling techniques.

Both the trier and the coring tube are used to collect undisturbed soil samples.

Samples will be transferred from the sampling device to glass jars with Teflon-lined lids by means of dedicated wooden tongue depressors or decontaminated stainless steel spoons. Those samples to be analyzed for volatile and semi-volatile compounds will be collected in 40 ml VOA vials with Teflon septa. Ice or ice packs will be used to preserve lab samples at 4 degrees Celsius immediately after collection. Soils to be screened for organic compounds in the field

will be separated from the rest of the sample prior to the screening procedure so that the portion submitted to the lab will remain intact. Methods of soil screening are discussed in a later section of the text.

Equipment which comes into contact with the sample will be thoroughly decontaminated or replaced between soil samples as outlined in SOP 040000.

## **SUB-SURFACE SOIL SAMPLING**

A hand auger, soil core device, hand Geoprobe®, or direct push drill rig can be used to collect sub-surface soil samples (see SOP 080200). During monitoring well and test boring operations, sub-surface soil samples will be collected with a 24-inch split-tube sampler (hollow-stem auger rig) described in SOP 080300, or a 2-4 foot Geoprobe® sampler (direct-push rig) described in SOP 111000.

Sampling equipment will be cleaned between samples as outlined in SOP 040000. When necessary, drilling equipment will be steam-cleaned prior to the start of each boring.

## **METHODS OF SOIL SCREENING**

The following are methods by which screening for volatile organic compounds in soil samples may be carried out: the split-tube method, headspace screening, the bag method, and through on-site gas chromatography. A detailed description of the collection of soil vapor for on-site gas chromatography is discussed in "Soil Gas Testing" later in the text.

### **Split-Tube Method**

Volatile organic compounds may be detected by running the probe of a photoionization detector (PID) along the length of a split sample while it is in the split tube. This technique involves gently opening and agitating the sample in one or two places with a wooden tongue depressor and inserting the probe of the PID into this space before a reading is taken. Care should be taken not to touch the probe tip to the soil.

Between readings, the portable screening instrument will be allowed to pump ambient air until the instrument reading reaches background levels.

### **Headspace Screening**

When headspace screening is conducted in conjunction with laboratory analysis, the sample for the laboratory will be collected first. The remaining soil will be placed in a jar, or more preferably, a resealable polyethylene bag. Any container utilized will be measured for organic vapors prior to use. The soil will be agitated within the sealed container to disaggregate the soil and liberate any volatile organic compounds into the headspace. The probe of the screening instrument will then be inserted into the headspace of the container and a reading will be obtained. Several readings of the vapors in the headspace will be measured in an attempt to duplicate the highest value. Between readings, the portable screening instrument will be

allowed to pump ambient air until the reading reaches background levels.

### **The Bag Method**

A third technique of soil screening utilizing a portable PID or FID is the bag method (Robbins, 1989). Due to the destructive nature of this method, a portion of each soil sample will be separated out for laboratory analysis prior to soil screening by this method.

### **Apparatus**

A resealable polyethylene bag will be fitted with a three way valve to allow access to the headspace of the bag while it is sealed from the ambient air. One port of the valve will access the bag itself, while a second will permit the bag to be inflated by a hand pump once it has been sealed. The third valve port will be connected to the probe of the PID or FID by means of a length of inert tubing. Movement of air in and out of the sealed bag may consequently be controlled by the position of the valve. Figure 080430 depicts the polyethylene bag sampling system.

### **Method Blanks**

Prior to a screening event, the relative accuracy of the bag method system will be determined by conducting a series of blanks.

First, ambient air within an empty polyethylene bag will be screened to determine the impact of background conditions upon sample integrity. Background conditions in this situation would include both the ambient air and the materials out of which the apparatus is constructed. The polyethylene bag will be sealed and inflated by means of a hand pump. After four minutes of agitation, the three-way valve will be opened to allow any vapor present to flow into the probe of the instrument. The reading obtained will represent the background concentration of organic vapors for this particular sampling event. If this concentration is unusually high, the sample collector will determine the source of the contamination and eliminate it, if possible, prior to continuing soil screening.

A second blank will be conducted to determine whether the deionized water utilized to liquidate the sample affects sample integrity. The polyethylene bag containing deionized water will be sealed and agitated for four minutes. The three-way valve will subsequently be opened to allow the vapor in the headspace to flow into the probe of the instrument. The concentration of any organic vapor emanating from the deionized water will then be measured.

A soil standard blank will be prepared prior to each sampling event in order to construct a calibration curve for the contaminant of concern. This curve is necessary to properly relate the results of headspace screening to the actual concentration of the contaminant in the soil.

A soil sample will be collected from an unaffected area of the site. After sealing the uncontaminated soil and water mix and inflating the bag as described above, a measured amount of tetrachloroethylene, m-xylene or the contaminant of concern will be injected by

microliter syringe into the bag through a fitting with a Teflon septum.

The soil/water mix will be agitated for four minutes and an organic vapor concentration will subsequently be measured. This procedure will be repeated utilizing a series of concentrations within a calibration curve will then be constructed. Once this relationship is derived under experimental conditions, it can be used to calculate the concentrations of unknown volatile organics relative to the calibrated standards.

### **Gas Chromatography**

Screening of soil samples may be conducted by an on-site gas chromatograph. Samples for this analysis will be collected in Teflon-lined glass jars, such as VOA vials.

# Soil Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

Sample Location Info (sketch map including location of stockpile)

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Geoprobe / Shovel / Split Spoon / Trowel / Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____  Sample Depth: _____ PetroFLAG / OVM _____			

Comments:





## INTRODUCTION

Soil samples will be described in the field following ASTM D2488-69 methods. The soil color will be determined by consulting a Munsell Color Chart, while the description of grain size will be based on a particle size chart. This descriptive information, along with a thorough explanation of the sample location and sampling conditions, will be recorded on a field data sheet. Examples of soil sampling field data sheets and boring log are provided at the end of this document.

## SAMPLING EQUIPMENT FOR SOIL SAMPLING

The following list includes the equipment which may be necessary to the sampling of soils:

### 1. Documentation

- General paperwork (maps, sampling and H&S plans, etc.)
- Logbook
- Pen and permanent marker
- Soil sampling field data sheets
- Sample labels
- Chain-of-custody forms

### 2. Personal Equipment

- Equipment required by Site Safety Plan
- Munsell Color Chart
- Grain size chart
- Disposable or rubber gloves
- 6' Ruler or small tape measure
- Water level measurement device
- Hand sprayers
- Paper towels
- Plastic garbage bags
- Bucket
- Wooden stakes

### 3. Sampling Equipment

- Excavation tools (shovels, post-hole diggers, pick, pry-bar and other tools)
- Approved sampling device (stainless steel hand auger, trier, aluminum coring tube, syringe sampler, etc.)
- Stainless steel mixing bowl
- Wooden tongue depressors or stainless steel spoon
- Zip-lock poly bags
- Organic vapor detector
- Coolers with ice packs or ice

**4. Decontamination Equipment**

- Non-phosphate detergent
- Nitric acid solution
- Methanol solution
- Deionized water
- Tap water

**5. Site-Specific Equipment**

- Keys to site facilities (when applicable)
- Sample containers for lab parameters

**6. SS Soil Auger - Available Parts List**

<u>Qty</u>	<u>Description</u>
3	Soil Auger Tool Kits
in each:	<ul style="list-style-type: none"><li>- 1-1/4" or 2-1/2" auger w/bail</li><li>- 7/8" open end combination wrench</li><li>- 15/16" open end combination wrench</li><li>- strap wrench</li><li>- 3-1/4" or 2-1/2" auger brush</li><li>- sampling tube extraction tool</li><li>- container of vegetable lubricant</li></ul>
3	Extension handles
4	6' extensions
1	3' extension

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## **METHODS OF SOIL SCREENING**

The following are methods by which screening for volatile organic compounds in soil samples may be carried out: the split-tube method, headspace screening, the bag method, and through on-site gas chromatography. A detailed description of the collection of soil vapor for on-site gas chromatography is discussed in "Soil Gas Testing" later in the text.

### **Split-Tube Method**

Volatile organic compounds may be detected by running the probe of a photoionization detector (PID) along the length of a split sample while it is in the split tube. This technique involves gently opening and agitating the sample in one or two places with a wooden tongue depressor and inserting the probe of the PID into this space before a reading is taken. Care should be taken not to touch the probe tip to the soil.

Between readings, the portable screening instrument will be allowed to pump ambient air until the instrument reading reaches background levels.

### **Headspace Screening**

When headspace screening is conducted in conjunction with laboratory analysis, the sample for the laboratory will be collected first. The remaining soil will be placed in a jar, or more preferably, a resealable polyethylene bag. Any container utilized will be measured for organic vapors prior to use. The soil will be agitated within the sealed container to disaggregate the soil and liberate any volatile organic compounds into the headspace. The probe of the screening instrument will then be inserted into the headspace of the container and a reading will be obtained. Several readings of the vapors in the headspace will be measured in an attempt to duplicate the highest value. Between readings, the portable screening instrument will be

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### **Apparatus**

A resealable polyethylene bag will be fitted with a three way valve to allow access to the headspace of the bag while it is sealed from the ambient air. One port of the valve will access the bag itself, while a second will permit the bag to be inflated by a hand pump once it has been sealed. The third valve port will be connected to the probe of the PID or FID by means of a length of inert tubing. Movement of air in and out of the sealed bag may consequently be controlled by the position of the valve. Figure 080430 depicts the polyethylene bag sampling system.

### **Method Blanks**

Prior to a screening event, the relative accuracy of the bag method system will be determined by conducting a series of blanks.

First, ambient air within an empty polyethylene bag will be screened to determine the impact of background conditions upon sample integrity. Background conditions in this situation would include both the ambient air and the materials out of which the apparatus is constructed. The polyethylene bag will be sealed and inflated by means of a hand pump. After four minutes of agitation, the three-way valve will be opened to allow any vapor present to flow into the probe of the instrument. The reading obtained will represent the background concentration of organic vapors for this particular sampling event. If this concentration is unusually high, the sample collector will determine the source of the contamination and eliminate it, if possible, prior to continuing soil screening.

A second blank will be conducted to determine whether the deionized water utilized to liquidate the sample affects sample integrity. The polyethylene bag containing deionized water will be sealed and agitated for four minutes. The three-way valve will subsequently be opened to allow the vapor in the headspace to flow into the probe of the instrument. The concentration of any organic vapor emanating from the deionized water will then be measured.

A soil standard blank will be prepared prior to each sampling event in order to construct a calibration curve for the contaminant of concern. This curve is necessary to properly relate the results of headspace screening to the actual concentration of the contaminant in the soil.

A soil sample will be collected from an unaffected area of the site. After sealing the uncontaminated soil and water mix and inflating the bag as described above, a measured amount of tetrachloroethylene, m-xylene or the contaminant of concern will be injected by

microliter syringe into the bag through a fitting with a Teflon septum.

The soil/water mix will be agitated for four minutes and an organic vapor concentration will subsequently be measured. This procedure will be repeated utilizing a series of concentrations within a calibration curve will then be constructed. Once this relationship is derived under experimental conditions, it can be used to calculate the concentrations of unknown volatile organics relative to the calibrated standards.

### **Gas Chromatography**

Screening of soil samples may be conducted by an on-site gas chromatograph. Samples for this analysis will be collected in Teflon-lined glass jars, such as VOA vials.

# Soil Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

Sample Location Info (sketch map including location of stockpile)

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Geoprobe / Shovel / Split Spoon / Trowel / Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____  Sample Depth: _____ PetroFLAG / OVM _____			

Comments:





A hand auger is a common sampling tool for shallow soils. The tool is composed of a bucket auger which comes in various shapes and sizes, a shaft, and a T-bar handle. Extensions of the shaft are available to allow sampling at deeper intervals. Typically, this tool is only effective to 5 feet since the sample hole usually begins to collapse at this depth.

- a. Begin collection of the sample by applying a downward pressure while rotating the auger clockwise. When the auger is full of solid material it should be removed from the hole, and the material transferred into a stainless steel bowl using a stainless steel spoon. Continue sampling in this manner until the bottom of the sampling interval is reached.
- b. At desired collection depths, collect the sample for VOC analysis immediately after removal from the auger and transfer to the appropriate glass container to minimize volatile loss.
- c. Composite the remaining material in the sampling bowl by using the stainless steel spoon to break apart any large chunks of material, then mix and stir the material enough to thoroughly homogenize the sample.
- d. Transfer the material into a labeled sample jar using the stainless steel spoon.
- e. Place all labeled sample containers in an iced cooler.
- f. Record observations and soil type onto a Field Data Sheet.
- g. Measure the total volatile concentration inside a plastic zip closure bag using an Organic Vapor Monitor. Record readings on the Field Data Sheet.



## APPENDIX D

### FUSS & O'NEILL, INC. STANDARD OPERATING PROCEDURES FOR FIELD EQUIPMENT OPERATION

## **PRINCIPLE OF OPERATION**

The YSI-63 Series is a field portable pH, conductivity, salinity and temperature water analyzer.

The pH portion has a combination electrode consisting of a proton selective glass reservoir filled with a buffer at a pH of approximately 7 and an Ag/AgCl reference electrode that utilizes gelled electrolyte. A silver wire coated with AgCl is immersed in the buffer solution. Protons (H<sup>+</sup> ions) on both sides of the glass (media and buffer reservoir) selectively interact with the glass, setting up a potential gradient across the glass membrane. Since the hydrogen ion concentration in the internal buffer solution is invariant, this potential difference, determined relative to the Ag/AgCl reference electrode, is proportional to the pH of the media.

The conductivity cell utilizes four pure nickel electrodes for the measurement of solution conductance. Two of the electrodes are current driven and two are used to measure the voltage drop. Conductivity expressed in micromhos/cm is the measure of the electric conductance that would be exhibited by a sample if it were measured between opposite faces of a 1 centimeter cube.

## **INSTRUMENT MAINTENANCE**

The following calibration and maintenance procedures are conducted at Fuss & O'Neill before instruments are released for use.

- Calibration of the instrument will be performed as described in the calibration procedure.
- The pH glass electrode requires maintenance only when obviously coated with sediment or biological growth. The glass can be cleaned by carefully removing the guard and wiping with a soft cloth soaked with soapy water and rinsed with clean water, followed by a routine decontamination process. The glass electrode is very delicate, be sure to wipe the electrode gently and do not put the probe down until the guard is replaced. Then a moistened cotton swab can remove any material that may be blocking the reference electrode junction of the sensor.
- The temperature and specific conductivity portion of the probe have openings that allow fluid access to the conductivity electrodes and must be cleaned regularly. This is accomplished by inserting a small cleaning brush into each hole several times using a mild detergent with the brush if necessary. After cleaning and decontamination, the probe will be checked with a calibration standard.

## **INSTRUMENT CALIBRATION**

A complete calibration check will be performed before, after and once at a mid point of each sampling day at Fuss & O'Neill. Checks will be carried out in the field if there is a possibility that any memory has been cleared or readings seem anomalous.

1. pH Calibration using the 4, 7 and 10 pH buffer solutions

The pH calibration is accomplished by pressing MODE until pH is displayed. Using a thermometer take the temperature of the standard solution and verify that the temperature displayed on the meter is the as the temperature on the thermometer. Rinse the probe with de-ionized water and wipe dry or do a final rinse with pH 7 buffer solution. Place the probe in the pH 7 buffer solution. Simultaneously press the up down arrow keys to enter the calibration menu. Press ENTER. The pH of the buffer solution will be displayed. When the decimal point stops flashing press ENTER again. Remove and rinse the probe with de-ionized water and wipe dry. Place the probe in either a pH 4 or 10 solution. Press ENTER and the pH of the solution will be displayed and the decimal point will flash. When the decimal point stops flashing press and hold ENTER until the display reads SAVE. At the end of the calibration press MODE to begin pH measurements. Remove the probe and rinse with DI water and wipe dry. Return the probe to the last pH solution, press read and when the decimal point stops flashing record the pH. The pH should be within +/- 0.15 pH units. Re-calibrate if outside of this range.

2. Specific Conductance Calibration

Turn the instrument on and allow it to complete its self test. Press MODE until conductivity is displayed. The proper display shows degrees C flashing. This is the temperature compensated mode. Insert the probe into the KCl calibration solution deep enough to cover both of the conductivity ports. Allow at least 60 seconds for the temperature reading to become stable. Move the probe from side to side to dislodge any air bubbles from the electrodes. Press the up arrow and down arrow simultaneously until the CAL symbol is displayed. Use the up arrow and down arrow keys to adjust the reading on the display until it matches the concentration of the solution being used for calibration. Once the display reads the exact value of the calibration solution being used press the ENTER key. The word SAVE will momentarily appear in the display indicating the calibration has been accepted. Remove the probe, rinse with DI water and dry. Place the probe back into the last solution and determine the Specific conductance. A correctly calibrated meter will read within +/- 25 micromhos/cm of the true value. If the reading falls outside of this range it will need to be re-calibrated.

## **PRINCIPLE OF OPERATION**

The YSI Model 85 is a portable, battery-powered, micro-processor based, digital meter, designed to measure salinity, conductivity, and temperature. The probe for the Model 85 is both a rugged plastic conductivity cell and a precision YSI thermistor temperature sensor with a dissolved oxygen probe, combined in a single unit.

## **YSI 85 PARAMETER SPECIFICATIONS**

The YSI Model 85 SCT + DO has the following specifications:

Ranges	Conductance: User selected or Autoranging 0 to 499.9 $\mu\text{S}/\text{cm}$ 0 to 4999 $\mu\text{S}/\text{cm}$ 0 to 49.99 $\text{mS}/\text{cm}$ 0 to 200.0 $\text{mS}/\text{cm}$
	Salinity: 0 to 80 PPT Temperature: $-5^{\circ}\text{C}$ to $+65^{\circ}\text{C}$ Dissolved Oxygen: Autoranging 0 to 200% Air Saturation 0 to 20 $\text{mg}/\text{L}$
Readability (Resolution):	Conductance: 0.1 $\mu\text{S}/\text{cm}$ 1.0 $\mu\text{S}/\text{cm}$ .01 $\text{mS}/\text{cm}$ 0.1 $\text{mS}/\text{cm}$
	Salinity: 0.1 ppt Temperature: $0.1^{\circ}\text{C}$ Dissolved Oxygen: 0.1% Air Saturation 0.01 $\text{mg}/\text{L}$
Accuracy:	Conductance: $\pm 0.5\%$ fs Salinity: $\pm 2\%$ or $\pm 0.1$ ppt Temperature: $\pm 0.1^{\circ}\text{C}$ ( $\pm 1$ lsd) Dissolved Oxygen: $\pm 2\%$ Air Saturation $\pm 0.3$ $\text{mg}/\text{L}$



2. With instrument on, press the mode button until dissolved oxygen is displayed in mg/L or % and let reading stabilize. Fifteen minutes is usually required for stabilization of the temperature and dissolved oxygen readings.
3. With two fingers, press and release both the UP ARROW and DOWN ARROW buttons at the same time.
4. The display will prompt user to enter the local altitude in hundreds of feet ( 1 = 100ft). Press the arrow keys to increase or decrease the altitude. When the proper altitude appears on the display, press the ENTER button once.
5. CAL should now be at the lower left of the display, the calibration value should be displayed in the lower right of the display and the current % reading should be on the main display. When the % reading is stable press the ENTER button. The display should read SAVE and return to the normal operation mode.

#### **YSI 85 METER DOCUMENTATION**

Daily calibration will be documented. The unit number, date, and calibration information will be recorded in a calibration log ([Appendix S](#)). The following information will be recorded on a standard field data sheet as the specific conductance data is obtained:

- Project name and number.
- Operator's name.
- Date and time of specific parameter measurement.
- Meter ID number.
- Sample code
- Conductivity reading
- Dissolved oxygen reading
- Temperature reading

## **PRINCIPLE OF OPERATION**

The YSI-600 Series Water Analyzer consists of a sonde, a monitoring device that is placed in water to gather water quality data that is measured by individual probes, the probes and a 610-D. The probes are fastened at the base of the sonde and take measurements of oxidation/reduction potential, dissolved oxygen, temperature, and temperature-compensated pH and specific conductance. Readings are displayed on the 610-D display unit in units of mV, mg/l, °C, S.U., and  $\mu\text{Mhos/cm}$  ( $\mu\text{S/cm}$ ) respectively.

## **INSTRUMENT MAINTENANCE**

The following calibration and maintenance procedures are conducted at Fuss & O'Neill before instruments are released for use.

- Calibration of the instrument will be performed as described in the calibration procedure.
- The Sonde must be thoroughly dried before removing or replacing a probe or probe port plug. When a probe or port is replaced, the connector port inside the Sonde is to be examined for moisture. If any moisture is found, the connector is to be completely dried. If any corrosion is found the unit will be replaced with another comparable unit. The cable connector port at the top of the Sonde should be covered at all times. Any moisture found in this area will be completely dried before continuing.
- The pH glass electrode and ORP platinum button require maintenance only when obviously coated with sediment or biological growth. The glass and platinum can be cleaned by carefully removing the guard and wiping with a soft cloth soaked with soapy water and rinsed with clean water, followed by a routine decontamination process. The glass electrode is very delicate, be sure to wipe the electrode gently and do not put the probe down until the guard is replaced. Then a moistened cotton swab can remove any material that may be blocking the reference electrode junction of the sensor.
- The temperature and specific conductivity probe have openings that allow fluid access to the conductivity electrodes and must be cleaned regularly. This is accomplished by inserting a small cleaning brush into each hole several times using a mild detergent with the brush if necessary. After cleaning and decontamination, the probe will be checked with a calibration standard.
- Maintenance of the DO sensor is required when the instrument does not calibrate properly or when the membrane is dirty or damaged. Following cleaning and decontamination, recalibration is performed. The membrane and electrolyte is changed on a regular schedule to maximize the accuracy and life of the sensor.

## **CALIBRATION PROCEDURES**

A complete calibration check will be performed before and after each sampling day at Fuss & O'Neill. Checks will be carried out in the field if there is a possibility that any memory has been cleared or readings seem anomalous.

When not in use, the YSI-600's sensors are protected with a screw on storage cup filled with tap water. This storage cap is utilized during all calibration procedures. A description of each calibration procedure is below.

### 1. pH Calibration

The pH calibration is accomplished by pressing ESC and scrolling through the menu to CAL MODE, and pressing enter. The pH is then entered from the calibration menu. A three point calibration is chosen, the display then prompts the user to enter pH buffer that will be checked first, typically the 7.00 buffer. Buffer 7.00 is poured into the storage cup and placed under the probe, the user then presses enter, the unit will then prompt user to press enter again and to enter the next buffer for calibration. The sensor and storage cup are then rinsed with deionized water and the same procedure is followed using pH 4 or pH 10 buffers. Following these steps, calibration checks of all buffers will be conducted.

### 2. Specific Conductance Calibration

The specific conductance calibration is accomplished by first rinsing the sensor and storage cup with deionized water several times, then filling the storage cup with KCl solution of a known standard molar concentration and specific conductance. When in CAL MODE the specific conductance, is chosen from the calibration menu and the known specific conductance value is entered in the display unit and the calibration is then saved. Following these steps, the storage cup is rinsed again with deionized water and then refilled with the standard KCl solution to recheck the calibration.

### 3. Dissolved Oxygen Calibration

The dissolved oxygen calibration is accomplished by filling the storage cup with tap water to a level just below the membrane and then capping the storage cap. When in the CAL MODE the Barometer reading for the day is entered. These values are used to determine the correct calibration value to be entered into the display unit. Once the correct pressure has been entered, press the ENTER key to save the value.

### 4. ORP Calibration

The ORP calibration is accomplished by filling the storage cup with Zobel solution to a level to cover the ORP probe, about one half inch. While in CAL MODE scroll to ORP and press enter. The unit will then prompt for a value to be entered (Zobel has a value of 225), after entering the value, press enter to calibrate.

When the values for all the parameters have all been entered for calibration, press ESC until the MAIN MENU is displayed. Scroll back to run and press enter. Check the calibration for all parameters before continuing.

## **YSI 600 METER DOCUMENTATION**

Daily calibration will be documented. The unit #, date, and calibration information will be recorded in a calibration log. The following information will be recorded on a standard field data sheet as the specific conductance data is obtained:

- Project name and number.
- Operator's name.
- Date and time of specific parameter measurement.
- Meter ID number.
- Sample code
- Conductivity reading
- Dissolved oxygen reading
- Oxidation/Reduction potential reading
- pH reading
- Temperature reading

## **YSI 600 COMPONENT SPECIFICATIONS**

### **600XL Sonde**

#### Operating Environment

Medium:	Fresh, sea, or polluted water
Temperature:	-5 to +45 °C
Depth:	0 to 200 feet (61 meters)

Storage Temperature:	-40 to +60 °C for sonde and all sensors except pH and ORP
	-20 to +60 °C for pH and ORP sensors

Material:	Polyurethane, PVC, Stainless Steel
Maximum Diameter:	1.6 inches (4.06 cm)
Maximum Length:	20.75 inches (52.7 cm)
Maximum Weight:	4.9 pounds (2.22 kg)
Internal logging memory size:	384 KB ( 150,000 individual parameter readings )
Computer Interface:	RS-232C, SDI-12
Power:	External 12 VDC ( 8 to 13.8 VDC )

### **Conductivity Probe**

Sensor type	4 electrode cell with autoranging
Range	0 to 100 mS/cm
Accuracy	+/- 0.5 % of reading + 0.001 mS/cm
Resolution	0.001 mS/cm to 0.1 mS/cm ( range dependent )
Depth	200 meters

### **Temperature**

Sensor Type	Thermistor
Range	-5 to 45 °C
Accuracy	+/- 0.15 °C
Resolution	0.01 °C
Depth	200 meters

### **Dissolved Oxygen, % saturation**

Sensor Type	Rapid Pulse-Clark type, polarographic
Range	0 to 500 % air saturation
Accuracy	0-200 % air saturation, +/- 2 % of the reading or 2% air saturation, whichever is greater 200-500 % air saturation, +/- 6 % of reading
Resolution	0.1 % air saturation
Depth	200 meters

**Dissolved Oxygen, mg/L** (calculated from % air saturation, temperature and salinity)

Sensor Type	Rapid Pulse-Clark type, polarographic
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Range 0 to 50 mg/L  
Accuracy 0 to 20 mg/L, +/- 2 % of the reading or 0.2 mg/L,  
whichever is greater 20 to 50 mg/L, +/- 6 % of the reading

Resolution 0.01 mg/L  
Depth 200 meters

**Salinity**

Sensor Type Calculated from conductivity and temperature  
Range 0 to 70 ppt  
Accuracy +/- 1.0 % of reading or 0.1 ppt, whichever is greater  
Resolution 0.01 ppt

**pH**

Sensor Type Glass combination electrode  
Range 0 to 14 units  
Accuracy +/- 0.2 units  
Resolution 0.01 units  
Depth 200 meters

**ORP**

Sensor Type Platinum button  
Range -999 to +999 mV  
Accuracy +/- 20 mV  
Resolution 0.1 mV  
Depth 200 meters



## APPENDIX E

PREMIER ANALYTICAL LABORATORY

STANDARD OPERATING PROCEDURES FOR  
ANALYTICAL METHODS



## Organic Extraction and Sample Preparation Method 3500B

Prepared by:   
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Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 2, December 1996, Method 3500B.

### I. Applicability:

Analyte: All extractions  
Matrix: Water, wastewater, soil, sludge, waste extracts  
Regulation: RCRA

### II. Scope And Application:

Method 3500 provides general guidance on the selection of methods used in the quantitative extraction (or dilution) of samples for analysis by one of the semivolatile or nonvolatile determinative methods. Cleanup and/or analysis of the resultant extracts are described in Chapter Two as well as in Method 3600 (Cleanup) and Method 8000 (Analysis).

Method 3580 may be used for the solvent dilution of non-aqueous semivolatile and nonvolatile organic samples prior to cleanup and/or analysis.

Methods 3545, 3560, and 3561 are techniques that utilize pressurized solvent extraction to reduce the amount of solvent needed to extract target analytes and reduce the extraction time when compared to more traditional techniques such as Soxhlet extraction.

Prior to employing this method, analysts are advised to consult the disclaimer statement at the front of the manual and the information in Chapter Two of SW 846 for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in this procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to meet the data quality objectives or needs for the intended use of the data.



### III. Summary of Method:

A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method choices for aqueous samples include liquid-liquid extraction by separatory funnel or by continuous extractor and solid-phase extraction (SPE). Method choices for soil/sediment and solid waste samples include standard solvent extraction methods utilizing either Soxhlet, automated Soxhlet, or ultrasonic extraction. Solids may also be extracted using pressurized extraction techniques such as supercritical fluid extraction or heated pressurized fluid extraction.

The resultant extract is dried and concentrated in a Kuderna-Danish (K-D) apparatus. Other concentration devices or techniques may be used in place of the Kuderna-Danish concentrator if the quality control requirements of the determinative methods are met

(SW 846 Method 8000, Sec. 8.0).

NOTE: Solvent recovery apparatus is recommended for use in methods that require the use of Kuderna-Danish evaporative concentrators. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program.

### IV. Interferences:

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method for specific guidance on quality control procedures.

Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary. Refer to Method 3600 for guidance on cleanup procedures.

Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

### V. Apparatus and Materials:

Refer to the specific method of interest for a description of the apparatus and materials needed.

Solvent recovery apparatus is recommended for use in methods that require the use of Kuderna-Danish evaporative concentrators. Incorporation of this apparatus may be required by State or local municipality regulations that govern air emissions of volatile organics. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction



program. Solvent recovery is a means to conform with waste minimization and pollution prevention initiatives.

## VI. Reagents:

Refer to the specific method of interest for a description of the solvents needed.

Organic free reagent water. All references to water in this method refer to organic-free reagent water.

Stock standards for spiking solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. The stock solutions used for the calibration standards are acceptable (dilutions must be made in a water miscible solvent) except for the quality control check sample stock concentrate which must be prepared independently to serve as a check on the accuracy of the calibration solution.

Prepare stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in a water miscible solvent (i.e., methanol, acetone, 2-propanol, etc.) and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially-prepared stock standard solutions can be used at any concentration if they are certified by the manufacturer or by an independent source.

Stock standard solutions should be stored in polytetrafluoroethylene (PTFE)-sealed containers at 4°C or below. The solutions should be checked frequently for stability. Refer to the determinative method for holding times of the stock solutions.

Surrogate standards - A surrogate (i.e., a compound that is chemically similar to the analyte group but is not expected to occur in an environmental sample) should be added to each sample, blank, laboratory control sample (LCS), and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits.

Recommended surrogates for certain analyte groups are listed in each method. For methods where no recommended surrogates are listed, the lab is free to select compounds that fall within the definition provided above. Even compounds that are on the method target analyte list may be used as a surrogate as long as historical data are available to ensure their absence at a given site. Normally one or more standards are added for each analyte group.

Prepare a surrogate spiking concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially prepared spiking solutions are acceptable. The concentration for semivolatile/nonvolatile organic and pesticide analyses should be such that a 1-mL aliquot into 1000 mL of a sample provides a concentration of 10 times the quantitation limit or near the mid-point of the calibration curve. Where volumes of less than 1000 mL are extracted, adjust the volume of surrogate standard proportionately. For matrices other than water, 1 mL of surrogate standard is still the normal spiking volume. However, if gel permeation chromatography will be used for sample cleanup, 2 mL should be added to the sample. The spiking volumes are normally listed in each extraction method. Where concentrations are not listed in a method, a



## Organic Extraction and Sample Preparation

### Doc. 07

Method 3500B

Revision 1

Effective: March 21, 2000

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concentration of 10 times the quantitation limit is recommended. If the surrogate quantitation limit is unknown, the average quantitation limit of method target analytes may be utilized to estimate a surrogate quantitation limit. As necessary or appropriate to meet project objectives, the surrogates listed in the method may be modified by the laboratory. The concentration of the surrogate in the sample (or sample extract) should either be near the middle of the calibration range or approximately ten times the quantitation limit.

**Matrix spike standards** - The following are recommended matrix spike standard mixtures for a few analyte groups. Prepare a matrix spike concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially-prepared spiking solutions are acceptable. The matrix spike standards should be independent of the calibration standard. A few methods provide guidance on concentrations and the selection of compounds for matrix spikes.

**Base/neutral and acid matrix spiking solution** - Prepare a spiking solution in methanol that contains each of the following base/neutral compounds at 100 mg/L and the acid compounds at 200 mg/L for water and sediment/soil samples. The concentration of these compounds should be five times higher for waste samples.

#### Base/neutrals

1,2,4-Trichlorobenzene  
Acenaphthene  
2,4-Dinitrotoluene  
Pyrene  
N-Nitroso-di-n-propylamine  
1,4-Dichlorobenzene

#### Acids

Pentachlorophenol  
Phenol  
2-Chlorophenol  
4-Chloro-3-methylphenol  
4-Nitrophenol

**Organochlorine pesticide matrix spiking solution** - Prepare a spiking solution in acetone or methanol that contains the following pesticides in the concentrations listed for water and sediment/soil. The concentration should be five times higher for waste samples.

#### Pesticide

#### Concentration (mg/L)

Lindane	0.2
Heptachlor	0.2
Aldrin	0.2
Dieldrin	0.5
Endrin	0.5
4,4'-DDT	0.5

For methods with no guidance, select five or more analytes (select all analytes for methods with five or less) from each analyte group for use in a spiking solution. Where matrix spike



concentrations in the sample are not listed it should be at or below the regulatory concentration or action level, or 1 to 5 times higher than the background concentration, whichever, concentration would be larger.

As necessary or appropriate to meet project objectives, the matrix spiking compounds listed in each method may be modified by the laboratory. When the concentration of an analyte is not being checked against a regulatory limit or action level, the concentration of the matrix spike compound in the sample (or sample extract) should be near the middle of the calibration range or approximately ten times the quantitation limit.

Laboratory control spike standard - Use the matrix spike standard as the spike standard for the laboratory control sample (LCS). The LCS should be spiked at the same concentration as the matrix spike.

## **VII. Sample Collection, Preservation, and Handling**

See Chapters Two and Four of SW 846 for guidance on sample collection.

## **VIII. Procedure**

Water, soil/sediment, sludge, and waste samples requiring analysis for semivolatile and nonvolatile organic compounds (within this broad category are special subsets of analytes, i.e., the different groups of pesticides, explosives, PCBs etc.), must undergo solvent extraction prior to analysis. This manual contains method choices that are dependent on the matrix, the physical properties of the analytes, the sophistication and cost of equipment available to a given laboratory, and the turn-around time required for sample preparation.

The laboratory should be responsible for ensuring that the method chosen for sample extraction will provide acceptable extraction efficiency for the target analytes in a given matrix. There are several approaches that may be employed to ensure the appropriateness of the extraction method.

Prior to employing any extraction procedure on samples submitted for regulatory compliance monitoring purposes, the laboratory should complete the initial demonstration of proficiency described in each method. This demonstration applies to all SW-846 extraction methods, including those for which specific performance data are provided in a determinative method.

In addition, when a new or different extraction technique is to be applied to samples, the laboratory should also demonstrate that their application of the technique provides acceptable performance in the matrix of interest for the analytes of interest. One approach to demonstrating extraction method performance is to make a direct comparison between the chosen method and either Method 3520 (continuous liquid-liquid extraction of aqueous samples) or Method 3540 (Soxhlet extraction of solid samples), as these methods have the broadest applicability to environmental matrices.

When direct comparisons are performed, they should be conducted using either standard reference materials derived from real-world matrices or samples from a given site that can be reasonably expected to contain the analytes of interest. Because of concerns with the incorporation of spiking materials into samples, the use of samples spiked by the laboratory is generally a less useful comparison relative to either real-world contaminated samples or standard



## Organic Extraction and Sample Preparation

### Doc. 07

Method 3500B

Revision 1

Effective: March 21, 2000

---

reference materials, and thus should generally only be employed when neither of these latter materials are available. Analyze at least four portions of a well homogenized sample by the extraction method of interest and either Method 3520 or Method 3540, depending on the matrix.

When direct comparisons between methods are conducted, the laboratory may use statistical tests such as an F-test to determine if the results are comparable between the methods. The laboratory may employ the method of interest provided that the demonstrated performance can be shown to be either as good or better than that of the “reference” method, or adequate for project needs, that is, meeting the requirements of the QA Project Plan for a specific project.

Whatever approaches are taken to ensure the adequacy of the extraction procedure for the matrix of interest, it is the responsibility of the laboratory to document the results and maintain records of such demonstrations.

Each method has QC requirements that normally include the addition of surrogates to each analytical sample and QC sample as well as the inclusion of a matrix spike/matrix spike duplicate (or, matrix spike and duplicate sample), a laboratory control sample, and a method blank in each sample extraction batch. As defined in Chapter One of SW 846, a “batch” consists of up to 20 environmental samples processed as a unit. In the case of samples that must undergo extraction prior to analysis, each group of 20 samples extracted together by the same method constitutes an extraction batch.

The decision of whether to prepare and analyze a matrix spike/matrix spike duplicate pair or a matrix spike and a duplicate sample should be based on knowledge of the samples in the extraction batch. If the samples are expected to contain the analytes of interest, then the analysis of a duplicate sample may yield data on the precision of the analytical process and the analysis of the internal spike will yield data on the accuracy of the process. In contrast, when the samples are not known or expected to contain the analytes of interest, then the batch should include a matrix spike/matrix spike duplicate pair to ensure that both accuracy and precision data will be generated within the extraction batch.

Method 3510 - Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is solvent extracted using a separatory funnel. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Separatory funnel extraction utilizes relatively inexpensive glassware and is fairly rapid (three, 2-minute extractions followed by filtration) but is labor intensive, uses fairly large volumes of solvent and is subject to emulsion problems. Method 3520 should be used if an emulsion forms between the solvent-sample phases, which cannot be broken by mechanical techniques.

Method 3520 - Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is extracted with an organic solvent in a continuous liquid-liquid extractor. The solvent must have a density greater than that of the sample. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Continuous extractors are excellent for samples with particulate (of up to 1% solids) that cause emulsions, provide more efficient extraction of analytes that are more difficult to extract and once loaded, require no hands-on manipulation. However, they



## Organic Extraction and Sample Preparation

### Doc. 07

Method 3500B

Revision 1

Effective: March 21, 2000

---

require more expensive glassware, use fairly large volumes of solvent and extraction time is rather lengthy (6 to 24 hours).

Method 3535 - Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of water is pumped through an appropriate medium (e.g., disk or cartridge) containing a solid phase that effects the extraction of organics from water. A small volume of extraction solvent is passed through the medium to elute the compounds of interest. The eluant is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Appropriate solid-phase extraction media allow extraction of water containing particulates, are relatively fast and use small volumes of solvent. However, they do require some specialized pieces of equipment.

Method 3540 - This method is applicable to the extraction of nonvolatile and semivolatile organic compounds from solids such as soils, relatively dry sludges, and solid wastes. A solid sample is mixed with anhydrous sodium sulfate, placed into an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Soxhlet extraction uses relatively inexpensive glassware, once loaded requires no hands-on manipulation, provides efficient extraction, but is rather lengthy (16 to 24 hours) and uses fairly large volumes of solvent. It is considered a rugged extraction method because there are very few variables that can adversely affect extraction efficiency.

Method 3541 - This method utilizes a modified Soxhlet extractor and is applicable to the extraction of semivolatile/nonvolatile organic compounds from solids such as soils, relatively dry sludges, and solid wastes. A solid sample is mixed with anhydrous sodium sulfate, placed into an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in an automated Soxhlet extractor. This device allows the extraction thimble to be lowered into the boiling liquid for the first hour and then extracted in the normal thimble position for one additional hour. The automated Soxhlet allows equivalent extraction efficiency in 2 hours, combines the concentration step within the same device but requires a rather expensive device.

Method 3542 - This method is applicable to the extraction of semivolatile organic compounds from the Method 0010 air sampling train. The solid trapping material (i.e., glass or quartz fiber filter and porous polymeric adsorbent resin) are extracted using Soxhlet extraction and the condensate and impinger fluid are extracted using separatory funnel extraction.

Method 3545 - This method is applicable to the extraction of nonvolatile/semivolatile organic compounds from solids such as soils, relatively dry sludges, and solid wastes. A solid sample is mixed with anhydrous sodium sulfate or diatomaceous earth, placed into an extraction cell and extracted under pressure with small volumes of solvent. The extract is concentrated and, if necessary, exchanged into a solvent compatible with further analysis. The method is rapid and efficient, in that it uses small volumes of solvent, but does require the use of an expensive extraction device.

Method 3550 - This method is applicable to the extraction of nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes using the technique of ultrasonic extraction. Two procedures are detailed depending upon the expected concentration of organics in the sample; a low concentration and a high concentration method. In both, a known weight of



sample is mixed with anhydrous sodium sulfate and solvent extracted using ultrasonic extraction. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Ultrasonic extraction is fairly rapid (three, 3-minute extractions followed by filtration) but uses relatively large volumes of solvent, requires a somewhat expensive device and requires following the details of the method very closely to achieve acceptable extraction efficiency (proper tuning of the ultrasonic device is very critical). This technique is much less efficient than the other extraction techniques described in this section. This is most evident with very non-polar organic compounds (e.g., PCBs, etc.) that are normally strongly adsorbed to the soil matrix. EPA has not validated Method 3550 for the extraction of organophosphorus compounds from solid matrices. In addition, there are concerns that the ultrasonic energy may lead to breakdown of some organophosphorus compounds. As a result, this extraction technique should not be used for organophosphorous compounds without extensive validation on real-world samples. Such studies should assess the precision, accuracy, ruggedness, and sensitivity of the technique relative to the appropriate regulatory limits or project-specific concentrations of interest.

Methods 3560 and 3561 - These methods are applicable to the extraction of total recoverable petroleum hydrocarbons and PAHs from solids such as soils, sludges, and wastes using the technique of supercritical fluid extraction (SFE). SFE normally uses CO<sub>2</sub> (which may contain very small volumes of solvent modifiers). Therefore, there is no solvent waste for disposal, may be automated, provides relatively rapid extraction, but, is currently limited to total recoverable petroleum hydrocarbons and PAHs. It also requires a rather expensive device and sample size is more limited. Research on SFE is currently focusing on optimizing supercritical fluid conditions to allow efficient extraction of a broader range of RCRA analytes in a broad range of environmental matrices.

Method 3580 - This method describes the technique of solvent dilution of non-aqueous waste samples. It is designed for wastes that may contain organic chemicals at a level greater than 20,000 mg/kg and that are soluble in the dilution solvent. When using this method, the analyst must use caution in the addition of surrogate compounds, so as not to dilute out the surrogate response when diluting the sample.

Sample analysis - Following preparation of a sample by one of the methods described above, the sample is ready for further analysis. Samples prepared for semivolatile/nonvolatile analysis may, if necessary, undergo cleanup prior to application of a specific determinative method.

## **IX. Quality Control**

Refer to Chapter One for specific guidance on quality control procedures. Each laboratory using SW-846 methods should maintain a formal quality assurance program. Each extraction batch of 20 or less samples should contain: a method blank; either a matrix spike/matrix spike duplicate or a matrix spike and duplicate samples; and a laboratory control sample, unless the determinative method provides other guidance.

Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This will include a combination of the sample extraction method (usually a 3500 series method for



extractable organics) and the determinative method (an 8000 series method). The laboratory should also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

The reference samples are prepared from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate should be made using stock standards prepared independently from those used for calibration.

The procedure for preparation of the reference sample concentrate is dependent upon the method being evaluated. Guidance for reference sample concentrations for certain methods are listed below. In other cases, the determinative methods contain guidance on preparing the reference sample concentrate and the reference sample. If no guidance is provided, prepare a reference sample concentrate in methanol (or other water miscible solvent). Spike the reference sample at the concentration on which the method performance data are based. The spiking volume added to water should not exceed 1 mL/L so that the spiking solvent will not decrease extraction efficiency. If the method lacks performance data, prepare a reference standard concentrate at such a concentration that the spike will provide a concentration in the dean matrix that is 10 - 50 times the MDL for each analyte in that matrix.

The concentration of target analytes in the reference sample may be adjusted to more accurately reflect the concentrations that will be analyzed by the laboratory. If the concentration of an analyte is being evaluated relative to a regulatory limit or action level, see each method for information on selecting an appropriate spiking level.

To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. Therefore, 1 mL (unless the method specifies a different volume) of the reference sample concentrate is spiked into each of four (minimum number of replicates) 1-L aliquots of organic-free reagent water (now called the reference sample), extracted as per the method. For matrices other than water or for determinative methods that specify a different volume of water, add 1.0 mL of the reference sample concentrate to at least four replicates of the volume or weight of sample specified in the method. Use a dean matrix for spiking purposes (one that does not have any target or interference compounds) e.g., organic-free reagent water for the water matrix or sand or soil (free of organic interferences) for the solid matrix.

## **X. Preparation of Reference Samples**

The following sections provide guidance on the QC reference sample concentrates for many SW-846 determinative methods. The concentration of the target analytes in the QC reference sample for the methods listed below may need to be adjusted to more accurately reflect the concentrations of interest in different samples or projects. If the concentration of an analyte is being evaluated relative to a regulatory limit or action level, see each method for information on selecting an appropriate spiking level. In addition, the analyst may vary the concentration of the spiking solution and the volume of solution spiked into the sample. However, because of concerns



about the effects of the spiking solution solvent on the sample, the total volume spiked into a sample should generally be held to no more than 1 mL.

## **XI. Sample Quality Control for Preparation and Analysis**

Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair per analytical batch. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. See each method for additional guidance on matrix spike preparation and for guidance on establishing the concentration of the matrix spike compounds in the sample chosen for spiking. The choice of analytes to be spiked should reflect the analytes of interest for the specific project. Thus, if only a subset of the list of target analytes provided in a determinative method are of interest (e.g., Method 8270 is used for the analysis of only PAHs), then these would be the analytes of interest for the project. In the absence of project-specific analytes of interest, it is suggested that the laboratory periodically change the analytes that are spiked with the goal of obtaining matrix spike data for most, if not all, of the analytes in a given determinative method.

A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume: e.g., organic-free reagent water for the water matrix or sand or soil (free of organic interferences) for the solid matrix. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

The concentration of the matrix spike sample and/or the LCS should be determined as described in the following sections.

If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit or action level, the spike should be at or below the regulatory limit or action level, or 1 - 5 times the background concentration

(if historical data are available), whichever concentration is higher.

If historical data are not available, it is suggested that an uncontaminated sample of the same matrix from the site be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the spike should be at the same concentration as the reference sample or 20 times the quantitation limit in the matrix of interest. It is again suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.



## Organic Extraction and Sample Preparation

### Doc. 07

Method 3500B

Revision 1

Effective: March 21, 2000

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Analyze these QC samples (the LCS and the matrix spikes or the optional matrix duplicates) following the procedure of the selected determinative method. Calculate and evaluate the QC data as outlined in Sec. 8.0 of Method 8000.

Blanks - Use of method blanks and other blanks are necessary to track contamination of samples during the sampling and analysis processes. Refer to Chapter One for specific quality control procedures.

Surrogates - A surrogate is a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample. Surrogate should be added to all samples when specified in the appropriate determinative method

The laboratory must have procedures in place for documenting and charting the effect of the matrix on method performance. Refer to Chapter One and Method 8000 for specific guidance on developing method performance data.

## **XII. Method Performance**

The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.

The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.



## **Pressurized Fluid Extraction**

### **METHOD 3545**

Prepared by:   
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Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 0, December 1996, Method 3545.

#### **I. Applicability:**

Analyte:            Pest/PCB's, Fuels, BNA  
Matrix:            Soil, sludge, wastes  
Regulation:        RCRA

#### **II. Important Notes:**

All samples should be treated as potential health hazards.

Hexane is a flammable substance, handle with caution and use in the hood only.

All glassware, glass wool, and boiling chips used in this procedure must be organic cleaned following the Cleaning SOP; additional heating to 400<sup>0</sup>C for 4 hours is also suggested.

Care must be taken to avoid phthalate contamination from gloves, plastic, or any object that will transfer phthalates to the sample extract.

All extractions and pH adjustments must be performed in the hood.

#### **III. Extraction solvents:**

PCB's are extracted with hexane. BNA are extracted with Methylene Chloride and Acetone.  
Fuels are extracted with Methylene Chloride.

#### **IV. Extraction Procedure:**

Prepare a sample by transferring a 30g aliquot (10g for fuels) to a tared solvent rinsed glass beaker. Lesser amounts may be used to comply with specific project needs.



The sample particle reduction must be performed on a sample that will not pass through a 100-200 mesh (150-75 um) sieve. The reduction must be accomplished through grinding with solvent clean apparatus.

Add approximately 30 g of Diatomaceous Earth (DE) and mix thoroughly until a free flowing homogenous mixture is established. **Samples must be dry and free flowing for a proper extraction.** Alternately,  $\text{Na}_2\text{SO}_4$  may be used as a drying agent when hexane is the lone solvent. Spike with the appropriate solution the LCS and MS/MSD and surrogate set directly into the sample matrix.

Setup a solvent cleaned 60 mL cell with a bottom filter and cap (Dionex logo on the top end). Add additional Diatomaceous Earth (DE) through a funnel to cover the bottom of the cell. Alternately, use 100 mL cells to accommodate the sample volume required.

Transfer the sample mixture to the cell and add the spiking solution; rinse the beaker with 5-10 mL of solvent into the cell.

Clean the screw threads, and hand tighten top cap.

Repeat procedure for all samples to be analyzed in the batch (20 or less).

Load the cells into the appropriate locations in the ASE tray with the Dionex logos on the top.

Set up the labeled collection bottles according to the manufacturer's recommendations.

Run one to two rinse cycles (priming) prior to analysis if changing over solvent types, refilling the reservoir, or if air bubbles are trapped in the solvent line.

Extract the samples under the developed ASE time/ temperature sequence method used to establish proficiency.

Transfer the solvent through a sodium sulfate filter setup from each collection bottle (approximate volume should be 50 mL) to an individual solvent cleaned Turbovap tube.

Rinse each collection bottle with 5-10 mL of solvent; add the washings to the appropriate sample extract in the Turbovap tube.

Proceed with the concentration process below.

**Note: Allow the extraction cells to cool adequately prior to cleaning. All solid waste must be disposed of in accordance to the laboratory CHP.**

## V. Sample Concentration

1. Turn on the Turbovap unit along with the nitrogen supply to the unit. Check the level of the water in the water bath. If necessary, add deionized water so as to bring the level to the evaporation tube holder.
2. Set the water bath temperature at 42<sup>0</sup>C.
3. Make sure the control labeled DRYNESS OF SAMPLE is set to NO for each tube.
4. When the temperature of the water bath reaches 42<sup>0</sup>C, open the cover and place the tubes with extracts in the appropriate positions. Place closures on any empty positions. Close the cover; this starts the concentration process. When the evaporation is completed, the



Turbovap will emit an audible signal. Rinse the sides of the Turbovap tube periodically with solvent during the concentration process.

5. Adjust the final volume of the extract to 2.0 mL with the solvent.

Transfer the extract to a labeled 4.0 mL screw cap vial.

Determine if the extract requires additional cleanup steps due to matrix, color, or high viscosity. If extract is clean it is ready for analysis.

## **VI. Calculations:**

Record appropriate dilutions as required.

## **VII. Quality Assurance:**

All quality control data should be maintained and available for easy reference or inspection.

Analyze a reagent blank with each batch of samples analyzed. The result for the reagent blank must be less than the quantitation limit.

Analyze a laboratory control sample at a minimum frequency of one per batch of 20 samples or less per matrix, per day. The LCS for PCBs must be from a second source and have two concentrations of chromatographically distinct target Aroclors at or near the mid-point of the calibration range.

Analyze matrix spikes and matrix spike duplicates at a minimum frequency of one per 20 per matrix or one per month, whichever is more frequent. The spiking must be the same as for the LCS. Refer to the Premier Laboratory recovery data for control limits for this method.

## **VIII. Reagents and Materials:**

Turbovap concentration unit.

ASE 300- Accelerated Solvent Extractor, Dionex Corp.

Extraction Vessels, 60 mL, 100 mL

Drying oven, 105<sup>o</sup>C.

Desiccator.

Beakers, 400 mL

Spatula, disposable wood or Teflon® coated



Glass wool.

Solvent vessel

Syringes, 10 uL, 25 uL 100 uL.

Vials, 2.5 mL, 4.0 mL, screw cap.

Collection bottles, 250 mL

Methylene chloride, pesticide grade.

Sodium sulfate, anhydrous, heated to 400<sup>0</sup>C for 4 hours.

Diatomaceous Earth, heated to 400<sup>0</sup>C for 4 hours

Hexane, pesticide grade.

Acetone, pesticide grade.

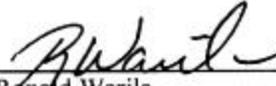
PCB surrogate spiking solution. Purchased at 8 ug/ mL.

Whatman # 41 filters.



## Acid-Base Partition Cleanup Method 3650B

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 2, December 1996, Method 3650B.

### I. Applicability:

Analyte: Acids-Bases  
Matrix: Extracts  
Regulation: RCRA

### II. Important Notes:

This technique will separate acids, organic acids, and phenols, from base/neutral analytes, using pH adjustments. It may also be used for cleanup of petroleum waste prior to analysis.

All samples should be treated as potential health hazards.

All glassware, glass wool, and boiling chips used in this procedure must be organic cleaned by rinsing with acetone and methylene chloride, or by heating to 400°C for 4 hours.

Methylene chloride creates excessive pressure in the separatory funnel very rapidly. Initial venting should be done immediately after the separatory has been sealed and shaken once.

### III. Procedure:

1. Place 10 mL of the solvent extract from a prior extraction procedure into a 125 mL separatory funnel.
2. Add 20 mL of methylene chloride to the separatory funnel.
3. Slowly add 20 mL of pre-chilled organic free reagent water, which has been previously adjusted to a pH of 12-13 with 10 N sodium hydroxide.
4. Seal and shake the separatory funnel for at least 2 minutes with periodic venting to release excess pressure.



5. Allow the organic layer to separate from the aqueous phase for a minimum of 10 minutes. If emulsions occur, appropriate techniques must be employed to break them up.
6. Separate the aqueous phase and transfer it to a 125 mL Erlenmeyer flask. Repeat the extraction two more times using 20 mL aliquots of dilute sodium hydroxide ( pH 12-13). Combine the aqueous extracts. The acids and phenols are contained in the aqueous phase. The base/neutrals are in the methylene chloride phase. Proceed to step 9 if only the base neutrals are of interest.
7. If acids are of interest externally cool the 125 mL Erlenmeyer flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid ( 1:1 ). Quantitatively transfer the cool aqueous phase to a clean 125 mL separatory funnel. Add 20 mL of methylene chloride to the separatory funnel and shake for at least 2 minutes. Allow the methylene chloride to separate from the aqueous phase and collect the methylene chloride in an Erlenmeyer flask.
8. Add 20 mL of methylene chloride to the separatory funnel and extract at pH 1-2 a second time. Perform a third extraction in the same manner combining the extracts in the Erlenmeyer flask.
9. Dry the acid and base/neutral fractions by passing them through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried fractions in a Turbovap tube.
10. Proceed to concentrating the solvent.

#### **IV. Calculations:**

Record all dilutions in the prep logbook.

#### **V. Quality Assurance:**

All quality control data should be maintained and available for easy reference or inspection.

Analyze a reagent blank with each batch of samples analyzed. The result for the reagent blank must be less than the quantitation limit.

#### **VI. Reagents and Materials:**

Chromatography column: 20 mm ID, with Pyrex glass wool at bottom and a PTFE stopcock.

Beakers, appropriate sizes.

Erlenmeyer flasks, 125 mL.

Boiling chips, solvent extracted, approximately 10/40 mesh.

pH paper.

Sodium hydroxide, 10N: Dissolve 40 g of sodium hydroxide in 100 mL of reagent grade water.

Sulfuric acid, 1:1 : Slowly add 50 mL sulfuric acid to 50 mL of reagent grade water.



Separatory funnel, 125 mL.

Water bath.

Vials, 2.5 mL.

Disposable Pasteur pipettes.

Sodium sulfate, anhydrous, heated to 400°C for 4 hours.

Methylene chloride, pesticide grade.

Acetone, pesticide grade.



## Sulfuric Acid Cleanup for PCBs METHOD 3665A

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 1, December 1996, Method 3665A.

### I. Applicability:

Analyte: PCB's  
Matrix: Hexane extracts  
Regulation: RCRA

### II. Important Notes:

Concentrated sulfuric acid is corrosive, and extremely irritating to skin and mucous membranes. Work with concentrated acids in a fume hood. When preparing diluted acid solutions, always add the concentrated acid to the water.

This technique will not destroy chlorinated benzenes, chlorinated naphthalenes, and a number of chlorinated pesticides.

This method is only used to clean up for PCB analysis.

This method will destroy many target analytes not associated with PCB analysis.

All extracts must be previously exchanged into hexane before acid cleanup.

### III. Procedure:

#### Acid Cleanup

Using a disposable pipette, transfer 1.0 mL of hexane extract to a 4 mL vial while working in the fume hood.

Carefully add 3 mL of 1:1 sulfuric acid to the vial with another Pasteur pipette.



Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

Cap the vial tightly; agitate for approx. 20 sec. and vortex for one minute. A vortex must be visible in the vial. Stop the vortex immediately if the vial leaks, the acid will cause severe burns the skin.

Allow the phases to separate for 1 minute. Examine the top ( hexane ) layer. If initial cleanup is successful, the hexane layer will be color free with no visible emulsion or cloudiness present.

If an emulsion forms and persists for several minutes, carefully remove the sulfuric acid layer from the vial (do not remove any hexane)and dispose of it properly. Add another 5 mL of 1:1 sulfuric acid and repeat the cleanup procedure.

If the hexane layer is color free and has no visible emulsion or cloudiness present then proceed.

Transfer the hexane layer to a clean 10 mL (or appropriate volume) vial.

Add an additional 1-2 mL of hexane to the acid layer, seal and shake. This second extraction ensures the quantitative transfer of the target compounds.

If the cleanup does not produce a clean hexane layer, either repeat the acid cleanup on the hexane fractions or proceed to the permanganate cleanup.

Upon successful cleanup, reduce the combined hexane layers to the original sample extract volume using the method prescribed in the extraction SOP.

### **Permanganate Cleanup**

The permanganate cleanup should be used prior to reduction of the hexane fractions when the sulfuric acid cleanup has not removed all of the color from an extract.

Add 5 mL of a 5% permanganate solution to the hexane fractions.

Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

Cap the vial tightly and vortex for 1 minute. A vortex must be visible in the vial. Stop the vortex immediately if the vial leaks, the permanganate will cause severe burns the skin.

Allow the phases to separate for a least 1 minute. Examine the top (hexane) layer for coloration, emulsion or cloudiness.

If an emulsion forms and persists for several minutes, carefully remove the permanganate layer from the vial (do not remove any hexane)and dispose of it properly. Add another 5 mL of 5% permanganate and repeat the cleanup procedure.

If further cleanup is not required, proceed by transferring the hexane layer to a clean 10 mL vial.

Add an additional 1-2 mL of hexane to the permanganate layer, seal, and shake.

Collect the second hexane extract and combine with the first hexane layer in the clean 10 mL vial.

Reduce the hexane layers to the original sample extract volume by quantitatively transferring the hexane layers to a Turbovap tube and using the method prescribed in the extraction SOP for reduction.



#### **IV. Calculations:**

Record all dilutions in the prep log book.

#### **V. Quality Assurance:**

All quality control data should be maintained and available for easy reference or inspection.

Blanks and replicate analysis samples must be processed with this cleanup procedure.

#### **VI. Reagents and Materials:**

Vortex mixer.

Vials, scintillation, 2.5 mL.

Disposable Pasteur pipettes.

Sulfuric acid, 1:1 solution with reagent water (v/v).

Potassium Permanganate Solution: 5%  $\text{KMnO}_4$  dissolved in reagent water, (w/v).

Hexane, Pesticide grade.



## Mercury in Solids (Automated)

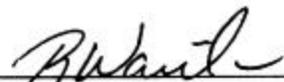
Method 7471A

Revision 2.2

Effective Date: January 24, 2004

### Mercury in Solids (Automated) SW-846 7471A

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

#### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 1, September 1994, Method 7471A.

#### I. Applicability:

Analyte: Mercury

Matrix: Soil, sludge, and waste extracts.

Regulation: RCRA

#### II. Important Notes:

Method 7471A was developed to perform mercury in soil analysis via manual determination by cold vapor. Premier Laboratory employs the use of a Perkin Elmer FIMS 100 automated mercury analysis system. The FIMS 100 system was developed to replace manual determination of mercury by cold-vapor atomic absorption with an automated approach. Digestates are placed on the FIMS 100 autosampler where reagents are automatically added to the samples. The FIMS 100 software controls the addition of reagents, construction of the calibration curves, and the calculations for mercury determination in the samples.

During the digestion, potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm.



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mg/L).

Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

The instrument method detection limit study must achieve a minimum detection of 0.005 mg/Kg.

The reporting limit for soils before dry weight adjustment is 0.02 mg/Kg.

The following cleaning sequence must be used for all glassware that will contact samples to be analyzed for metals:

Detergent wash

Tap water rinse

1:1 nitric acid rinse

Reagent water rinse

1:1 hydrochloric acid rinse

Reagent water rinse

### III. Procedure:

Transfer 1.0 g to 2.0g of sample to a 300 mL BOD bottle.

Add 10 mL of 1:1 Aqua Regia / Water, cover and transfer to a 95°C water bath for 2 minutes.

Remove and allow to cool. Add 50 mL of reagent water and 15 mL of  $\text{KMnO}_4$ . Sewage samples and samples containing a high salt content may require additional portions of potassium permanganate solution. If necessary, add 3.0 mL portions until the purple color persists for at least 15 minutes. Be sure to mix sample after each addition. Track and record the additions to determine final volume.

Add 8 mL of potassium persulfate solution, return to the water bath for 30 minutes, covered.

Cool and add 6 mL of hydroxylamine hydrochloride solution and 50 mL of distilled water.

When the solution has been de-colored, transfer to the autosampler.

All standards, quality control samples, and unknown samples are prepared at the same time and in the same manner.

#### Standard Concentrations



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

**Intermediate Stock Solution:** Combine 0.1 mL of (1000 ppm ) Hg stock, 5.0 mL H<sub>2</sub>SO<sub>4</sub>, 2.5 mL HNO<sub>3</sub>, and 91.5 mL of reagent water in a 100 mL volumetric flask. The final concentration of the stock solution is 1000 ppb Hg.

**Calibration standards:** All standards are prepared by transferring the appropriate aliquot of intermediate stock solution directly to digestion vessels. All standards are then carried through the entire digestion procedure.

**The following is an example of dilutions made from a 1000ppm working solution into a 100 mL final volume.**

<u>Standards</u>	<u>Aliquot added in mL</u>	<u>Final Conc. in ppb</u>
STD 1(blank)	0.0	0.0
STD 2	0.02	0.2
STD 3	0.1	1.00
STD 4	0.2	2.00
STD 5	0.5	5.00
STD 6	1.0	10.00

Proceed with calibration and analysis per FIMS 100 manual. The Zero Intercept Linear Calibration is represented by a straight line defined using the equation:

$$C = K_0 ( -K_1A )$$

A calibration curve defined using this equation is forced to go through zero absorbance and zero concentration. A least squares technique is use to determine the K1 coefficient when two or more standards are used for calibration. Ko is the re-slope coefficient, which is set to 1.0 during initial calibration.



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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### Hg Analysis Sequence

1 to 6	Calibration Standards 1 to 6
7	ICB
8	ICV
9	LRB (Laboratory Reagent Blank)
10	LFB (Laboratory Fortified Blank)
11	LLCS (Low Level Check Standard)
12	Sample 1
13	Sample 1 Duplicate
14 to 15	Sample 1 LFM/LFMD
16 to 20	5 Samples
21	CCB
22	Low CCV
23	Mid CCV
24 to 33	10 Samples
34	CCV
35	CCB

Transfer the samples to the FIMS 100 autosampler and proceed with analysis per FIMS manual setup.

### IV. Calculations:

Direct reading in ug/L from the mercury autoanalyzer.

### V. Quality Assurance:

All quality control data should be maintained and available for easy reference or inspection.

#### Calibration Solutions

The calibration solutions are to be made using the same or similar acid matrix as the samples to be analyzed.

#### Initial Calibration Verification (ICV)

The ICV must be made from an outside second source different from that of the calibration standards' stock solutions.



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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The ICV is run immediately following the calibration and is used to verify calibration standards or stock solutions. The ICV recovery must be within  $\pm 10\%$  of the true value for each analyte of interest.

### **Continuing Calibration Verification (CCV)**

The CCV must be run periodically (every 10 samples) and at the end of each analytical sequence. The CCV is made from the same source as the calibration standards at mid-level concentration.

The recovery must be  $\pm 20\%$  of the true value. The CCV may be run one additional time if the specified recoveries are not met, however if the second analysis fails, corrective action must be taken and any samples analyzed after the previous valid CCV must be re-analyzed.

### **Calibration Blank**

The calibration blank contains the same acid matrix as the calibration standards and run with each ICV. The calibration blank is also used as the Continuing Calibration Blank (CCB) solution. See note 1. The criteria by which the blank results are to be evaluated is the following:

The results of the calibration blank are to agree within  $\pm$  the PQL. If not, repeat the analysis two more times and average the results. If the average is not within  $\pm$  the PQL, terminate the analysis, correct the problem; re-calibrate; and reanalyze the previous 10 samples.

### **Laboratory Reagent Blank (LRB)**

The LRB is a reagent blank carried through the entire sample preparation process.

Employ a minimum of one laboratory reagent blank with each batch of 20 or fewer samples of the same matrix, to verify the absence of contamination. The LRB must be less than the reported detection limit.

### **Laboratory Fortified Blank (LFB)**

A laboratory fortified blank (LFB) must be run with each sample batch. If the recovery falls outside the control limit of 80-120% or established control limits, the problem is to be identified and resolved before continuing.

### **Sample Duplicate**

Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of  $\pm 20\%$  for RPD shall be used for sample values greater than 5 times the method detection limit. Samples less than 5 times the detection limit should be within  $\pm$  the method detection limit. Method 245.1 does not require the sample duplicate to be run, but it should remain an element of the normal QA protocol.

### **Laboratory Fortified Matrix / Duplicate (LFM/LFMD)**

The LFM/LFMD pair must be run with each batch of 20 or fewer samples of the same matrix.

The LFM/LFMDs are prepared from fresh sample aliquots, spiked in the same manner as the LFB and carried through the entire preparation process.



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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The matrix spike and matrix spike duplicate spike recovery should be within  $\pm 25\%$  of the true value, or documented control limits. Recovery calculations are not made if the spike concentration is less than 25% of the sample concentration.

### Linear Dynamic Range (LDR)

Dilute and reanalyze samples that are  $>90\%$  of the established linear calibration limit or use an alternate, less sensitive line for which quality control data is established.

Linear range should be determined at least every 6 months.

### Method Detection Limit (MDL)

MDLs must be maintained for each analyte of interest and updated once every year.

The determination of MDLs must be made in accordance with the following:

Fortify reagent water at a concentration of 2 to 3 times the estimated instrument detection limit.

Take seven replicate aliquots of the fortified reagent water and process through the entire analytical method.

Perform all calculations defined in the method and report the concentration values in the appropriate units.

Calculate the MDL as follows:

$$\text{MDL} = (t) \times (s)$$

where:

t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.143 for seven replicates].

S = standard deviation of the replicate analyses.

The final calculated MDL must be greater than 20% of the original analyte spike level.

### Matrix Evaluation

It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. They are as follows:

**Serial dilution:** If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected and a post spike must be run.



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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**Post (digestion) Spike:** An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value or the established control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The use of a standard-addition analysis procedure may be used to compensate for this effect.

## VI. Reagents and Materials:

### Hg Analyzer:

Perkin-Elmer FIMS 100 automated mercury system with associated software.

### Analysis vessels:

300 mL BOD bottles, 50 mL polypropylene centrifuge tubes

### Hydrochloric acid:

Concentrated, metals analysis grade.

### Sulfuric acid:

Concentrated, metals analysis grade.

### Sulfuric acid solution: 0.5N

Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.

### Nitric acid:

Concentrated, metals analysis grade.

### Stannous chloride, ( SnCl<sub>2</sub>):

Add 11 g stannous chloride to 1000 mL of 3% hydrochloric acid solution.

### Sodium chloride-hydroxylamine hydrochloride solution:

Dissolve 12 g of sodium chloride (NaCl) and 24 g of hydroxylamine hydrochloride in reagent water and dilute to 100 mL.

### Potassium permanganate,(KMnO<sub>4</sub>):

5% w/v. Dissolve 50 g of potassium permanganate in 1000 mL of reagent water.

### Potassium persulfate, ( K<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>),

5% w/v. Dissolve 50g of potassium persulfate in 1000 mL of reagent water.

### Stock mercury standard:

1000 mg/L, purchased from a certified vendor

### Mercury working standard:



## Mercury in Solids (Automated)

Method 7471A

Revision 2.2

Effective Date: January 24, 2004

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Make successive dilutions of the stock mercury standard to obtain a working standard. The dilutions of the stock mercury standard must be prepared fresh daily. Acidity of the working standard should be 2.5% nitric acid and 5% sulfuric acid. This acid should be added to the volumetric flask before addition of the standard.

### **Safety:**

Every sample should be considered a hazardous when performing the analysis. Standard laboratory safety guidelines must be adhered to. Gloves, eye protection, and lab coats must be worn during sample retrieval, analysis and disposal.

### **Pollution Prevention:**

Any and all remaining unused sample must be returned to the 4°C storage, sealed tightly in the original container. Benches and surrounding surfaces must be cleaned and wiped dry with paper toweling.

### **Waste management:**

Analyzed sample and used disposable equipment must be collected and disposed of in a manner consistent with the Premier Laboratory Chemical Hygiene Plan.

### **Method Performance:**

Performance data is not currently available.



## Volatile Organics by GC/MS

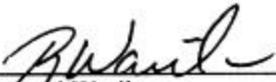
Method 8260B

Revision 2.21

Effective: April 24, 2005

## Volatile Organics by GC/MS Method 8260B

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### REFERENCE

*Test Methods for Evaluating Solid Waste*, SW-846, 3rd Edition, December 1996,  
Method 8260B, Revision 2.

### **Safety:**

All samples submitted to an environmental laboratory should be treated as potential health hazards.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each compound should be treated as a potential health hazard.

### **Definitions:**

BFB: 4-Bromofluorobenzene.

CCC: Calibration check compound.

D: Drift. %D = Percent drift.

EICP: Extracted ion current profile; a plot of ion abundance vs. time or scan number.

EP: Extraction procedure.

FC-43: Perfluoro-tri-N-butylamine.

Field Sample: All samples submitted by the client, including field quality control samples such as field blanks and trip blanks.

GC: Gas chromatograph.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

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GC/MS: Gas chromatograph/mass spectrometer system.

Ion: As used in this document, the  $m/z$  ratio.

LCS: Laboratory control sample; sometimes called a blank spike.

MS: Matrix spike.

MSD: Matrix spike duplicate.

PTFE: Polytetrafluoroethylene (Teflon®)

QL: Quantitation limit.

Quality Control Sample: Samples prepared at the laboratory for quality control purposes, including method blanks, matrix spikes, replicates, blank spikes, *etc.* Calibration standards are not included.

RF: Response factor.

RPD: Relative percent difference.

RRT: Relative retention time.

RSD: Relative standard deviation. %RSD = percent relative standard deviation.

SPCC: System performance check compound.

TCLP: Toxicity characteristic leachate procedure.

### Scope And Application:

Analytes: See Table 1  
Matrices: All liquids, sludges, particulate solids  
Regulations: RCRA and equivalent state regulations

### Sample Collection, Preservation, And Handling:

Using an appropriate sampling device, collect soil samples as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere.

Using the sample collection device, add about 10 grams of soil to a pre-weighed 40-mL, PTFE-lined screw cap, septum sealed vial containing 10 mL of methanol. It is required that



the soil be completely covered by the methanol. Quickly brush off any soil on the vial threads and cap the vial securely. Store samples at 4 °C.

For dry weight determinations, a second 40-mL vial, which does not contain methanol, must be filled with sample.

**Note: Each sampling group must be accompanied by a methanol trip blank for low level and high-level soil sampling.**

### 1.0 Important Notes:

1.1 Every analyst performing this procedure must be familiar with the requirements of the Quality Control and Corrective Action section. For convenience, quality control information required to assess data is referenced in the Procedure section. However, if the criteria referenced are not met, the Quality Control and Corrective Action section must be consulted for additional information and appropriate actions to be taken.

1.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-PTFE tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

1.3 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrupole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

Note: The laboratory where volatile analysis is performed should be completely free of solvents.

### 2.0 Summary of Method:

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass



spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

### 3.0 Interferences:

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve-purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often



results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct Injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

## 4.0 Apparatus And Materials:

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Gas chromatography/mass spectrometer/data system

4.3.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.3.1.1 The GC should be equipped with variable constant differential flow



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.3.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.3.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.3.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

### 4.3.2 Gas chromatographic columns

#### GC Columns

Column	---	Restek, RTX-YMS, 40m x 0.18mm x 1um Restek, RTX-624, 75m x 0.53mm x 3um Restek, RTX-502.2 105m x 0.53mm x 3um
Carrier gas	---	helium

4.3.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB), which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

4.3.4 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB, which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.3.5 GC/MS interface - Two alternatives may be used to interface the GC to the



mass spectrometer.

4.3.5.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25-0.32 mm ID columns.

4.3.5.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.3.5.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.4.0 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time and scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.4.1 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000- $\mu$ L.

4.1.2 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.1.3 Syringes - 5-10-, or 25-mL, gas-tight with shutoff valve.

4.1.4 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1g.

4.1.5 Glass scintillation vials - 40-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.1.6 Disposable pipets - Pasteur.

4.1.7 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.1.8 Spatula -Stainless steel.

## 5.0 Reagents:

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One, Method 8000.

5.3 Methanol, CH<sub>3</sub>OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.5 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.6 Stock solutions are typically prepared using purchased certified solutions, see table 4 and 5 for specific mixes prepared.

5.7 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

### 5.7.1 Frequency of Standard Preparation

5.7.1.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.1.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

### 5.7.6 Optional Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially available gaseous analyte mixture of bromomethane, chloromethane, chloroethane,



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

vinyl chloride, dichlorodifluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

- a) Connect either the 100- $\mu$ L or 500- $\mu$ L Luer syringe to the inlet fitting of the cylinder.
- b) Make sure the on/off valve on the syringe is in the open position.
- c) Slowly open the valve on the cylinder and withdraw a full syringe volume.
- d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.
- e) Expel the gas from the syringe into a well-ventilated area.
- f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is 30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

**NOTE:** Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/L
400 µL	200 µg/L

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed, certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d<sub>8</sub>, 4-bromofluorobenzene, 1,2-dichloroethane-d<sub>4</sub>, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. An internal standard/ surrogate spiking solution is purchased at a concentration of 250 µg/mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with the spiking solution prior to analysis, this is performed by the autosampler in most cases. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d<sub>5</sub>, and 1,4-dichlorobenzene-d<sub>4</sub>. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. An internal standard/surrogate spiking solution is purchased at a concentration of 250 µg/mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with the spiking solution prior to analysis, this is performed by the autosampler in most cases. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/ $\mu$ L of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 All target analytes for a particular analysis should be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

## 6.0 Reagents:

Instrument Performance Check Solution, 4-Bromofluorobenzene. 25.00 µg/mL in methanol.

Matrix Spiking Standard Mix, 200 µg/mL each in methanol.

Methanol, purge and trap grade.

Purgeable Internal Standard/ Surrogate Standard Mix, 250 µg/mL each in methanol.

8260B Liquid 54 Compound Mix, 2000 µg/mL each in methanol.

8260B Gaseous Compounds Mix (6 components), 2000 µg/mL each in methanol.

Various Independent Compounds, certified concentrations between 1000 to 20000 µg/mL each in methanol

Reagent Water: Deionized water in which no contamination is observed at or above the QL for any target compound.

## 7.0 PROCEDURE:

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 The purge-and-trap of aqueous samples is performed at 40°C in addition to the soil/solid samples being performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds, which have poor purging efficiencies at ambient temperatures.

7.1.3 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

### 7.2 Recommended chromatographic conditions

#### Instrument Setup

##### Purge Conditions:

Trap	---	Supelco, Vocab 3000 <sup>®</sup>
Purge gas	---	helium
Purge time	minutes	11
Purge flow rate	mL/minute	30 - 40
Purge temperature	°C	40

##### Desorb Conditions:

Temperature	°C	260
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## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

Flow rate	mL/minute	0
Time	minutes	1-2

### Trap Initial Conditioning:

Temperature	°C	265
Flow rate	mL/minute	15-40 (instrument dependent)
Time	minutes	90

### Trap Reconditioning:

Temperature	°C	265
Flow rate	mL/minute	15
Time	minutes	8

### Trap Reconditioning Between Analyses:

Temperature	°C	265
Flow rate	mL/minute	15-40 (instrument dependent)
Time	minutes	8

Optimize purge and trap conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same purge and trap conditions must be used for the analysis of all standards, field samples, and quality control samples.

Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, quality control samples, and field samples.

### Mass spectrometer

Electron energy	volts(nominal)	70
Mass range	amu	35 - 300
Scan time	seconds	0.6 - 2 sec/scan
Non-target reference library	---	EPA/ NIST

- The GC/MS must be tuned to meet the manufacturer's specifications using FC-43.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

- Prior to the analysis of any standards, quality control samples, or field samples, the analyst must establish that the GC/MS meets the mass spectral ion abundance criteria for BFB. The BFB Performance Check Solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are to be analyzed. The 12-hour tune period for GC/MS instrument performance check, calibration standards, and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after 12 hours has elapsed according to the system clock. The BFB performance check may be taken from the daily calibration standard.
- Analyze 50 ng of BFB Performance Check Solution by direct injection or purge and trap using the gas chromatograph and mass spectrometer conditions specified above.
- All subsequent standards, field samples, and quality control samples associated with a BFB analysis must use identical mass spectrometer instrument conditions.
- The relative ion abundance criteria for BFB are listed in Table 2.
- Internal and surrogate standards must be added to all standards, blanks, and samples. The recommended internal standards are fluorobenzene, chlorobenzene-d<sub>5</sub>, and 1,4-dichlorobenzene-d<sub>4</sub>. The recommended surrogate standards are: toluene-d<sub>8</sub>, 4-bromofluorobenzene, 1,2-dichloroethane-d<sub>4</sub>, and dibromofluoromethane

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 50 ng injection or purging of 4-bromofluorobenzene (2-pL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 2 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

**NOTE:** All subsequent standards, samples, MS/MSDs, LCS's, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples, or Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10  $\mu$ L of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention--times of 0.80-1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2  $\mu$ L into the GC/MS system. The injection volume will depend upon the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

$A_s$  = Peak area (or height) of the analyte or surrogate.

$A_{is}$  = Peak area (or height) of the internal standard.

$C_s$  = Concentration of the analyte or surrogate.

$C_{is}$  = Concentration of the internal standard.



7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1, 1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1, 2, 2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$



### RF

Where:

—

RF = mean RF for each compound from the initial calibration

RF = RF for each of the calibration standards

n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1, 1-Dichloroethene

Toluene

Chloroform

Ethylbenzene

1 2-Dichloropropane

Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before re-attempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

### 7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

**NOTE:** Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 2 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

**NOTE:** The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination then it is appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

### 7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

### 7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of



Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion. Calculate as follows:

$$\% \text{ Drift} = \frac{|C_c - C_t|}{C_t} * 100$$

$$\% \text{ Difference} = \frac{|RF_v - \text{mean RF}|}{\text{mean RF}} * 100$$

where:  $C_c$  = measured concentration  
 $C_t$  = theoretical concentration

$RF_v$  = verification standard response factor  
Mean RF is from the initial calibration

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

## 7.5 GC/MS analysis of samples

All initial and re-analyses of samples must be performed within 14 days of sample collection if samples are preserved, 7 days if not preserved.

TCLP leachates are initially analyzed at a 1:5 dilution.



### Water-Miscible Liquid Technique

All initial and re-analyses of samples must be performed within 14 days of sample collection.

Water-miscible liquids must be diluted at least 1:50 prior to analysis. Either of the following techniques may be used.

Transfer 2 mL of sample or sample dilution to a 100-mL volumetric flask and dilute to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

Prepare the dilution directly in the 5-mL gas-tight syringe by injecting at least 20  $\mu\text{L}$  but no more than 100  $\mu\text{L}$  into a syringe containing 5-mL of reagent water.

Continue the analysis as for water samples.

### Low Concentration Soil/Sediment Technique

All initial and reanalysis of samples must be performed within 14 days of sample collection.

Analyze water-miscible wastes by the Water-Miscible Liquid Analysis technique.

To manually prepare the reagent water containing the surrogate spike compounds and the internal standards, remove the plunger from a 5-mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 5  $\mu\text{L}$  of 8260B Internal/Surrogate Spiking Solution to the syringe through the valve.

The sample for volatile organics consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g of sample into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

If a 5-g sample will cause one or more target compounds to exceed the initial calibration range, the analyst may use a smaller aliquot but not less than 0.5 g. If a 0.5-g aliquot causes one or more target compounds to exceed the initial calibration range, the Medium/High\_Concentration Soil/Sediment Analysis technique must be used.

Immediately add the spiked reagent water to the purge device and connect the device to the purge and trap system.

After preparing the sample, weigh an additional 5-10 g aliquot of sample into a tared crucible. Determine the percent dry weight by drying overnight at 105  $^{\circ}\text{C}$ . Allow it to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of wet sample}} * 100$$

Proceed with the analysis as outlined above for Water Sample Analysis starting with paragraph 7.

### Medium/High Concentration Soil/Sediment Technique

All initial and reanalysis of samples must be performed within 14 days of sample collection. Initial weight is recorded in the Methanol Prep Logbook for methanol preserved VOA vials prior to field sampling. Weigh the received preserved sample vial to determine the actual sample weight by subtracting the initial weight recorded from the total. Add any methanol to bring the ratio to 1:1 of sample to methanol. Apply a factor in cases of a low sample weight versus methanol. The sample for volatile organics consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g (wet weight) into a tared 20-mL vial. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

Quickly add 5 mL of methanol. Cap and shake for 2 minutes. Let the suspended material settle.

These extracts must be stored in the dark at  $\leq 6$  °C prior to analysis.

Generally, a 1:50 dilution is performed on the extracts prior to analysis. If the extract is too concentrated to keep all target compounds within the initial calibration range, use an appropriate dilution in methanol.

After preparing the sample. Weigh an additional 5-10 g aliquot of sample into a tared crucible. Determine the percent dry weight by drying overnight at 105 °C. Allow it to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of wet sample}} * 100$$

Proceed with the analysis as outlined above for Water Sample Analysis, starting with paragraph 7.

### Method blank analysis

A volatile method blank must be prepared and analyzed each 12 hour shift, preferably after the calibration standards and after any high concentration samples.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

The method blank is prepared in the same manner as samples, substituting appropriate blank material for the sample.

The method blank analysis must meet all the requirements of the Quality Control and Corrective Action section before field and other quality control samples can be analyzed. The concentration of each target compound must be less than its required quantitation limit for the project.

### LCS analysis

A volatile LCS should be prepared and analyzed at a frequency of 1 per batch of 20 samples per matrix, not to exceed 30 days between analyses.

The LCS is prepared by adding 12.5  $\mu\text{L}$  of the 8260B MS/MSD Spiking Solution (see table 4) to 5 mL of reagent water. Internal standards/surrogate mixture is added by the autosampler or manually at the time of spiking.

Calculate the LCS recovery.

$$\% \text{ Rec} = \frac{C_S}{C_A} * 100$$

where: %Rec = percent recovery

$C_S$  = analyte concentration recovered

$C_A$  = concentration of analyte added

The LCS should meet the recovery range requirements determined by the laboratory. Although no action is required, frequent failure to meet these limits indicates a problem in the analytical system that should be investigated.

### MS/MSD analysis

A volatile MS/MSD must be prepared and analyzed at a frequency of 1 per 20 samples per matrix prepared by the same technique, not to exceed 30 days between analysis.

If the client has designated a sample to be used for the MS/MSD, this sample must be spiked. Otherwise, a field sample (not a quality control or performance evaluation sample) shall be chosen by the analyst. A known blank (field rinse blank or like) must not be used as a spike sample.

The MS/MSD is prepared as follows.

Add a 12.5  $\mu\text{L}$  of the 8260B MS/MSD Spiking Solution 8260B(see table 4) at a concentration of 200ppm to each of two aliquots of the sample chosen for spiking. Internal standards/surrogate



mixture is added by the autosampler or manually at the time of spiking. The sample volume used for the MS/MSD must be the same as used for the unspiked sample analysis.

Calculate the MS and MSD recoveries.

$$\% \text{Rec} = \frac{C_S - C_U}{C_A} * 100$$

where: %Rec = percent recovery

$C_S$  = analyte concentration in spiked sample

$C_U$  = analyte concentration in unspiked sample

$C_A$  = Concentration of analyte added

Calculate the percent recovery and RPD for the MS/MSD.

$$\% \text{Recovery} = \frac{\text{spiked sample result} - \text{sample result}}{\text{spike added}} * 100$$

$$\text{RPD} = \frac{|\text{matrix spike recovery} - \text{matrix spike duplicate recovery}|}{1/2 * (\text{matrix spike recovery} + \text{matrix spike duplicate recovery})} * 100$$

The MS/MSD must meet the requirements in the Quality Control and Corrective Action section. The quality control limits for MS/MSD recovery are generated by the laboratory on an annual basis and are advisory. Although no action is required, frequent failure to meet these limits indicates a problem in the analytical system that should be investigated. If an MS/MSD fails to meet the established criteria, an LCS must be analyzed to determine if the sample is exhibiting matrix interference or if the system is out of control.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe or alternately be performed by the autosampler.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 5  $\mu\text{L}$  of the combined surrogate/internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards are mixed and added as a single spiking solution. The addition of 5  $\mu\text{L}$  of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50  $\mu\text{g/L}$  of each surrogate standard. The addition of 5  $\mu\text{L}$  of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50  $\mu\text{g/kg}$  of each standard. If a more sensitive mass spectrometer is employed to achieve lower detection levels more dilute surrogate and internal standard solutions may be required. The sample and spike amounts may vary dependent upon the equipment requirements however, the spike levels remain the same as prescribed.

7.5.9 Add 5  $\mu\text{L}$  of the matrix spike solution to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50  $\mu\text{g/L}$  of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2  $\mu\text{L}$  into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2  $\mu\text{L}$  injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

---

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within  $\pm 0.06$  RRT units of the RRT of the internal standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.



7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

- (1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

### 7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing



internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas  $A_x$  and  $A_{is}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

## 8.0 Quality Control:

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. The maintain records to document the quality of the data generated according to laboratory's Quality Manual.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff is trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis – Documentation is required demonstrating the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and



reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) must be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard is to be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

## 9.0 Method Performance:

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. Water and Solid MDLs were calculated using the appropriate preparation methods and are presented in Table 1.



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

The following chart outlines the quality control and corrective action requirements for this procedure. Where re-extraction or re-analysis is indicated, this must be performed within the applicable holding time.

Quality Control Problem	Corrective Action
BFB ion abundance criteria outside acceptance windows (see Table 2)	Retune and recalibrate the GC/MS. It may be necessary to clean the ion source and/or the quadrupoles before retuning.
Initial calibration SPCC's RF's outside acceptance window	<p>Recalibrate the GC/MS. It may be necessary to clean the source, change the column, or take other corrective action before recalibrating.</p> <ul style="list-style-type: none"> <li>• Low response for chloromethane usually indicates that the purge rate is too fast</li> <li>• Low response for bromoform may be caused by               <ul style="list-style-type: none"> <li>• Purge rate too slow</li> <li>• Cold spots or active sites in the transfer lines</li> <li>• Relative abundance of the high m/z ions (174 and 176) too low</li> </ul> </li> <li>• Low response for 1,1,2,2-tetrachloroethane and/or 1,1-dichloroethane usually indicates contaminated transfer lines or active sites in the trap materials</li> </ul>
Initial calibration CCC's %RSD outside acceptance window	Check for system leaks or active sites and re-calibrate the instrument.
Continuing calibration RF and/or %D outside acceptance window	See the corrective action above for the initial calibration
Continuing calibration internal standard retention time not within $\pm 30$ seconds of the mid-point level internal standards of the most recent initial calibration	Check the GC system for malfunction. Perform a new initial or continuing calibration and reanalyze all samples analyzed with the failed standard.
Continuing calibration internal standard areas not within -50% to + 100% of the mid-point level internal standards of the most recent initial calibration	<ul style="list-style-type: none"> <li>• Check for calculation error. If an error is found, correct the calculations and verify that the recalculated responses meet the acceptance criteria.</li> <li>• Check for mass spectrometer malfunction. If a problem is found, correct the malfunction, retune, recalibrate, and reanalyze the affected samples.</li> </ul>
Method blank contamination $\geq$ QL	The method blank and all associated samples must be reanalyzed.
Method blank surrogate recovery outside acceptance window	Reanalyze the method blank and all associated samples.
Interference with primary quantitation ion	Use a secondary quantitation ion listed in Table 1 and document problem in Case Narrative.
Quantitation ion saturates detector	Standards: Adjust the analytical system to eliminate saturation while maintaining sufficient sensitivity to meet QL requirements. Retune and perform a new initial calibration.
	Field and quality control samples: Dilute and reanalyze.
Quality Control Problem	Corrective Action



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

<p>Sample internal standard retention times not within <math>\pm 30</math> seconds the mid-point level internal standards of the most recent initial calibration</p>	<p>Check the system for malfunction and re-analyze the affected samples.</p>
<p>Sample internal standard areas not within a factor of (-50% to +100%) of associated continuing calibration standard</p>	<p>All analyses: Check for calculation error. If an error is found, correct the calculations and verify that the recalculated responses meet the acceptance criteria.</p>
	<p>All analyses: Check for mass spectrometer malfunction. If a problem is found, correct the malfunction, retune, recalibrate, and reanalyze the affected samples.</p>
	<p>Field samples and MS/MSD: Reanalyze the sample. If the internal standard area criteria are met, the original analysis is invalid and only the reanalysis is to be submitted. If the criteria are not met, matrix interference is assumed and both analyses are to be submitted. NOTE: For the sample selected for MS/MSD, the MS and MSD serve as reanalysis for this purpose. All three analyses must confirm or reject the matrix interference assumption; otherwise, reanalysis is required to resolve the situation. Submit only the valid analyses. Document matrix interference in the Case Narrative.</p>
<p>RRT for surrogate not within <math>\pm 0.06</math> RRT units of RRT in the associated calibration standard</p>	<p>Check the GC system for malfunction. Reanalyze all failed standards, field samples, and quality control samples.</p>
<p>Surrogate recovery outside acceptance window</p>	<ul style="list-style-type: none"> <li>• Check for calculation error. If an error is found, correct the calculations and verify that the recalculated recoveries meet the acceptance criteria.</li> <li>• If no calculation error is found, reanalyze the sample. If the surrogate recovery criteria are met, the original analysis is invalid and only the reanalysis is to be submitted. If the criteria are not met, matrix interference is assumed. NOTE: For the sample selected for MS/MSD, the MS and MSD serve as re-extractions and re-analyses for this purpose. All three analyses must confirm or reject the matrix interference assumption; otherwise, re-extraction and reanalysis is required to resolve the situation. Submit only the valid analyses. Document matrix interference in the Case Narrative.</li> </ul>



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

**Table 1:**

CAS No.	Compound	Primary Ion	Secondary Ion	Water PQL	Soil PQL
				ug/L	ug/kg
67-64-1	Acetone	43	58	10	20
107-02-8	Acrolein	56	55	20	20
107-13-1	Acrylonitrile	53	52	20	20
71-43-2	Benzene	78	77	5	5
108-86-1	Bromobenzene	156	77	5	5
74-97-5	Bromochloromethane	128	130	5	5
75-27-4	Bromodichloromethane	83	85	5	5
75-25-2	Bromoform	173	171	5	5
74-83-9	Bromomethane	94	96	10	10
78-93-3	2-Butanone (MEK)	43	72	10	10
104-51-8	n-Butylbenzene	134	91	5	5
135-98-8	sec-Butylbenzene	105	134	5	5
98-06-6	tert-Butylbenzene	134	91	5	5
75-15-0	Carbon disulfide	76		5	5
56-23-5	Carbon tetrachloride	117	119	5	5
108-90-7	Chlorobenzene	112	77	5	5
75-00-3	Chloroethane	64	66	10	10
110-75-8	2-Chloroethyl vinyl ether	63	106	5	5
67-66-3	Chloroform	83	85	5	5
74-87-3	Chloromethane	50	52	10	10
95-49-8	2-Chlorotoluene	91	126	5	5
106-43-4	4-Chlorotoluene	91	126	5	5
96-12-8	1,2-Dibromo-3-chloropropane	75	155	5	5
124-48-1	Dibromochloromethane	129	127	5	5
106-93-4	1,2-Dibromoethane (EDB)	109	107	5	5
74-95-3	Dibromomethane	93	95	5	5
95-50-1	1,2-Dichlorobenzene	146	111	5	5
541-73-1	1,3-Dichlorobenzene	146	111	5	5
106-46-7	1,4-Dichlorobenzene	146	111	5	5
75-71-8	Dichlorodifluoromethane	85	87	10	10
75-34-3	1,1-Dichloroethane	63	65	5	5
107-06-2	1,2-Dichloroethane	62	64	5	5
75-35-4	1,1-Dichloroethene	96	61	5	5
156-59-4	cis-1,2-Dichloroethene	96	61	5	5
156-60-5	trans-1,2-Dichloroethene	96	61	5	5
78-87-5	1,2-Dichloropropane	63	62	5	5
142-28-9	1,3-Dichloropropane	76	78	5	5
590-20-7	2,2-Dichloropropane	77	97	5	5
563-58-6	1,1-Dichloropropene	75	110	5	5
10061-01-5	cis-1,3-Dichloropropene	75	77	5	5
10061-02-6	trans-1,3-Dichloropropene	75	77	5	5
100-41-4	Ethylbenzene	91	106	5	5
87-68-3	Hexachlorobutadiene	225	227	5	5



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

CAS No.	Compound	Primary Ion	Secondary Ion	PQL	
				Water ug/L	Soil ug/kg
591-78-6	2-Hexanone	58	43	10	10
98-82-8	Isopropylbenzene	105	120	5	5
99-87-6	4-Isopropyltoluene	119	134	5	5
1634-04-4	Methyl tert-butyl ether (MTBE)	73	57	5	5
108-10-1	4-Methyl-2-pentanone (MIBK)	43	58	10	10
75-09-2	Methylene chloride	84	86	5	5
91-20-3	Naphthalene	128	129	5	5
103-65-1	n-Propylbenzene	91	120	5	5
100-42-5	Styrene	104	78	5	5
96-18-4	1,2,3-Trichloropropane	110	77	5	5
630-20-6	1,1,1,2-Tetrachloroethane	133	131	5	5
79-34-5	1,1,2,2-Tetrachloroethane	83	131	5	5
127-18-4	Tetrachloroethene (PCE)	164	166	5	5
108-88-3	Toluene	92	91	5	5
87-61-6	1,2,3-Trichlorobenzene	180	182	5	5
120-82-1	1,2,4-Trichlorobenzene	180	182	5	5
71-55-6	1,1,1-Trichloroethane	97	99	5	5
79-00-5	1,1,2-Trichloroethane	97	99	5	5
79-01-6	Trichloroethene (TCE)	130	132	5	5
75-69-4	Trichlorofluoromethane	101	103	10	10
76-13-1	Trichlorotrifluoroethane	151	153	10	10
95-63-6	1,2,4-Trimethylbenzene	105	120	5	5
108-67-8	1,3,5-Trimethylbenzene	105	120	5	5
108-05-4	Vinyl acetate	43	86	10	10
75-01-4	Vinyl chloride	62	64	10	10
95-47-6	o-Xylene	91	106	5	5
133-02-07	m,p-Xylenes	91	106	5	5
107060-07-0	1,2-Dichloroethane-d <sub>4</sub> (S)	67	65	5	5
462-06-6	Fluorobenzene (IS)	96	77	5	5
3114-55-4	Chlorobenzene-d <sub>5</sub> (IS)	117		5	5
3855-82-1	1,4-Dichlorobenzene-d <sub>4</sub> (IS)	152		5	5
353-55-9	Dibromofluoromethane (S)	113		5	5
2037-26-5	Toluene-d <sub>8</sub> (S)	98		5	5
460-00-4	4-Bromofluorobenzene (S)	176	174	5	5



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

**Table 2: Ion Abundance Criteria for BFB**

Mass (m/z)	Required Relative Abundance
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	Base peak, 100% relative abundance
96	5% to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5% to 9% of mass 174
176	Greater than 95% of mass 174 but less than 101% of mass 174
177	5% to 9% of mass 176

**Table 3: Acceptance Criteria for Initial and Continuing Calibration**

Compound		Initial Calibration		Continuing Calibration	
		Min. Avg. RF	Max. % RSD	Min. RF	Max. % D
Bromoform	SPCC	0.100	---	0.100	---
Chlorobenzene	SPCC	0.300	---	0.300	---
Chloroform	CCC	---	30	---	20
Chloromethane	SPCC	0.100	---	0.100	---
1,1-Dichloroethane	SPCC	0.100	---	0.100	---
1,1-Dichloroethene	CCC	---	30	---	20
1,2-Dichloropropane	CCC	---	30	---	20
Ethylbenzene	CCC	---	30	---	20
1,1,2,2-Tetrachloroethane	SPCC	0.300	---	0.300	---
Toluene	CCC	---	30	---	20
Vinyl chloride	CCC	---	30	---	20



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

**Table 4: 8260 Second Source Spike Standards**

Compound	Initial Stock Concentration	Initial Amount Stock Used	Final Volume	Final Concentration
<b>8260 SPIKE MIX 1</b>				
MTBE	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm
Vinyl Acetate	2000ppm	100ul	1000ul	200ppm
Carbon Disulfide	2000ppm	100ul	1000ul	200ppm
2-CEVE	2000ppm	100ul	1000ul	200ppm
54 Comp.	2000ppm	100ul	1000ul	200ppm
Acrylonitrile	20,000ppm	10ul	1000ul	200ppm
Methacrylonitrile	2000ppm	100ul	1000ul	200ppm
<b>8260 SPIKE MIX 2</b>				
1,1,2 TCTFE	2000ppm	100ul	1000ul	200ppm
Diethyl ether	5000ppm	40ul	1000ul	200ppm
6 Comp. Gas Mix	2000ppm	100ul	1000ul	200ppm
Tetrahydrofuran	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm



## Volatile Organics by GC/MS

Method 8260B

Revision 2.21

Effective: April 24, 2005

**Table 5: 8260B Calibration Standards Mix**

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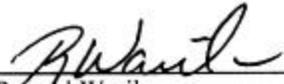
Compound	Initial Stock Concentration	Initial Amount Stock Used	Final Volume	Final Concentration
<b>MIX 1</b>				
MTBE	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm
Acrolein	20,000ppm	10ul	1000ul	200ppm
Acrylonitrile	20,000ppm	10ul	1000ul	200ppm
Carbon Disulfide	20000ppm	10ul	1000ul	200ppm
Vinyl Acetate	20,000ppm	10ul	1000ul	200ppm
Diethyl ether	20,000ppm	10ul	1000ul	200ppm
<b>MIX 2</b>				
2-CEVE	1000ppm	200ul	1000ul	200ppm
1,1,2 TCTFE	1000ppm	100ul	1000ul	200ppm
Tetrahydrofuran	20,000ppm	10ul	1000ul	200ppm
Methacrylonitrile	20,000ppm	10ul	1000ul	200ppm
54 Component	2000ppm	100ul	1000ul	200ppm
6 Comp. Gas Mix	2000ppm	100ul	1000ul	200ppm

---



**Cyanide (Total)**  
SM 4500-CN-C+E/9012A

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

References:

Test Methods for Evaluating Solid Waste, SW-846, Revision 3, December 1996, Method 9012A.

Standard Methods for the Evaluation of Water and Wastewater, 20<sup>th</sup> Edition, 1998, method 4500-CN-C+E.

**I. Scope and Application:**

Analyte: Cyanide  
Matrix: Water, wastewater, soil, sludge  
Regulation: NPDES, CWA, RCRA

**II. Summary of Method:**

Cyanide in the form of hydrocyanic acid (HCN) is released from samples containing cyanide by means of a reflux-distillation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated colorimetry. In the colorimetric determination, the cyanide is converted to cyanogen chloride (CNCL) by reaction with Chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. Color is formed on the addition of pyridine-barbituric acid reagent and read against a curved plotted with standards containing the same concentration of sodium hydroxide as the scrubber.

**III. Important Notes:**

Sulfides, nitrates, and nitrites interfere with the cyanide determination. A drop of the sample on lead acetate paper will turn black if the sample needs to be treated for sulfide removal with cadmium carbonate. A method detection limit of 0.02 mg/L is required. The procedure is also modified to perform cyanide determination on solids. All solids are reported on a dry weight basis.



#### **IV. Sample Collection, Preservation and Storage:**

Samples should be collected in plastic 1-liter bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

Samples must be preserved with 2 mL of 10 N sodium hydroxide per liter of sample (pH>12) at the time of collection.

Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples must be stored at 4°C.

Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper; a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample volume.

Aldehydes convert cyanide to cyanohydrin. Longer contact times between cyanide and the aldehyde and the higher ratios of aldehyde to cyanide both result in increasing losses of cyanide that are not reversible during analysis. If the presence of aldehydes is suspected, stabilize with NaOH at time of collection and add 2 mL 3.5% ethylene diamine solution per 100 mL of sample.

#### **V. Distillation Procedure:**

Place 500 mL of an aqueous sample, a sample diluted to 500ml, or 10 grams of a soil sample ( diluted to 500 mL with DI H<sub>2</sub>O ) into a 1000ml distillation flask.

Add 50 mL of 1.25N NaOH to the gas scrubber (trap).

Connect the boiling flask, condenser, gas scrubber and vacuum trap. Check all joints to ensure an air tight seal.

Turn on the cooling water to the condensers.

Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately two bubbles of air per second enter the boiling flask through the air inlet tube.

Repeat the above steps for all samples.

Allow the vacuum to draw for 5 minutes.

Add 50ml of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

Slowly add 50 mL of 18N H<sub>2</sub>SO<sub>4</sub> through air inlet. Rinse the tube with DI H<sub>2</sub>O and allow vacuum draw for three minutes.



Add 20 ml of 2.5M magnesium chloride through the air inlet and wash the inlet tube with a stream of DI H<sub>2</sub>O.

Heat the solution to boiling adjusting the vacuum valves as necessary. Reflux for 1 hour. Turn off the heat and continue the airflow while cooling for at least 15 minutes.

Turn off all vacuum valves. Disconnect all reflux flasks to the gas scrubber.

Transfer the solution from the scrubber into a 250 ml volumetric flask. Rinse the scrubber into the flask and dilute to volume with DI H<sub>2</sub>O

Remove glassware and clean before re-use.

## VI. Definitions:

**Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

**Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

**Field Duplicates (FD)** -- Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

**Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

**Laboratory Fortified Blank (LFB)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. **The LFB is analyzed exactly like a sample**, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

**Laboratory Fortified Sample Matrix (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

**Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

**Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear (correlation coefficient >0.995).

**Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.



**Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

**Performance Evaluation Sample (PE)** -- A solution of method analytes distributed by the Quality Assurance Research Division (QARD), Environmental Monitoring Systems Laboratory (EMSL-Cincinnati), U. S. Environmental Protection Agency, Cincinnati, Ohio, to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used by QARD to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.

**Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

**Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

## VII. Interferences

For strong acid dissociable and weak acid dissociable cyanide, non-volatile interferences are eliminated or minimized by the distillation procedure.

Sulfides adversely affect the colorimetric procedure. If a drop of the sample on lead acetate test paper indicates the presence of sulfide, treat 25 mL more of the stabilized sample (pH >12) than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiocyanide, thiosulfate, and sulfide. Multiple interferences may require the analysis of a series of laboratory fortified sample matrices (LFM) to verify the suitability of the chosen treatment. See Standard Methods section 412A and for details of preliminary sample treatment to remove volatile interferences see Standard Methods 18<sup>th</sup> ed. Sources Section 16.

## VIII. Safety:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.



The following chemicals have the potential to be highly toxic or hazardous. For detailed explanations consult the MSDS.

Cyanide  
Sulfuric Acid  
Pyridine  
Sodium Hydroxide

## **IX. Equipment and Supplies:**

Balance – analytical, capable of accurately weighing to the nearest 0.0001 g.

Glassware – Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

Autosampler

Multichannel proportioning pump

Reaction unit or manifold

Colorimetric detector

Data system

Heating Unit

Macro Distillation Glassware Setup (see figure 1)

## **X. Reagents and Standards:**

### **REAGENTS**

**Reagent Water:** Distilled or deionized water, free of the analyte of interest. ASTM Type II or equivalent.

**Ascorbic acid:** Crystal (CASRN-50-81-7)

**Sodium Hydroxide Solution, 1.25 N:** Dissolve 50 g NaOH (CASRN-1310-73-2) in about 600 mL of reagent water in a 1 L beaker. (**Caution:** this is an exothermic reaction producing heated fumes, therefore it must be performed in a hood.) Allow the solution to cool, transfer to a 1 liter volumetric flask, and dilute to the mark with reagent water.

**Sulfamic Acid (H<sub>2</sub>NSO<sub>3</sub>H) Solution, 0.4 N:** Dissolve 40 g H<sub>2</sub>NSO<sub>3</sub>H in reagent water in a 1-liter volumetric flask.

**Sulfuric Acid, 18 N:** Slowly add 500 mL of conc. H<sub>2</sub>SO<sub>4</sub> (CASRN-5329-14-6) to 500 mL of reagent water.



## XI. PREPARATION OF STANDARDS

### 1. Standard 1. Stock Standard 1250 mg CN/L

In a **1 L** volumetric flask, dissolve **2.0 g potassium hydroxide (KOH)** in approximately **500 mL water**. Add **3.129 g potassium cyanide (KCN)**. CAUTION: KCN IS HIGHLY TOXIC. AVOID INHALATION OF DUST OR CONTACT WITH THE SOLID OR SOLUTIONS. Dilute to the mark with DI water and invert three times. Prepare fresh weekly **or** re- standardize weekly.

### 2. Standard 2. Stock Standard 12.5 mg /L CN<sup>-</sup> in Deionized Water

In a **100 mL** volumetric flask add **1 mL of Standard 1** (1250 mg /L). Dilute to the mark with DI water and invert to mix. Prepare fresh weekly.

## DISTILLED STANDARDS

(Standards that will be taken through the complete distillation procedure with the samples to demonstrate distillation efficiency.)

These standards will be distilled

Working Standards (prepare daily)						
Concentration mg /L CN <sup>-</sup>	0.500	0.300	0.100	0.050	0.010	0.005
By Volume						
Volume (mL) of <b>stock standard 2</b> Diluted to 500 mL with DI water	20.0	12.0	4.0	2.0	---	---
Volume (mL) of <b>standard A</b> diluted to 500 mL with DI water	---	---	---	---	10.0	5.0

## NON-DISTILLED STANDARDS

(Standards that are prepared at a 2:1 concentration, and analyzed directly to offset the concentration step performed during a distillation.)



### Distillation Matrix (0.25 M NaOH)

**By Volume:** In a 1 L volumetric flask containing approximately 600mL DI water, add 10 g of sodium hydroxide (NaOH). Dilute to the mark and invert to mix.

### Standard 2. Stock Standard 12.5 mg /L CN<sup>-</sup> in Distillation Matrix

In a 100 mL volumetric flask add 1 mL of Standard 1 (1250 mg CN<sup>-</sup>/L). Dilute to the mark with 0.25 M NaOH and invert to mix. Prepare fresh weekly.

These standards **will not** be distilled

Working Standards (prepare daily)						
<i>Actual Concentration mg/L CN<sup>-</sup></i>	1.000	0.600	0.200	0.100	0.020	0.010
Label as: mg/L CN <sup>-</sup>						
See note below	0.500	0.300	0.100	0.050	0.010	0.005

By Volume

Volume (mL) of <b>standard 2</b> diluted to 250 mL with 0.25 M NaOH	20.0	12.0	4.0	2.0	---	---
Volume (mL) of <b>standard A</b> diluted to 250 mL with 0.25 M NaOH	---	---	---	---	5.0	2.5

Note: Following the macro distillation procedure, the cyanide ion present in the original samples is concentrated by a factor of two from the distillation procedure. The initial sample volume is 500 mL; however, the cyanide which has distilled over into the 1.25 M NaOH absorbing solution is diluted to a 250 mL final volume. Because the non-distilled standards are not carried through the distillation procedure, they are not concentrated by factor of two. To compensate for this factor, the standard concentrations are doubled, but still labeled as the expected concentrations.

## XII. Automated Colorimetric Determination

Set up manifold per manufacturers specifications.



Set temperature of the heating bath to 150°C.

Allow the colorimeter and recorder to warm up for 30 minutes.

Place appropriate standards in the sampler in order of decreasing concentration. Complete loading of sampler tray with unknown samples.

Refer to the Lachat manual for determination of cyanide for setup and operation.

### **XIII. Calculations:**

#### **Aqueous Samples**

Direct reading in mg/L from the Lachat data system.

#### **Solid samples**

Proceed with analyzing the aqueous extract from the soil preparation. Obtain results in mg/L.

Refer to the following soil calculation:

$$\text{Total cyanide mg/kg (wet weight)} = \frac{(x)(y)(1000)}{(g)}$$

$$\text{Total cyanide mg/kg (dry weight)} = \frac{(x)(y)(1000)}{(g)(\%S)}$$

where:

x = cyanide concentration in sediment digest, mg/L

y = final volume of sediment digest, L

g = wet weight of sample digest, g

%S = percent of solids in sediment sample as a decimal fraction

### **XIV. Quality Control:**

The minimum requirements for this method consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

#### **Initial Demonstration of Performance:**

The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by 10%, linearity must be reestablished. If any portion



of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

**Quality Control Sample (QCS)** -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

**Method Detection Limit (MDL)** -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units.

Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]

S = standard deviation of the replicate analyses

MDLs should be determined every six months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

#### **Assessing Laboratory Performance:**

**Laboratory Reagent Blank (LRB)** -- The laboratory must analyze at least one LRB with each batch of 20 samples or less. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

**Laboratory Fortified Blank (LFB)** -- At least one LFB with each batch of 20 samples or less. Calculate accuracy as percent recovery (Section 9.4.2). If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

The LFB analyses data must be used to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery ( $\bar{x}$ ) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \bar{x} + 3S$$

$$\text{LOWER CONTROL LIMIT} = \bar{x} - 3S$$

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to 10 new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish



an on-going precision statement for the level of concentrations included in the LFB. This data must be kept on file and be available for review.

At least quarterly, replicates of LFBs should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.

**Instrument Performance Check Solution (IPC)** -- For all determinations the IPC (a mid-range check standard) must be analyzed and a calibration blank immediately following daily calibration, and after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

#### **Assessing Analyte Recovery and Data Quality:**

**Laboratory Fortified Sample Matrix (LFM)** -- The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples.

In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The added analyte concentration should be the same as that used in the laboratory fortified blank. If the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_s - C}{S} \times 100$$

where, R = percent recovery

C = fortified sample concentration

C<sub>s</sub> = sample background concentration

s = concentration equivalent of analyte added to sample

Until sufficient data becomes available (usually a minimum of 20-30 analysis), assess laboratory performance against recovery limits of 90-110%. When sufficient internal performance data becomes available, develop control limits from percent mean recovery and the standard deviation of the mean recovery.

If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.



Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

## **XV. Pollution Prevention**

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

**Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.**

## **XVI. Waste Management:**

All waste is handled in accordance with Premier Laboratory's Chemical Hygiene Plan, which is made available to all employees and interested parties.



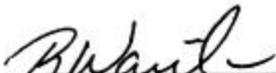
**Semi-Volatiles**  
Method 8270C  
Revision 1.2  
December 19, 2001

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## **Semi volatile Organics Analysis**

### **SW-846 8270C**

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

#### **REFERENCE:**

Test Methods for Evaluating Solid Waste, SW-846, Revision 3, December 1996, method 8270C.

#### **Applicability:**

Analyte: Semivolatile organic compounds listed in Table 1  
Matrix: extracts from solid waste matrices, soils, and water samples  
Regulation: RCRA, ECRA

#### **Important Notes:**

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross contamination.

#### **Apparatus And Materials:**

1. Gas chromatograph/mass spectrometer/data system with the most recent version of the EPA/NIST Mass Spectral Library.

Previous revision: 12/2004-No changes  
Next revision: 12/2005



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

2. Chromatography column: DB-5, 30 m x 0.25-mm I.D.
3. 10 $\mu$ L, 25 $\mu$ L, 100 $\mu$ L, and 1000 $\mu$ L syringes.

### REAGENTS:

1. Stock standard solutions: 2000mg/L. Purchased from commercial suppliers of certified standards.
2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at  $-10^{\circ}\text{C}$  or less and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

3. Internal standard solutions: 4000 ng/ $\mu$ L. The required internal standards are 1,4-dichlorobenzene- $d_4$ , naphthalene- $d_8$ , acenaphthene- $d_{10}$ , phenanthrene- $d_{10}$ , chrysene- $d_{12}$ , and perylene- $d_{12}$ . Each 1-mL sample extract undergoing analysis must be spiked with 10  $\mu$ L of the internal standard solution, resulting in a concentration of 40 ng/ $\mu$ L of each internal standard. Store at  $-10^{\circ}\text{C}$  or less when not being used.
- 4.
5. GC/MS tuning standard: 50 ng/ $\mu$ L. A methylene chloride solution of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/ $\mu$ L each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at  $-10^{\circ}\text{C}$  or less when not being used.
6. Calibration standards: Calibration standards are prepared by diluting commercial standards to provide concentrations of 5, 10, 20, 40, 50, 80, and 100 ng/ $\mu$ L. Each 1 -mL aliquot of calibration standard must be spiked with 10  $\mu$ L of the internal standard solution prior to analysis. All standards should be stored at  $-10^{\circ}\text{C}$  to  $-20^{\circ}\text{C}$  and should be freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard (50 ng/ $\mu$ L) should be prepared weekly and stored at  $4^{\circ}\text{C}$ .
7. Surrogate standards: The required surrogate standards are phenol- $d_6$ , 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene- $d_5$ , 2-fluorobiphenyl, and p-terphenyl- $d_{14}$ . These are added to the samples prior to extraction and must be included in the initial and continuing calibrations at the same concentrations as target analytes.



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

## Procedure:

### GC/MS operating conditions

Mass range:	35 to 500 amu
Scan time:	1 second or less/scan
Initial column temperature and hold time:	40°C for 1 minute
Column temperature program:	50°C to 115°C at 25°C/minute 115°C to 330°C at 10°C/minute, hold for 1.5 minutes
Injector temperature:	270°C
Transfer line temperature:	290°C
Source temperature:	According to manufacturer specifications
Injector:	Grob-type, splitless
Sample volume:	1.0 µL
Carrier gas:	Helium

## Tuning

Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 50-ng injection of DFTPP. Analyses may not begin until all these criteria are met. Background subtraction must be straightforward and designed only to eliminate column bleed or instrument background ions. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6 to 12 inches of the capillary column.

## Initial Calibration:

1. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion, *i.e.*, for 1,4-dichlorobenzene- $d_4$  use  $m/z$  152 for quantitation.
2. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). A

Previous revision: 12/2004-No changes

Next revision: 12/2005



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

set of at least 5 calibration standards is required. Calculate response factors (RF's) for each compound as follows:

$$RF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

where:  $A_X$  = Area of the characteristic ion for the compound being measured

$A_{IS}$  = Area of the characteristic ion for the specific internal standard

$C_X$  = Concentration of the compound being measured ( $\mu\text{g/L}$ )

$C_{IS}$  = Concentration of the specific internal standard ( $\mu\text{g/L}$ )

### Calibration Verification:

3. SPCC: The relative retention times of each compound in each calibration run must agree within 0.06 relative retention time units. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCC's) are: N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum acceptable average RF for these compounds (SPCC's) is 0.050.
4. Calibration Check Compounds (CCC): Calculate the average RF and percent relative standard deviation (%RSD) for each compound. If the RSD of any target analytes is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD is > 15% for any target analyte, an average RF can not be used. Refer to section 7.0 in Method 8000 for additional calibration options, such as linear or quadratic regression calculations. However, the RSD for each individual CCC (see Table 3) must be less than or equal to 30%.

### Continuing Calibration:

1. Prior to analysis of samples, the GC/MS tuning standard must be analyzed for each 12 hour clock. A 50-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 2. These criteria must be demonstrated during each 12-hour shift.
2. A calibration standard at 50 ng/ $\mu\text{L}$  concentration containing all semivolatile analytes, including all required surrogates, must be performed every 12-hours during analysis. Compare the response factor data from the standards every 12 hours with the average response factor from the initial calibration for a specific instrument for the SPCC, CCC, and internal standards criteria list below.

Previous revision: 12/2004-No changes

Next revision: 12/2005



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

3. System Performance Check Compounds (SPCC's): A system performance check must be made during every 12-hour shift. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCC's is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.
4. Calibration Check Compounds (CCC's): After the system performance check is met, CCC's listed in Table 3 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\text{RF}_I - \text{RF}_C}{\text{RF}_I} * 100$$

where:  $\text{RF}_I$  = average response factor from initial calibration.

$\text{RF}_C$  = response factor from current verification check standard.

Calculate the percent % Drift :

$$\% \text{ Drift} = \frac{\text{Calculated concentration \& Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

Where:

the calculated concentration is determined using the mean calibration factor or response factor from the initial calibration.

the theoretical concentration is the concentration at which the standard was prepared.

If the percent difference or drift for any compound is greater than 20%, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criteria is not met (>20% difference or drift) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCC's could affect this criteria. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion must be met before sample analysis can begin. In the absence of a CCC in the calibration standards for a specific project all compounds must meet the 20% drift or difference criteria.



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

5. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the mid-point calibration level of the last initial calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.
6. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the mid-point calibration level of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

### **GC/MS Analysis:**

1. Spike the 1-mL extract obtained from sample preparation with 10  $\mu\text{L}$  of the internal standard solution just prior to analysis.
2. Analyze a 1.0- $\mu\text{L}$  injection of the 1-mL extract. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/ $\mu\text{L}$  of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

### **Qualitative Analysis**

1. Target analytes are identified by comparison of the sample mass spectrum obtained on the same GC/MS system. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.
  - (1) The sample component RRT must compare within  $\pm 0.06$  RRT units of the RRT of the standard component in the daily calibration standard. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.
  - (2) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of these ions must agree within 30% between the standard and sample spectrum. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent).

### **Quantitative Analysis**

Previous revision: 12/2004-No changes

Next revision: 12/2005



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

1. When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique.
2. Calculate the concentration of each identified analyte in the sample as follows:

Water:

$$C = \frac{A_X * I_S * V_T}{A_{IS} * RF * V_O * V_I}$$

where: C = sample concentration ( $\mu\text{g/L}$ )

$A_X$  = area of characteristic ion for compound being measured

$I_S$  = amount of internal standard injected (ng)

$V_T$  = volume of total extract, taking into account dilutions ( $\mu\text{L}$ )

$A_{IS}$  = area of characteristic ion for the internal standard

RF = response factor for compound being measured

$V_O$  = volume of water extracted (mL)

$V_I$  = volume of extract injected ( $\mu\text{L}$ )

Soil, Sediment, Sludge, or Waste:

$$C = \frac{A_X * I_S * V_T}{A_{IS} * RF * V_I * W_S * D}$$

where: C = sample concentration ( $\mu\text{g/kg}$ )

$A_X$ ,  $I_S$ ,  $V_T$ , RF and  $V_I$  = same as for water.

$W_S$  = weight of sample extracted (g)

$$D = \frac{100 - \% \text{ Moisture}}{100}, \text{ (} = 1 \text{ for wastes)}$$

3. Where applicable, an estimate of concentration for non-calibrated components in the sample should be made. The formulas given above should be used with the following modifications: The areas  $A_X$  and  $A_{is}$  should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The resulting concentration should be reported as estimated.



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

4. If the response for any analyte exceeds the calibration range of the instrument, dilute the extract so that the response of the analyte with the greatest concentration falls within the upper half of the calibration range.
5. When the extracts are not being used for analysis, store them at  $< -10^{\circ}\text{C}$ . All initial and reanalysis of sample extracts must be performed within 40 days of extraction.

### Quality Control:

1. Refer to Chapter One of SW-846 and method 8000 for further guidance in the application of quality control procedures to demonstrate the ability to generate data of acceptable accuracy and precision for the target analytes specified.
2. Method Blank Analysis
  - a. A method blank must be prepared and analyzed at a frequency of 1 per batch of samples extracted per matrix, not to exceed 20 samples per batch.
  - b. The concentration of each target compound found in the blank must be less than the required quantitation limit for the project.
  - c. A solvent blank should be analyzed whenever a new lot of solvent is introduced to check for potential contamination.
3. Matrix Spike/ Laboratory Control Spike Analysis
  - a. A matrix spike and matrix spike duplicate pair must be extracted and analyzed at a frequency of 1 per 20 samples extracted per matrix.
  - b. One MS/MSD pair must be extracted and analyzed at least every 30 days for each matrix.
  - c. The laboratory must generate MS/MSD recovery data control charts at least annually for each matrix.
  - d. The control charts will be used to define acceptable recovery ranges of spike compounds.
  - e. If one or more compounds are outside of the control limits then an LCS (QC check standard) must be analyzed to check for matrix interference.
  - f. The LCS must meet the acceptance criteria for those compounds that failed acceptance criteria in the MS/MSD.
  - g. Matrix spike recovery calculation:

$$\% \text{ Recovery} = \frac{\text{spiked sample result} - \text{sample result}}{\text{spike added}} \times 100$$

- h. LCS recovery calculation:



$$\% \text{ Recovery} = \frac{\text{LCS sample result}}{\text{spike added}} \times 100$$

#### 4. Surrogate Recoveries

- a. Surrogate spike recovery limits must be generated and updated at least annually for each matrix through the use of control charts.
- b. If the recovery of one or more compounds is outside of the control limits, the sample must be re-analyzed. If after re-analysis the recovery is still not within the limits the sample must be re-extracted and re-analyzed. If the re-extracted sample surrogates do not meet the criteria, then the matrix interference problem must be noted in the project case narrative or non-conformance summary.

**TABLE 1**

CAS No.	Compound	Primary Ion	Secondary Ion	Est. Quantitation Limits	
				Water (ug/L)	Soil(ug/kg)
103-33-3	Azobenzene	77	182	5	167
83-32-9	Acenaphthene	154	153	5	167
208-96-8	Acenaphthylene	164	162	5	167
62-53-3	Aniline	93	66	10	330
120-12-7	Anthracene	178	179	5	167
92-87-5	Benzidine	184	92	20	670
56-55-3	Benzo[a]anthracene	228	229	5	167
50-32-8	Benzo[a]pyrene	252	253	5	167
205-99-2	Benzo[b]fluoranthene	252	253	5	167
191-24-2	Benzo[g,h,i]perylene	276	138	5	167
207-08-9	Benzo[k]fluoranthene	252	253	5	167
65-85-0	Benzoic acid	105	122	25	830
100-51-6	Benzyl alcohol	108	79	10	330
85-68-7	Benzyl butyl phthalate	149	91	5	167
111-91-1	Bis(2-chloroethoxy)methane	93	95	5	167
111-44-4	Bis(2-chloroethyl)ether	93	63	5	167
108-60-1	Bis(2-chloroisopropyl)ether	45	121	10	330
117-81-7	Bis(2-ethylhexyl)phthalate	149	167	5	167
101-55-3	4-Bromophenyl phenyl ether	248	250	5	167
59-50-7	4-Chloro-3-methylphenol	107	144	5	167
106-47-8	4-Chloroaniline	127	129	10	330
91-58-7	2-Chloronaphthalene	162	164	5	167
95-57-8	2-Chlorophenol	128	64	5	167
7005-72-3	4-Chlorophenyl phenyl ether	204	206	5	167

Previous revision: 12/2004-No changes

Next revision: 12/2005



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

---

218-01-9	Chrysene	228	226	5	167
84-74-2	Di-n-butyl phthalate	149	150	5	167
117-84-0	Di-n-octyl phthalate	149	167	5	167
53-70-3	Dibenz[a,h]anthracene	278	139	5	167
132-64-9	Dibenzofuran	168	139	10	330
95-50-1	1,2-Dichlorobenzene	146	148	5	167
541-73-1	1,3-Dichlorobenzene	146	148	5	167
106-46-7	1,4-Dichlorobenzene	146	148	5	167
91-94-1	3,3-Dichlorobenzidine	252	254	5	167
120-83-2	2,4-Dichlorophenol	162	164	5	167
84-66-2	Diethyl phthalate	149	177	5	167
131-11-3	Dimethyl phthalate	163	194	5	167
105-67-9	2,4-Dimethylphenol	122	107	5	167
51-28-5	2,4-Dinitrophenol	184	63	5	167
121-14-2	2,4-Dinitrotoluene	165	89	5	167
606-20-2	2,6-Dinitrotoluene	165	89	5	167
206-44-0	Fluoranthene	202	101	5	167



## Semi-Volatiles

Method 8270C

Revision 1.2

December 19, 2001

TABLE 1 (CONTINUED)

CAS No.	Compound	Primary	Secondary	Est. Quantitation Limits	
		Ion	Ion	Water (ug/L)	Soil(ug/kg)
86-73-7	Fluorene	166	165	5	167
118-74-1	Hexachlorobenzene	284	142	5	167
87-68-3	Hexachlorobutadiene	225	223	5	167
77-47-4	Hexachlorocyclopentadiene	237	235	5	167
67-72-1	Hexachloroethane	117	201	5	167
193-39-5	Indeno[1,2,3-cd]pyrene	276	138	5	167
78-59-1	Isophorone	82	138	5	167
534-52-1	2-Methyl-4,6-dinitrophenol	198	105	5	167
91-57-6	2-Methylnaphthalene	142	141	5	167
95-48-7	2-Methylphenol	108	107	5	167
	3- & 4-Methylphenols	108	107	5	167
91-20-3	Naphthalene	128	129	5	167
88-74-4	2-Nitroaniline	65	92	25	830
99-09-2	3-Nitroaniline	138	108	25	830
100-01-6	4-Nitroaniline	138	92	10	330
98-95-3	Nitrobenzene	77	123	5	167
88-75-5	2-Nitrophenol	139	65	5	167
100-02-7	4-Nitrophenol	109	65	5	167
621-64-7	N-Nitrosodi-n-propylamine	70	42	5	167
62-75-9	N-Nitrosodimethylamine	74	42	5	167
156-10-5	N-Nitrosodiphenylamine	169	168	5	167
87-86-5	Pentachlorophenol	266	264	5	167
85-01-8	Phenanthrene	188	94	5	167
108-95-2	Phenol	94	65	5	167
129-00-0	Pyrene	202	101	5	167
110-86-1	Pyridine	79	52	10	330
120-82-1	1,2,4-Trichlorobenzene	180	182	5	167
95-95-4	2,4,5-Trichlorophenol	196	198	5	167
88-06-2	2,4,6-Trichlorophenol	196	198	5	167



**TABLE 2**

**DFTPP KEY IONS AND ION ABUNDANCE CRITERIA<sup>1</sup>**

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	25-75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

<sup>1</sup> Criteria taken from EPA CLP SOW OLM 02.0

**TABLE 3**

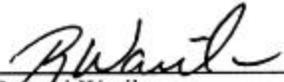
Calibration Check Compounds

<u>Base/Neutral Fraction</u>	<u>Acid Fraction</u>
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamone	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo (a) pyrene	



## ICP Metals Method 6010B

Prepared by:   
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Approved by:   
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Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 2, December 1996, Method 6010B.

## I. Applicability:

Analyte: Refer to ICP manual for installed spectral lines

Matrix: Digestates from procedures 3005A, 3010A, 3015, 3040A, 3051

Regulation: RCRA

## II. Important Notes:

The proper identification of interferences encountered while performing ICP analysis is vital to producing sound analytical data. The following is a brief summary of some of the major interferences that may produce either false positive or false negative results.

Spectral interferences are caused by (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomenon and (4) stray light from the line emission of high-concentration elements. Computer-correcting the raw data after monitoring and measuring the interfering element can compensate for spectral overlap. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Users of simultaneous multi-element instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended



as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interference can be assumed.

The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 383.231-nm wavelength interferes with the listed potassium line at 766.491 nm.

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump or by using the standard additions method. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

### **III. Procedure:**

Preliminary treatment of most matrices is necessary due to the complexity of sample matrices. The use of an internal standard or matrix matching must be used to determine concentrations of unknowns. The internal standard used is Yttrium.

Set up the instrument with proper operating parameters established by the instrument manufacturer. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Table 3. Flush the system with a reagent blank between each standard. Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error. In the case of multi-level calibrations the correlation coefficient must be > 0.995 for all elements.



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

NOTE: For boron concentrations greater than 500 mg/L, extended flush times >1 minute may be required.

The validity of the calibration must be verified by analyzing a second source standard (ICV) with concentrations of all elements of interest at or near the midpoint of the calibration.

The CCV and ICB must be run and meet the QC requirements before proceeding. Each may be rerun once before having to initiate corrective actions and recalibration.

Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. Rinse time may be reduced if data will support the absence of analytes above the stated MDLs. Analyze the instrument performance check and the calibration blank after every 10 samples.

An Interference Check solution (ICS) containing the major interferences must be run prior to analysis of samples. An ICSA and ICSAB must also be run at the beginning and end of each analytical sequence, this solution contains parts A (major interferences) only and A (major interferences) plus B (elements of interest), respectively.

A low-level check standard (or project required detection limit standard, however named) must be run at the beginning of each run and at the end. Unsatisfactory recoveries must be narrated in the final report based on project specific limits and requirements.

## IV. Standards Preparation:

Standard Concentrations: Enviro 61E

### Standard 1

50mL HNO<sub>3</sub>

450mL reagent H<sub>2</sub>O

total vol.= 500mL

This standard serves as the CCB/ICB

### Standard 2

2.5mL (1000ppm) Ag stock

5.0mL (1000ppm) As stock

5.0mL (1000ppm) B stock

2.0mL (10,000ppm) Ba stock

1.0mL (1000ppm) Be stock

5.0mL (1000ppm) Cd stock

5.0mL (1000ppm) Co stock

2.5mL (1000ppm) Cr stock

2.5mL (1000ppm) Cu stock

5.0mL (1000ppm) Mn stock

5.0mL (1000ppm) Mo stock

5.0mL (1000ppm) Ni stock

5.0mL (1000ppm) Pb stock

5.0mL (1000ppm) Sb stock

5.0mL (1000ppm) Se stock

5.0mL (1000ppm) Si stock

5.0mL (1000ppm) Sn stock

5.0mL (1000ppm) Ti stock

5.0mL (1000ppm) Tl stock

5.0mL (1000ppm) V stock

5.0mL (1000ppm) Zn stock

50mL HNO<sub>3</sub>

359.5mL reagent H<sub>2</sub>O



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

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total vol.=500mL

This sample serves as an initial calibration standard for the above analytes.

### Standard 3

2.5mL (10,000ppm) Al stock  
5.0mL (10,000ppm) Ca stock  
1.0mL (10,000ppm) Fe stock  
5.0mL (10,000ppm) Mg stock  
5.0mL (10,000ppm) K stock  
5.0mL (10,000ppm) Na stock

50mL HNO<sub>3</sub>

426.5mL reagent H<sub>2</sub>O

total vol.=500mL

This sample serves as an initial calibration standard for the above analytes.

### ICV

20mL \*CLPP-CAL 1  
10mL \*CLPP-CAL 2  
10mL \*CLPP-CAL 3  
10mL (1000ppm) Mo stock  
10mL (1000ppm) Ti stock

200mL HNO<sub>3</sub>

1760mL reagent H<sub>2</sub>O

total vol.=2000mL

\*(Certified vendor.)

#### \*CLPP-CAL 1

5000ppm Ca, Mg, K, Na  
1000ppm Al, Ba, Fe  
500ppm Co, Mn, Ni, V, Zn  
250ppm Ag, Cu  
200ppm Cr  
50ppm Be

#### \*CLPP-CAL 2

1000ppm Sb

#### \*CLPP-CAL 3

1000ppm As, Pb, Se, Tl  
50ppm Cd



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

---

### CCV

The CCV solution must be made from the same stock solutions as the calibration standards. CCV solutions can be made using the corresponding 61E Standards at a dilution of 1:5. In the instances where instability is not an issue, a combination of 61E Standards is acceptable.

### ICSAB

100mL \*\*CLPP-ICS-A  
10.0mL \*\*CLPP-ICS-B  
2.0mL (1000ppm) As stock  
1.0mL (1000ppm) B stock  
1.0mL (1000ppm) Mo stock  
1.0mL (1000ppm) Sb stock  
1.0mL (1000ppm) Se stock  
1.0mL (1000ppm) Si stock  
1.0mL (1000ppm) Sn stock  
1.0mL (1000ppm) Ti stock

100mL HNO<sub>3</sub>  
781mL reagent H<sub>2</sub>O  
total vol.=1000mL  
\*\*(Certified Vendor.)

\*\*CLPP-ICS-A  
5000ppm Al, Ca, Mg  
2000ppm Fe

### ICSA

100mL \*\*CLPP-ICS-A  
10.0mL \*\*CLPP-ICS-B  
  
100mL HNO<sub>3</sub>  
790mL reagent H<sub>2</sub>O  
total vol.=1000mL

\*\*CLPP-ICS-B  
100ppm Cd, Pb, Ni, Ag, Zn  
50ppm Ba, Be, Cr, Co, Cu, Mn, V

### Yttrium internal standard

#### Stock solution

0.39g Yttrium solid (Yttrium Nitrate Tetrahydrate, 99.999% or Yttrium Oxide, 99.9999%)  
10mL HNO<sub>3</sub>  
90mL reagent H<sub>2</sub>O  
(Note: Use of Yttrium oxide requires that the solution be gently heated.)

#### Working solution

50mL stock solution  
100mL HNO<sub>3</sub>  
850mL reagent H<sub>2</sub>O  
total vol.=1000mL

This solution is not used to determine a specific concentration but a constant absorbance, therefore has no QC required recovery limits.

#### 61E Profile solution

Next revision: 1/2006

5 of 18



**ICP Metals**

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

1.0mL (1000ppm) Cu stock

10mL HNO<sub>3</sub>

89mL reagent H<sub>2</sub>O

total vol.=100mL

This solution is used to generate a peak on the copper line in order to profile the instrument, the final solution is ≈ 10ppm.

**61E Table 3**

All values are in ( ppb )

Element	STD1	STD2	STD3	CCV	ICV	ICSA	ICSAB	LFB
Ag	BLANK	5,000	-	1,000	2,500	-	1,000	500
Al		-	50,000	10,000	20,000	250,000	250,000	10,500
As		10,000	-	2,000	5,000	-	2,000	500
B		10,000	-	2,000	5,000	-	1,000	500
Ba		40,000	-	8,000	20,000	-	500	500
Be		2,000	-	400	500	-	500	500
Ca		-	100,000	20,000	50,000	250,000	250,000	10,500
Cd		10,000	-	2,000	2,500	-	1,000	500
Co		10,000	-	2,000	5,000	-	500	500
Cr		5,000	-	1,000	2,000	-	500	500
Cu		5,000	-	1,000	2,500	-	500	500
Fe		-	20,000	4,000	10,000	100,000	100,000	500
K		-	100,000	20,000	50,000	-	-	25,000
Mg		-	100,000	20,000	50,000	250,000	250,000	10,500
Mn		10,000	-	2,000	5,000	-	500	500
Mo		10,000	-	2,000	5,000	-	1,000	500
Na		-	100,000	20,000	50,000	-	-	10,500
Ni		10,000	-	2,000	5,000	-	1,000	500
Pb		10,000	-	2,000	5,000	-	1,000	500
Sb		10,000	-	2,000	5,000	-	1,000	500
Se		10,000	-	2,000	5,000	-	1,000	500
Sn		10,000	-	2,000	5,000	-	1,000	500
Ti		10,000	-	2,000	5,000	-	1,000	500
Tl		10,000	-	2,000	5,000	-	1,000	500
V		10,000	-	2,000	5,000	-	500	500
Zn		10,000	-	2,000	5,000	-	1,000	500

Multipoint calibration is performed using the listed standards as is, @1:2 dilutions, and @1:4 dilutions.

Next revision: 1/2006



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

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A low level standard is run for all elements of interest at or near specific project reporting limits when applicable.

### Standard Concentrations: TRACE

#### **TRACE 1**

50mL HNO<sub>3</sub>

450mL reagent H<sub>2</sub>O

total vol.= 500mL

This standard serves as the CCB/ICB

#### **TRACE 2**

0.5mL (1000ppm) Ag stock

0.5mL (1000ppm) As stock

2.5mL (1000ppm) B stock

0.25mL (10,000ppm) Ba stock

0.5mL (1000ppm) Be stock

0.5mL (1000ppm) Cd stock

0.5mL (1000ppm) Co stock

0.5mL (1000ppm) Cr stock

0.5mL (1000ppm) Cu stock

0.5mL (1000ppm) Mn stock

0.5mL (1000ppm) Mo stock

0.5mL (1000ppm) Ni stock

0.5mL (1000ppm) Pb stock

2.5mL (1000ppm) Sb stock

2.5mL (1000ppm) Se stock

0.5mL (1000ppm) Ti stock

0.5mL (1000ppm) Tl stock

0.5mL (1000ppm) V stock

50mL HNO<sub>3</sub>

435.25mL reagent H<sub>2</sub>O

total vol.=500mL

This sample serves as an initial calibration standard for the above analytes.

#### **TRACE 3**

0.5mL (10,000ppm) Al stock

0.5mL (10,000ppm) Ca stock

1.0mL (10,000ppm) Fe stock

0.5mL (10,000ppm) Mg stock

5.0mL (10,000ppm) Na stock

50mL HNO<sub>3</sub>

442.5mL reagent H<sub>2</sub>O

Next revision: 1/2006

7 of 18



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

---

total vol.=500mL

This sample serves as an initial calibration standard for the above analytes.

### TRACE 4

2.5mL (1000ppm) Sn stock

0.5mL (1000ppm) Zn stock

50mL HNO<sub>3</sub>

447mL reagent H<sub>2</sub>O

total vol.=500mL

This sample serves as an initial calibration standard for the above analytes.

### TRACE 5

2.5mL (10,000ppm) K stock

50mL HNO<sub>3</sub>

447.5mL reagent H<sub>2</sub>O

total vol.=500mL

This sample serves as an initial calibration standard for the above analyte.

### ICV

100mL 61E ICV

0.25mL (1000ppm) Ag stock

2.0mL (1000ppm) B stock

0.50mL (1000ppm) Be stock

0.25mL (1000ppm) Cd stock

0.25mL (1000ppm) Cr stock

0.25mL (1000ppm) Cu stock

0.90mL (10,000ppm)Fe stock

2.0mL (10,000ppm) K stock

0.45mL (10,000ppm) Na stock

2.0mL (1000ppm) Se stock

2.0mL (1000ppm) Sb stock

2.0mL (1000ppm) Sn stock

100mL HNO<sub>3</sub>

787.15mL reagent H<sub>2</sub>O

total vol.=1000mL

This sample serves as the quality control sample for the above analytes.

**All stock solutions used must be from a source independent of the calibration standards.**

### CCV

The CCV solution must be made from the same stock solutions as the calibration standards.

CCV solutions can be made using the corresponding Trace Standards at a dilution of 1:5. In the instances where instability is not an issue, a combination of Trace Standards is acceptable.

### ICSAB

25mL \*\*CLPP-ICS-A

2.5mL \*\*CLPP-ICS-B



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

0.50mL (1000ppm) As stock  
0.25mL (1000ppm) B stock  
0.25mL (1000ppm) Mo stock  
0.25mL (1000ppm) Sb stock  
0.25mL (1000ppm) Se stock  
0.25mL (1000ppm) Sn stock  
0.25mL (1000ppm) Ti stock

100mL HNO<sub>3</sub>  
870.5mL reagent H<sub>2</sub>O  
total vol.=1000mL

**\*\*(Certified Vendor.)**

**\*\*CLPP-ICS-A**

5000ppm Al, Ca, Mg

2000ppm Fe

**\*\*CLPP-ICS-B**

100ppm Cd, Pb, Ni, Ag, Zn

50ppm Ba, Be, Cr, Co, Cu, Mn, V

Alternately, the TRACE SICAB may be created through a 1:4 dilution of the 61E SICAB.

**ICSA-** The ICSA is created through a 1:4 dilution of the 61E ICSA solution.

### **TRACE only-Yttrium/Lithium internal standard**

#### **Yttrium stock solution**

0.39g Yttrium solid (Yttrium Nitrate Tetrahydrate, 99.999%,  
or Yttrium Oxide, 99.999%)

10mL HNO<sub>3</sub>

90mL reagent H<sub>2</sub>O

#### **Lithium stock solution**

0.25 g (99.999%)

10mL HNO<sub>3</sub>

90mL reagent H<sub>2</sub>O

(Note: Use of Yttrium oxide requires that the solution be gently heated to dissolve.)

#### **Working solution**

25mL Yttrium solution

5mL Lithium

100mL HNO<sub>3</sub>

870mL reagent H<sub>2</sub>O

total vol.=1000mL

This solution is not used to determine a specific concentration but a constant absorbance.

#### **TRACE Profile solution**

0.5 mL (1000ppm) As stock

10mL HNO<sub>3</sub>

89.5 mL reagent H<sub>2</sub>O

total vol.=100mL

In TRACE analysis this solution is used to generate a peak on the arsenic line in order to profile the instrument, the final solution is ≈ 5ppm.



**TRACE Table 3**

All values are in ( ppb )

Element	TRACE 1	TRACE2	TRACE3	TRACE4	TRACE5	CCV	ICV	ICSA	ICSAB	LFB
Ag	BLANK	1,000	---	---	---	500	500	---	250	500
Al	BLANK	---	10,000	---	---	5,000	2,000	62,500	62,500	10,500
As	BLANK	1,000	---	---	---	500	500	---	500	500
B	BLANK	5,000	---	---	---	2,500	2,000	---	250	500
Ba	BLANK	5,000	---	---	---	2,500	2,000	---	125	500
Be	BLANK	1,000	---	---	---	500	550	---	125	500
Ca	BLANK	---	10,000	---	---	5,000	5,000	62,500	62,500	10,500
Cd	BLANK	1,000	---	---	---	500	500	---	250	500
Co	BLANK	1,000	---	---	---	500	500	---	125	500
Cr	BLANK	1,000	---	---	---	500	450	---	125	500
Cu	BLANK	1,000	---	---	---	500	500	---	125	500
Fe	BLANK	---	20,000	---	---	10,000	10,000	25,000	2,500	2,500
K	BLANK	---	---	---	50,000	25,000	25,000	---	---	25,000
Mg	BLANK	---	10,000	---	---	5,000	5,000	62,500	62,500	10,500
Mn	BLANK	1,000	---	---	---	500	500	---	125	500
Mo	BLANK	1,000	---	---	---	500	500	---	250	500
Na	BLANK	---	100,000	---	---	50,000	50,000	---	---	10,500
Ni	BLANK	1,000	---	---	---	500	500	---	250	500
Pb	BLANK	1,000	---	---	---	500	500	---	250	500
Sb	BLANK	5,000	---	---	---	2,500	2,500	---	250	500
Se	BLANK	5,000	---	---	---	2,500	2,500	---	250	500
Sn	BLANK	---	---	5,000	---	2,500	2,500	---	250	500
Ti	BLANK	1,000	---	---	---	500	500	---	250	500
Tl	BLANK	1,000	---	---	---	500	500	---	250	500
V	BLANK	1,000	---	---	---	500	500	---	125	500
Zn	BLANK	---	---	1,000	---	500	500	---	250	500



Multipoint calibration is performed using the listed standards as is, @1:2 dilutions, and @1:4 dilutions with the exception of Trace 3, which is diluted 1:10 instead of 1:4. All standard dilutions must be properly recorded in the working standards logbook.

A low level standard is run for all elements of interest at or near specific project reporting limits when applicable.

**TABLE 2**  
**POTENTIAL INTERFERENCES**  
**ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM**  
**INTERFERENCES AT THE 100-mg/L LEVEL<sup>a</sup>**

Analyte	Wavelength (nm)	Interferent <sup>a,b</sup>									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

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<sup>a</sup> Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 gm/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

<sup>b</sup> The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentrations to the interferent figure.

<sup>c</sup> Interferences will be affected by background choice and other interferences that may be present. Additional interference corrections are required with an axial view instrument.

## V. Quality Assurance:

All quality control data should be maintained and available for easy reference or inspection.

### Calibration Solutions

The calibration solutions are made using the same or similar acid matrix as the samples to be analyzed.

### High Standards Check (HSC)

The HSC is the highest level standard applied in a multi-point calibration for each analyte of interest. The HSC is run immediately after the calibration when required to meet specific project requirements. The HSC recovery must be within  $\pm 5\%$  of the true value for each analyte of interest.

### Initial Calibration Verification (ICV)

The ICV must be made from an outside second source different from that of the calibration standards' stock solutions.

The ICV is used to verify initially the calibration standards or stock solutions. The ICV must be run following the calibration. The ICV recovery must be within  $\pm 10\%$  of the true value for each analyte of interest.

### Continuing Calibration Verification (CCV)

The CCV must be run periodically (every 10 samples) and at the end of each analytical sequence. The CCV is made from the same source as the calibration standards.

All recoveries must be  $\pm 10\%$  of the true value. The CCV may be run one additional time if the specified recoveries are not met, however if the second analysis fails, corrective action must be taken and any samples analyzed after the previous valid CCV must be re-analyzed.



### **Calibration Blank**

The calibration blank contains the same acid matrix as the calibration standards and run with the ICV. The calibration blank is also used as the Continuing Calibration Blank (CCB) solution. See note 1.

The results of the calibration blank are to agree within two standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem; re-calibrate, and reanalyze the previous 10 samples.

### **Laboratory Reagent Blank (LRB)**

The LRB is a reagent blank carried through the entire sample preparation process.

Employ a minimum of one laboratory reagent blank with each batch of 20 or fewer samples of the same matrix, to verify the absence of contamination. The LRB must be less than the reported detection limit for each analyte of interest.

### **Laboratory Fortified Blank (LFB)**

A laboratory fortified blank (LFB) must be run with each sample batch. If the recovery falls outside the control limit of 80-120% \*or established control limits, the problem is to be identified and resolved before continuing. \*The more restrictive limits prevail. The LFB is spiked, from a source independent of both the standards and ICV, prior to digestion and brought through the entire process.

### **Interference Check Solutions (ICS)**

The ICS are analyzed in order to validate inter-element and background corrections applied to the samples.

The interference check solutions are prepared by combining known concentrations of interfering elements that will provide an adequate test of the correction factors, the "A fraction". Fortify the ICSAB solutions with the elements of interest in the 1 mg/L range, known as the "B fraction". In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero.

Analyze the ICSA and the ICSAB at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. Recoveries of elements of interest should be within  $\pm 20\%$  of the true values in the ICSAB and less than 2 times the reporting limit in the ICSA.

### **Sample Duplicate**

Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of  $\pm 20\%$  for RPD shall be used for sample values greater than 10 times the instrument detection limit.

### **Laboratory Fortified Matrix / Duplicate (LFM/LFMD)**

The LFM/LFMD pair must be run with each batch of 20 or fewer samples of the same matrix.



## ICP Metals

Method 6010B

Revision 3.1

Effective Date: January 21, 2004

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The LFM/LFMDs are prepared from fresh sample aliquots, spiked in the same manner as the LFB and carried through the entire preparation process.

The matrix spike and matrix spike duplicate spike recovery should be within  $\pm 25\%$  of the true value, or documented control limits. Recovery calculations are not made if the spike concentration is less than 25% of the sample concentrations.

### **Inter-Element Corrections (IECs)**

IECs are determined by analyzing a solution that contains an individual interfering element and is free of all other contaminants.

The positive or negative effects on the elements of interest are corrected by the following:

Correction value = true value of interfering element / concentration of the element of interest

IECs must only be evaluated and applied by analyst trained in their application.

IEC determination must be verified annually (at least) and updated, if necessary.

### **Linearity (L)**

Dilute and reanalyze samples that are  $>90\%$  of the established linear calibration limit or use an alternate, less sensitive line for which quality control data is established.

Linearity for all analytes must be updated quarterly.

### **Method Detection Limit (MDL)**

MDLs must be maintained for each analyte of interest and updated once every year.

The determination of MDLs must be made in accordance with the following:

Fortify reagent water at a concentration of 2 to 3 times the estimated instrument detection limit.

Take seven replicate aliquots of the fortified reagent water and process through the entire analytical method.

Perform all calculations defined in the method and report the concentration values in the appropriate units.

Calculate the MDL as follows:

$$\text{MDL} = (t) \times (s)$$

where:

t = student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.143 for seven replicates].

S = standard deviation of the replicate analyses.

The final calculated MDL must be greater than 20% of the original analyte spike level.

### **Matrix Evaluation**

It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests will ensure the



analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. They are as follows:

**Serial dilution:** If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

**Post (digestion) Spike:** An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 70% to 130% of the known value or the established control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The use of a standard-addition analysis procedure may be used to compensate for this effect.

**CAUTION:** The standard-addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation (IECs), an alternate wavelength, or comparison with an alternate method is recommended.

#### Method of Standard Additions:

The standard-addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume  $V_X$ , are taken. To the first (labeled A) is added a small volume  $V_S$  of a standard analyte solution of concentration  $C_S$ . To the second (labeled B) is added the same volume  $V_S$  of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration  $C_X$  is calculated:

$$C_X = \frac{S_B * V_S * C_S}{(S_A - S_B) * V_X}$$

where  $S_A$  and  $S_B$  are the analytical signals (corrected for the blank) of solutions A and B, respectively.  $V_S$  and  $C_S$  should be chosen so that  $S_A$  is roughly twice  $S_B$  on the average. It is best if  $V_S$  is made much less than  $V_X$ , and thus  $C_S$  is much greater than  $C_X$ , to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results of this technique to be valid, the following limitations must be taken into consideration:

The analytical curve must be linear, the correlation coefficient must be  $>0.995$ .

The chemical form of the analyte must respond the same way as the analyte in the sample.

The interference effect must be constant over the working range of concern.

The signal must be corrected for any additive interference.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of



the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

## VI. Calculations:

Results are read in ug/L directly from the ICP. Take into account any dilutions performed during the digestion process for total metals.

The recoveries of spikes and relative percent difference between duplicate determinations are to be calculated as follows:

$$RPD = |C_S - C_D| / ((C_S + C_D) / 2)$$

$$Rec = 100 * (C_M - C_S) / C_T$$

where RPD = relative percent difference, %

Rec = matrix spike recovery, %

C<sub>S</sub> = unspiked sample concentration, mg/L

C<sub>D</sub> = duplicate sample concentration, mg/mL

C<sub>M</sub> = matrix spike concentration, mg/L

C<sub>T</sub> = theoretical spike concentration, mg/L

Report recovery and RPD to the nearest 1 %.

## VII. Reagents and Materials:

### Thermo Jarrell Ash 61E Simultaneous ICAP:

Capable of analysis and background correction for multi-element analysis

### Thermo Jarrell Ash TRACE 61E Simultaneous ICAP:

Capable of trace analysis and background correction for multi-element analysis

### Argon gas supply:

High purity, liquid or high pressure cylinders

### Concentrated hydrochloric acid:

Metals analysis grade

### Hydrochloric acid, 1:1 dilution:

Add 500 mL concentrated hydrochloric acid to 400 mL reagent water and dilute to 1 liter

### Concentrated nitric acid:

Metals analysis grade

### Nitric acid, 1:1 dilution:

Add 500 mL concentrated nitric acid to 400 mL reagent water and dilute to 1 liter



**Standard stock solutions:**

Purchased from commercial suppliers

**Second source solutions:**

Purchased from commercial suppliers

**Mixed calibration standard solutions:**

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric. Add the appropriate types and volumes of acids to match sample matrix. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to PFE fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample and monitored for stability.

**Important:** If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of reagent water and warm the flask until the solution clears. Cool and dilute to 100 mL with reagent water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional hydrochloric acid.

**Note 1:** If the sample analysis solution has a different acid concentration from that given, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

## VIII. Safety:

Every sample should be considered a hazardous when performing the analysis. Standard laboratory safety guidelines must be adhered to. Gloves, eye protection, and lab coats must be worn during sample retrieval, analysis and disposal.

## IX. Pollution Prevention:

Any and all remaining unused sample must be returned to the 4°C storage, sealed tightly in the original container. Benches and surrounding surfaces must be cleaned and wiped dry with paper toweling.

## X. Waste management:

Analyzed sample and used disposable equipment must be collected and disposed of in a manner consistent with the Premier Laboratory Chemical Hygiene Plan.

## XI. Method Performance:

Performance data is not currently available.



**ICP Metals**  
Method 6010B  
Revision 3.1  
Effective Date: January 21, 2004

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## Instrument Maintenance Metals Department

Prepared by:   
Robert Stevenson  
Quality Assurance Officer

Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 1, July 1992, Method  
7000A.

### Table of Contents

Section	Title	Date
MP001	Frequencies and Triggering Events for Maintenance	August 24,1999
IP001	Cleaning the Torch	August 24,1999
IP002	Cleaning the Nebulizer	August 24,1999
IP003	Replacing the Peristaltic Pump Tubing	August 24,1999
IP004	Cleaning the Autosampler	August 24,1999
GF001	Cleaning the Contact Rings	July 28, 1998
GF002	Cleaning the Quartz Windows	July 28, 1998
GF003	Checking the Furnace Cooling Water	July 28, 1998
CV001	Cleaning the Quartz Cell	July 28, 1998
CV002	Replacing the Reagent Tubing	July 28, 1998



### ICP SPECTROPHOTOMETERS

Frequency or Triggering Event	Maintenance Required	Procedure
Daily As needed: loss of linearity or sensitivity	Clean the torch	IP001
Clogging or drop in sensitivity	Clean the nebulizer end cap	IP002
Every two weeks	Replace the peristaltic pump tubings	IP003
Every month	Clean the autosampler	IP004

### GRAPHITE FURNACE AA's

Frequency or Triggering Event	Maintenance Required	Procedure
Before the start of each run	Clean the contact rings	GF001
Daily	Clean the quartz windows	GF002
Weekly	Check the cooling water level	GF003

### COLD VAPOR AA's

Frequency or Triggering Event	Maintenance Required	Procedure
Daily	Clean the quartz cell	CV001
Every month	Replace the reagent tubing	CV002
Every six months	Clean the liquid/gas separator	CV003



Standard Operating Procedures  
Maintenance Procedure No. IP001

**CLEANING THE TORCH**

1. Remove the nebulizer and spray chamber.
2. Gently remove the torch.
3. Place the torch, inverted, in a beaker containing aqua-regia ( 1 part conc.  $\text{HNO}_3$  : 3 parts conc.  $\text{HCl}$  ) being careful not to submerge the ceramic base. Soak for one hour. Do not soak for extended periods of time, the torch is held in place only with adhesive.
4. Rinse with reagent water.
5. Sonicate in a beaker of 10% nitric acid in a water bath for at least 2 hours.
6. Rinse with reagent water and dry thoroughly with compressed air.
7. Replace the torch.



Standard Operating Procedures  
Maintenance Procedure No. IP002

**CLEANING THE NEBULIZER**

1. Disconnect the nebulizer from the spray chamber.
2. Rinse with reagent water and soak in a laboratory detergent solution.
3. Rinse with reagent water and 20% nitric acid solution.
4. Reattach the nebulizer to the spray chamber.
5. High salt build ups may be removed by soaking the nebulizer in 1:1 HCl, being careful to rinse all traces of HCl from the nebulizer afterwards.



Standard Operating Procedures  
Maintenance Procedure No. IP003

**REPLACING THE PERISTALTIC PUMP TUBING**

1. Loosen the tension on the pump windings.
2. Remove the tensioning plates from the peristaltic pump.
3. Remove and discard the old windings.
4. Install new windings.
5. Align the tensioning barbs on the tensioning plates and reattach the plates.
6. Adjust the tension for maximum flow.
7. Run the pumps for at least 30 minutes in order to condition the new windings prior to sample analysis.



Standard Operating Procedures  
Maintenance Procedure No. IP004

**CLEANING THE AUTO SAMPLER**

1. Remove all autosampler racks.
2. Rinse the reservoir with deionized water.
3. Wipe the autosampler tray with a cloth dampened with a laboratory detergent solution.
4. Spray light penetrating oil on all moving assemblies as necessary to facilitate motion.
5. Reassemble the autosampler.



Standard Operating Procedures  
Maintenance Procedure No. GF001

**CLEANING THE CONTACT RINGS**

1. Open the graphite furnace and remove the graphite tube.
2. Use a cotton swab with methanol to remove all visible graphite particles and sample or modifier remnants from the contact ring surface.
3. Blow the rings clear with compressed air.
4. Reinstall the graphite tube and close the furnace.



Standard Operating Procedures  
Maintenance Procedure No. GF002

**CLEANING THE QUARTZ WINDOWS**

1. Remove the left window by loosening the thumb screw.
2. Remove the right window by pulling it away from the furnace seat.
3. Gently wipe the window surfaces with a cotton swab dipped in methanol.
4. Examine the windows for streaks or spots. Repeat wiping with methanol as necessary until the windows are clean.
5. Replace windows.



Standard Operating Procedures  
Maintenance Procedure No. GF003

**CHECKING THE FURNACE COOLING WATER**

1. Check the fluid level indicator on the front of the cooling unit.
2. Fill to the top level indicator with 1 % glycerol in deionized water.



Standard Operating Procedures  
Maintenance Procedure No. CV001

**CLEANING THE QUARTZ CELL**

1. Remove the mercury vapor tube and ventilation tube from the cell.
2. Remove the quartz windows from the cell.
3. Pipette about 10 mL of methanol into the cell and swirl. Discard the methanol. Repeat this rinse 3 times.
4. Dry the quartz cell with compressed air.
5. Replace the cell and reconnect the tubes.



Standard Operating Procedures  
Maintenance Procedure No. CV002

**REPLACING THE REAGENT TUBING**

1. Disconnect the tubing.
2. Discard the old tubing.
3. Replace with the appropriate Tygon tubing.



Standard Operating Procedures  
Maintenance Procedure No. CV003

**CLEANING THE LIQUID/GAS SEPARATOR CELL**

1. Disconnect all tubing.
2. Remove the cell.
3. Flush the cell with 10% HCl, repeat until all stannous chloride and debris is cleared.
4. Replace cell and attached tubing and flush with method reagents prior to use.



## Maintenance

Revision 1.1

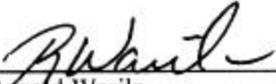
March 28, 2001

Effective Date: March 28, 2001

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### Maintenance (Organics)

Prepared by:   
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### TABLE OF CONTENTS

Section	Title
MP001	Frequencies and Triggering Events for Maintenance
GC000	Gas Chromatography Maintenance to be Performed by Contractors
GC001	Changing Septa
GC002	Clipping Capillary Columns
GC003	Changing Gas Filters
GC004	Changing PID Lamp
GC005	Changing GC Columns
GC006	Cleaning the ECD Detector
MS000	Mass Spectrometer Maintenance to be Performed by Contractors
MS001	Cleaning the Ion Source
PT001	Changing the Purge Trap



## Maintenance

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

### FREQUENCIES AND TRIGGERING EVENTS FOR MAINTENANCE

#### ***PURGE AND TRAP DEVICES***

Frequency or Triggering Event	Maintenance Required	Procedure
1) Trap contaminated 2) Sensitivity to gasses or ketones decreases below acceptable levels.	Change Trap	PT001

#### ***GAS CHROMATOGRAPHS***

Frequency or Triggering Event	Maintenance Required	Procedure
1) Annually 2) Baseline becomes elevated 3) Area counts become inconsistent	have ECD cleaned	GC000
Resolution becomes poor	Clip column	GC002
Annually or when indicated	Change gas filter	GC003
Loss of sensitivity	Change PID lamp	GC004
1) Column becomes too short for acceptable resolution 2) Column cannot be cleaned by bake out 3) Loss of sensitivity	Change column	GC005
Baseline becomes elevated	Bake out ECD detector	GC006

#### ***MASS SPECTROMETERS***

Frequency or Triggering Event	Maintenance Required	Procedure
Semi-Annually	Change rough pump oil	MS000
1) Instruments cannot be tuned or tunes are erratic 2) Sensitivity decreases below acceptable levels	Clean ion source	MS001



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. GCOOO

### **GAS CHROMATO GRAPH MAINTENANCE TO BE PERFORMED BY CONTRACTORS**

ECD: Hydrogen cleaning shall be performed by a certified vendor.



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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### Standard Operating Procedures Maintenance Procedure No. GCOO 1

#### **CHANGING SEPTA**

1. Allow the injector port to cool.
2. Remove the injector cap.
3. Remove and replace the septum.
4. Replace the injector cap and heat the injector.
5. Pierce the septum with a syringe.



## Maintenance

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. GC002

### CLIPPING CAPILLARY COLUMNS

1. Allow the injection port to cool.
2. Loosen the fitting on the injector end of the column.
3. Inspect and replace the ferrule if necessary.
4. Use a capillary column cutter to clip approximately one loop from the column. Inspect the cut end with a magnifier to ensure the cut is perpendicular to the tubing wall and is free of chips, burrs, or uneven areas. Make a new cut if necessary.
5. Inert the column into the injector so that the end of the column will be about 2 cm below the tip of the syringe when an injection is being made.
6. Tighten the fitting (DO NOT OVERTIGHTEN!) and heat the injector.



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. GC003

### **CHANGING GAS FILTERS**

1. Turn off the gas supply.
2. Loosen the nut at each end of the filter.
3. Remove the filter and replace it with a new filter.
4. Tighten the nut at each end of the filter.
5. Turn the gas supply on.
6. Check for leaks



## Maintenance

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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### Standard Operating Procedures Maintenance Procedure No. GC004

#### **CHANGING THE PID LAMP**

1. Turn the lamp power supply off and allow the detector to cool.
2. Remove the lamp shield by depressing and turning it counter-clockwise.
3. Remove the two lamp ring retaining screws.
4. Remove the lamp by lifting it straight up. The lamp retaining ring, wave spring, and contact ring will also be removed.
5. Replace the lamp followed by the contact ring, wave spring, and retaining ring
6. Tighten the two retaining screws.
7. Replace the lamp shield by pressing it into place and turning it clockwise.
8. Heat the detector and turn on the lamp power supply.



## Maintenance

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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### Standard Operating Procedures Maintenance Procedure No. GC005

## CHANGING GC COLUMNS

1. Allow the oven, injector, and detector to cool.

GC/MS: Cool the ion source. When cool, turn the pumps off and vent the source.

2. Loosen the retaining nut at each end of the column and remove the column.
3. Place the nuts on the new column with new ferrules.
4. Use a capillary column cutter to clip a few cm from each end of the new column. Inspect the cut ends with a magnifier to ensure the cut is perpendicular to the tubing wall and is free of chips, burrs, or uneven areas. Make a new cut if necessary.
5. Insert the column ends into the injector and detector and tighten the fittings (DO NOT OVERTIGHTEN).
6. Heat the detector, injector, and oven.
7. GC/MS: Turn the pumps on and heat the ion source.
8. Condition the column per manufacturer's specifications.



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. GC006

### **CLEANING THE ECD DETECTOR**

1. PERFORM THIS PROCEDURE ONLY IN THE EVENT OF A SEVERE SIGNAL PROBLEM.
2. Bake the detector at 350°C for 12 - 24 hours.



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. MS000

### **MASS SPECTROMETER MAINTENANCE TO BE PERFORMED BY CONTRACTORS**

1. Turbo Pumps: All service shall be performed by a certified vendor.
2. Rough pumps: Change pump oil every 6 months.



## **Maintenance**

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. MS001

### **CLEANING THE ION SOURCE**

1. Allow the mass spectrometer to cool.
2. Turn the pumps off.
3. Remove the analyzer.
4. Clean the ion source according to the directions in the Hewlett-Packard HP5970B MSD Hardware Manual section 4-15.
5. Replace the analyzer.
6. Turn the pumps and mass spectrometer heater on.



## Maintenance

Revision 1.1

March 28, 2001

Effective Date: March 28, 2001

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Standard Operating Procedures  
Maintenance Procedure No. PT001

### **CHANGING THE PURGE TRAP**

1. Remove the trap door.
2. Set the purge and trap device to Purge Ready.
3. Loosen the nut at the bottom of the trap while holding the bottom fitting in place.
4. Loosen the top nut.
5. Pull the trap straight down through the furnace sleeve.
6. Insert a new trap.
7. Replace the ferrules if necessary.
8. Tighten the top nut.
9. Tighten the bottom nut while holding the bottom fitting in place.
10. Replace the trap door.
11. Follow the directions in the applicable analytical procedure to condition the trap before use.

## **TEI-110-E, Particle Size Analysis of Soils**

### **Overview**

This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75  $\mu\text{m}$  (No. 200) is determined by sieving, while the distribution of particle sizes smaller than 75  $\mu\text{m}$  is determined by a sedimentation process, using a hydrometer to secure the necessary data.

If hydrometer analysis is not required, but a determination of material passing the 75  $\mu\text{m}$  (No. 200) sieve is desired, refer to Test Method TEI401-A, Sieve Analysis of Fine and Coarse Aggregate" for low P.I. materials, or Test Method "TEI-111-E, Determining the Amount of Material in Soils Finer than the 75  $\mu\text{m}$  (No. 200) Sieve" for clay materials.

### **Part I, Sieve Analysis of Material Retained on the 425 mm (No. 40) Sieve**

Part I details the necessary steps for sieve analysis of material retained on the 425  $\mu\text{m}$  (No. 40) sieve.

### **Apparatus**

The following apparatus is required:

- ◆ drying oven, maintained at  $110 \pm 5$  °C ( $230 \pm 9$  °F)
- ◆ mechanical sieve shaker
- ◆ balance, minimum capacity of 15 kg ( 33 lbs.), accurate and readable to at least 0.5 g or 0.1% of the test load, whichever is greater
- ◆ sample splitter, quartering machine, or quartering cloth
- ◆ standard U.S. sieves meeting the requirements of Test Method "TEI-907-K, Verifying the Accuracy of Wire-Cloth Sieves"
- ◆ pans.

## Samples

The mass of sample shall be sufficient for particle size analysis. The minimum amount required of material retained on the 425  $\mu\text{m}$  (No. 40) sieve depends on the maximum particle size. The size shall not be less than the amount shown in the following table. When the nominal maximum size is between sizes shown, use next larger minimum mass.

<b>Mass Requirement for Sieve Analysis</b>	
<b>Nominal Maximum Size</b>	<b>Approximate Minimum Mass</b>
9.5 mm (3/8 in.)	0.5 kg (1 lb.)
25 mm (1 in.)	2 kg (4 lb.)
37.5 mm (1-1/2 in.)	4 kg (8 lb.)
50 mm (2 in.)	5 kg (10 lb.)
75 mm (3 in.)	6 kg (12 lb.)

NOTE: The size of the portion passing the 425  $\mu\text{m}$  (No. 40) sieve shall be:

- ◆ for the hydrometer test, approximately 100 g for sandy soil and approximately 50 g for silty or clayey soils
- ◆ for hygroscopic moisture determination, at least 10 g.

## Procedure

The following table details the steps for sieve analysis of material retained on the 425 µm (No. 40) sieve.

<b>Sieve Analysis</b>	
<b>Step</b>	<b>Action</b>
1	<ul style="list-style-type: none"> <li>◆ Prepare a sample of material for analysis according to 'Dry Preparation (Method A)' of Test Method "TEI-101-E, Preparing Soil and Flexible Base Materials for Testing."</li> <li>◆ Record the mass of the material passing the 425 µm (No. 40) sieve (soil binder) as <math>W_S</math> under 'Calculations.'</li> </ul>
2	<ul style="list-style-type: none"> <li>◆ Obtain all sieve sizes required by the material specification.</li> <li>◆ Stack sieves in descending order with the sieve having the largest opening on top and a pan on the bottom.</li> </ul>
3	<ul style="list-style-type: none"> <li>◆ Pour the plus (+) 425 µm (No. 40) portion of the sample into the sieves.</li> <li>◆ Use a mechanical shaker and shake the sieves for five minutes.</li> </ul>
4	<ul style="list-style-type: none"> <li>◆ After shaking, remove the top sieve from the stack without losing any of the retained material.</li> <li>◆ Over a clean pan, hand sieve until not more than one percent, by weight, of the material retained on the sieve continues to pass through the sieve.</li> <li>◆ Combine any material passing the sieve with the material retained on the next smallest size sieve.</li> </ul>
5	<ul style="list-style-type: none"> <li>◆ Weigh the portion retained on the first sieve and record the mass as <math>W_1</math> under 'Calculations.'</li> <li>◆ Repeat Step 4 for the next largest sieve size and then add the material retained to the portion retained on the first sieve and record the combined weight as <math>W_2</math> under 'Calculations.'</li> <li>◆ Continue hand sieving and recording the combined masses, as <math>W_3</math>, <math>W_4</math>, etc., until all sieving is completed.</li> </ul>

## Calculations

- ◆ Calculate the total mass of the sample:

$$W_T = W_S + W$$

Where:

- $W_T$  = total mass of sample, g
  - $W_S$  = mass of material passing the 425 µm (No. 40) sieve, g
  - $W$  = cumulative mass of smallest sieve size, g.
- ◆ Calculate cumulative percent retained for each sieve:

$$\text{Cumulative \% Retained first sieve} = 100 \bullet W_1 / W_T$$

$$\text{Cumulative \% Retained second sieve} = 100 \bullet W_2 / W_T, \text{ etc.}$$

- ◆ Calculate individual percent retained for each sieve by subtracting the cumulative percent retained of one sieve size larger from the cumulative percent retained of the sieve size:

$$\text{Individual \% Retained} = \text{Cum. \% of } W_2 - \text{Cum. \% of } W_1, \text{ etc.}$$

Plot the cumulative percent retained, from above, versus the sieve size, on 'Form 481, Cumulative Mechanical Analysis' or semi-logarithmic paper.

The following table illustrates the calculation for sieve analysis.

<b>Sieve Analysis Calculations</b>			
<b>Sieve Size</b>	<b>Cumulative Weight Retained (g)</b>	<b>Cumulative Percent Retained</b>	<b>Individual Percent Retained</b>
12.5 mm (1/2 in.)	108.4	2.8	2.8
9.5 mm (3/8 in.)	412.5	10.8	8.0
4.75 mm (No. 4)	2285.0	59.6	48.8
2.36 mm (No. 8)	3523.0	91.9	32.3
Total - including minus (-) 425 µm (No. 40) material	3832.0	100	-

## Test Report

Report the individual percent retained on each sieve to the nearest whole number.

## Part II, Hydrometer Analysis of Soils Passing 425 mm (No. 40) Sieve

This part describes the analysis of soils passing the 425 µm (No. 40) sieve using a hydrometer.

## Apparatus

The following apparatus is required:

- ◆ balance, minimum capacity of 200 g, accuracy of 0.01 g or less
- ◆ stirring apparatus (either a 'Mechanical Stirring Device' or an 'Air Dispersion Device' )
- ◆ 'Hydrometer,' graduated in grams per liter, Type 151 H or 152 H
- ◆ sedimentation cylinder - a glass hydrometer cylinder approximately 457 mm (18 in.) in height and 63.5 mm (2.5 in.) in diameter, graduated for a volume of 1000 cc (shown in 'Air Dispersion Device')
- ◆ mercury thermometer, range of 0 to 104 °C (1 to 220 °F), accurate to 0.5 °C (1 °F)
- ◆ standard U.S. sieves, meeting the requirements of Test Method "TE1907-K, Verifying the Accuracy of Wire-Cloth Sieves," in these sizes:
  - 75 mm (3 in)
  - 50 mm (2 in)
  - 25 mm (1 in)
  - 9.5 mm (3/8 in)
  - 4.75 mm (No. 4)
  - 2 mm (No. 10)
  - 425 μm ( No. 40)
  - 75 μm (No. 200)
- ◆ oven, maintained at  $110 \pm 5$  °C ( $230 \pm 9$  °F)
- ◆ evaporating dishes
- ◆ water bath, or constant temperature room
- ◆ timing device with a second hand
- ◆ beaker, 250 mL (7.5 oz.).



Figure 1 -6. Mechanical Stirring Device.

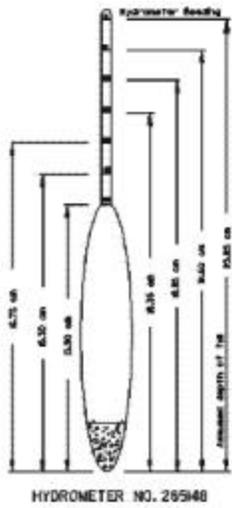


Figure 1 -8. Hydrometer with Dimensions.

## **Material**

- ◆ dispersing agent: A solution of sodium hexametaphosphate shall be used in distilled or demineralized water, at the rate of 40 g of Sodium Hexametaphosphate per liter of solution.
  - Solution of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersing action.
  - Solutions shall be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate.
  - Bottles containing solutions shall have the date of preparation marked on them.
- ◆ distilled or demineralized water
- ◆ source of compressed air, if 'Air Dispersion Device' is used.

## **Determining Composite Correction for Hydrometer Reading**

Equations for percentages of soil remaining in suspension, as given in 'Calculations' of 'Part II, Hydrometer Analysis of Soils Passing 425  $\mu\text{m}$  (No. 40) Sieves' are based on the use of distilled or demineralized water.

A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

The soil hydrometers are calibrated by the manufacturer at 20 °C (68 °F), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

For convenience, a graph or table of composite corrections for a series of 1 degree temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

Prepare 1,000 mL (30 fl. oz.) of liquid composed of distilled water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test.

Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used.

When the temperature of the liquid becomes constant, insert the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151 H the composite correction is the different between this reading and one; for hydrometer 152 H it is the difference between the reading and zero.

Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

## Procedure

### ◆ Hydrometer Analysis

The following table details the steps for hydrometer test.

<b>Hydrometer Analysis</b>	
<b>Step</b>	<b>Action</b>
1	<ul style="list-style-type: none"> <li>◆ Use soil binder prepared in 'Part I' or prepare the soil binder according to Test Method ""TEI-101-E, Preparing Soil and Flexible Base Materials for Testing" if Part I was not used.</li> <li>◆ Record the mass of the air-dried sample as <math>W_A</math> under 'Calculations.'</li> </ul>
2	<ul style="list-style-type: none"> <li>◆ Determine the hygroscopic moisture of the soil binder according to Test Method ""TEI-103-E, Determining Moisture Content in Soil Materials."</li> <li>◆ Record the percent hygroscopic moisture as <math>P_H</math> under 'Calculations.'</li> </ul>
3	Determine the specific gravity of the soil binder according to Test Method ""TEI-108-E, Determining the Specific Gravity of Soils."
4	<ul style="list-style-type: none"> <li>◆ Use a sample splitter to obtain 50 g (for silty or clayed soils) or 100 g (for sandy soil) of soil binder to the nearest 0.01 g.</li> <li>◆ Place the sample in a 250 cc beaker, or in the hydrometer cylinder if the dispersion tube is available.</li> <li>◆ Add approximately 125 cc of the sodium hexametaphosphate solution and cover with distilled water.</li> <li>◆ Stir the soil thoroughly and then allow to soak for at least 12 hours.</li> </ul>
5	<p>After the soaking period, disperse the soil with the stirring device or a soil dispersion tube as follows:</p> <ul style="list-style-type: none"> <li>·· <b>Stirring Device</b> <ul style="list-style-type: none"> <li>● Wash the soil into the dispersion cup and add distilled water until the cup is slightly more than half full.</li> </ul> </li> </ul>

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	<ul style="list-style-type: none"> <li>● Disperse the contents for a period of 1 minute in the mechanical stirring device.</li> <li>● After dispersion, transfer the soil slurry to the hydrometer cylinder and add sufficient distilled water (having the same temperature as the water bath) to bring the level of the water to the 1000 cc mark on the cylinder.</li> <li>● Place the cylinder in the constant temperature water bath.</li> </ul> <p>.. <b>Soil Dispersion Tube</b></p> <ul style="list-style-type: none"> <li>● Add about 300 cc of distilled water to the soaked sample and carefully place the dispersion tube into the hydrometer cylinder.</li> <li>● Adjust the air pressure by means of the valve and disperse the soil.</li> <li>● Disperse the soil-water mixture using an air pressure of 152 kPa (20 psi).</li> <li>● Disperse soils with a PI of 5 or less for 5 minutes; soils with a PI between 6 and 20 for 10 minutes; and soils with a PI greater than 20 for 15 minutes.</li> <li>● Soils containing large percentage of mica need be dispersed for 60 seconds only.</li> <li>● Wash the soil from the dispersion tube into cylinder and add sufficient amount of distilled water to bring the level of the water to the 1000 cc mark before placing into the constant temperature bath.</li> </ul>
6	<ul style="list-style-type: none"> <li>◆ When the soil suspension reaches the temperature of the bath, remove the graduate and thoroughly shake its contents for one minute, using the palm of the hand or a stopper over the open end of the cylinder.</li> <li>◆ Mix the contents of the cylinder by alternately turning the cylinder upside-down, then right-side up and by loosening any material remaining on the bottom while in the inverted position.</li> </ul>
7	<ul style="list-style-type: none"> <li>◆ At the conclusion of this shaking, place the hydrometer cylinder on the table, immediately lower the hydrometer into the suspension and record the time.</li> <li>◆ Read the hydrometer at the peak of the meniscus formed on the stem to the nearest 0.5 g per liter at the end of 2 minutes from the time the graduate was set on the table.</li> <li>◆ Remove the hydrometer and carefully place the cylinder with contents into the constant temperature bath.</li> <li>◆ Obtain hydrometer readings at time intervals of 5, 15, 30, 60, 250, and 1440 minutes after the beginning of sedimentation.</li> <li>◆ About 15 seconds before the time of each reading, slowly and carefully lower the hydrometer into the soil suspension and read the hydrometer after it has come to rest.</li> <li>◆ After each reading, remove the hydrometer from the cylinder in such a manner as to cause as little disturbance as possible.</li> <li>◆ Determine and record the temperature of the suspension each time a hydrometer reading is taken.</li> <li>◆ Record data on work card, to the nearest 0.1%.</li> </ul>

◆ Fine Sieve Analysis

The following table details the sieving steps for the portion of the soil sample from hydrometer test.

<b>Fine Sieve Analysis</b>	
<b>Step</b>	<b>Action</b>
1	At the conclusion of the final hydrometer reading, pour the soil suspension onto a 75 µm (No. 200) sieve and rinse the retained particles with tap water until the wash water is clear.
2	<ul style="list-style-type: none"> <li>◆ Flush any material retained on the 75 µm (No. 200) sieve to an evaporating dish and dry to a constant mass at a temperature of 110 ± 5 °C (230 ± 9 °F).</li> <li>◆ No water should be decanted from the evaporating dish, to avoid loss of material or after the material has settled and the water is clear, carefully decant water from the evaporating dish avoiding any loss of material.</li> <li>◆ A sieve analysis is made using the 425 µm (No. 40) and 75 µm (No. 200) sieves and such other sieves as may be required by the material under test.</li> </ul>

**Calculations**

- ◆ Calculate the percentage of hygroscopic moisture to the nearest 0.001 percent:

$$P_H = 100 \cdot (W_{Air\ Dry} - W_{Ovenr\ Dry}) / W_{Ovenr\ Dry}$$

Where:

- $W_{Air\ Dry}$  = mass of air-dry soil for hygroscopic moisture test, g
  - $W_{Ovenr\ Dry}$  = mass of oven-dry soil for hygroscopic moisture test, g.
- ◆ Calculate the mass of oven-dry soil sample for the hydrometer test:

$$W_O = 100 \cdot W_A / (100 + P_H)$$

Where:

- $W_O$  = mass of oven-dry soil sample for the hydrometer test
  - $P_H$  = percent hygroscopic moisture
  - $W_A$  = mass of air-dry sample for the hydrometer test.
- ◆ Calculate the percent soil binder in the total sample:

$$P_B = 100 \cdot W_S / W_T$$

Where:

- $P_B$  = percent soil binder in the total sample
  - $W_S$  = mass of material passing the 425  $\mu\text{m}$  (No. 40) sieve, g
  - $W_T$  = total mass of the sample, g (as calculated in 'Part I, Sieve Analysis of Material Retained on the 425  $\mu\text{m}$  [No. 40] Sieve').
- ◆ Calculate the percentage of the total original material that is retained on any given fine sieve analysis sieves:

$$\% \text{ of Original Retained} = P_B \cdot W_1 / W_B + (100 - P_B), \text{ etc.}$$

Where:

- $P_B$  = percent soil binder in the total sample
  - $W_B$  = mass of oven-dry soil sample for the hydrometer test, g
  - $W_1$  = mass of portion retained on the first sieve from fine sieve analysis, g.
- ◆ Calculate the percentage of soil in suspension:

$$P_S = (P_B \cdot R \cdot a / W_O)$$

Where:

- $P_S$  = percentage of soil binder in suspension
- $P_B$  = percent soil binder in the total sample
- $R$  = corrected hydrometer reading
- $a$  = constant depending on the density of the suspension and varies with the specific gravity of the soil ( $Gr.$ ), (shown in the 'Constant a' table)
- $W_O$  = mass of oven-dry soil sample for the hydrometer test.

The following table details the 'a' values for different specific gravities.

Constant a	
Specific Gravity, Gr.	Constant, a
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99

2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

- ◆ Calculate the maximum diameter,  $d$ , of the particles in suspension, corresponding to the percentages indicated by a given hydrometer reading, using modified Stoke's Law:

$$d = (K / 1000) \cdot (L / T)^{1/2}$$

Where:

- $L$  = distance from the surface of the suspension is being level at which the density of the suspension is being measured, cm. For a given hydrometer and sedimentation cylinder, the values vary according to the hydrometer readings. These values of distance  $L$ , known as the effective depth, are given in the 'Effective Depth' table.
- $T$  = an interval of time from beginning of sedimentation to the taking of the reading , minutes (2, 5, 15, 30, 60, 250, and 1440 min);
- $K$  = a constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of  $K$  for a range of temperatures and specific gravities are given in the 'Constant  $K$ ' table. The value of  $K$  does not change for a series of readings constituting a test, while values of  $L$  and  $T$  do vary.

## Test Report

Report the total percentages retained on each sieve, or the grain diameter to the nearest whole number, as follows:

- ◆ Sieves, opening in millimeters (inches or sieve numbers):
  - 75 mm (3 in.)
  - 50 mm (2 in.)
  - 25 mm (1 in.)
  - 9.5 mm (3/8 in.)
  - 4.75 mm (No. 4)
  - 2.00 mm (No. 10)
  - 425  $\mu\text{m}$  (No. 40)

- 75  $\mu\text{m}$  (No. 200)
- ◆ Grain diameter:
  - 0.02 mm
  - 0.002 mm
  - 0.001 mm.

The sieve sizes listed above are suggested sizes only and may be specified only in part.

### Plotting Test Results

- ◆ The percentage of grains of different diameters are plotted on semi-logarithmic paper to obtain a grain size accumulation curve.
- ◆ The data obtained from the hydrometer analysis are plotted as percent of material in suspension (% passing) against corrected grain diameter in millimeters.
- ◆ The data from the mechanical analysis are plotted as the percent retained against sieve size.

The following table gives the effective depth, L, based on hydrometer 152 H and sedimentation cylinder of specified sizes.

<b>Effective Depth, L</b>			
<b>Actual Hydrometer reading</b>	<b>Effective Depth, L, cm</b>	<b>Actual Hydrometer reading</b>	<b>Effective Depth, L, cm</b>
0	16.3	31	11.2
1	16.1	32	11.1
2	16.0	33	10.9
3	15.8	34	10.7
4	15.6	35	10.6
5	15.5	36	10.4
6	15.3	37	10.2
7	15.2	38	10.1
8	15.0	39	9.9
9	14.8	40	9.7
10	14.7	41	9.6
11	14.5	42	9.4
12	14.3	43	9.2
13	14.2	44	9.1

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14	14.0	45	8.9
15	13.8	46	8.8
16	13.7	47	8.6
17	13.5	48	8.4
18	13.3	49	8.3
19	13.2	50	8.1
20	13.0	51	7.9
21	12.9	52	7.8
22	12.7	53	7.6
23	12.5	54	7.4
24	12.4	55	7.3
25	12.2	56	7.1
26	12.0	57	7.0
27	11.9	58	6.8
28	11.7	59	6.6
29	11.5	60	6.5
30	11.4	-	--

The following table gives the constant K based on temperature and specific gravity of soil particles.

<b>Constant K</b>									
<b>Temp</b>	<b>Specific Gravity of Soil Particles</b>								
<b>°C</b>	<b>2.45</b>	<b>2.50</b>	<b>2.55</b>	<b>2.60</b>	<b>2.65</b>	<b>2.70</b>	<b>2.75</b>	<b>2.80</b>	<b>2.85</b>
16	15.10	15.05	14.81	14.57	14.35	14.14	13.94	13.74	13.56
17	15.11	14.86	14.62	14.39	14.17	13.96	13.76	13.56	13.38
18	14.92	14.67	14.43	14.21	13.99	13.78	13.59	13.39	13.21
19	14.74	14.49	14.25	14.03	13.82	13.61	13.42	13.23	13.05
20	14.56	14.31	14.08	13.86	13.65	13.44	13.25	13.07	12.89
21	14.38	14.14	13.91	13.69	13.48	13.28	13.09	12.91	12.73
22	14.21	13.97	13.74	13.53	13.32	13.12	12.94	12.76	12.58
23	14.04	13.81	13.58	13.37	13.17	12.97	12.79	12.61	12.43
24	13.88	13.65	13.42	13.21	13.01	12.82	12.64	12.46	12.29
25	13.72	13.49	13.27	13.06	12.86	12.67	12.49	12.32	12.15
26	13.57	13.34	13.12	12.91	12.72	12.53	12.35	12.18	12.01
27	13.42	13.19	12.97	12.77	12.58	12.39	12.21	12.04	11.88
28	13.27	13.04	12.83	12.64	12.44	12.25	12.08	11.91	11.75
29	13.12	12.90	12.69	12.49	12.30	12.12	11.95	11.78	11.62
30	12.98	12.76	12.56	12.36	12.17	11.99	11.82	11.65	11.49

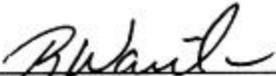




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**Determination of Total Cyanide  
(Macro Distillation Method)  
EPA 335.3/SM4500 CN E**

Prepared by:   
Robert Stevenson  
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Approved by:   
Ronald Warila  
Laboratory Director

Reviewed and  
Implemented by:   
Philip Ruscioni  
Managing Member

Reference:

Methods and Guidance for Analysis of Water, EPA 821-C99-004, Method 335.3, 1978.

Standard Methods for the Examination of Water And Wastewater, SM4500 CN E, 19<sup>th</sup> Edition, 1995.

Code of Federal Regulations 40 Ch. 1 (7-1-99), Part 136.3, page 15, note 20.

**I. Scope and Application:**

Matrix: Drinking water, surface water, mixed domestic and industrial wastewaters.

Regulation: NPDES, RCRA  
SDWA, CWA

The applicable range is 0.005 to 0.500 mg CN/L.

The method detection limit is 0.010 mg CN/L.

**II. Summary of Method**

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a manual reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solutions. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reactions with Chloramine-T, which subsequently reacts with pyridine and barbituric acid to give a red-color complex.



### III. Definitions:

- 3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.
- 3.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.3 **Field Duplicates (FD)** -- Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.3.1 **Sample Duplicates (DUP)** -- A duplicate sample is a second sample aliquot brought through the entire sample preparation and analytical process.
- 3.4 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
- 3.5 **Laboratory Fortified Blank (LFB)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. **The LFB is analyzed exactly like a sample**, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 3.6 **Laboratory Fortified Sample Matrix (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 3.7 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.8 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear( correlation coefficient >0.995).
- 3.9 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
- 3.10 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
- 3.11 **Performance Evaluation Sample (PE)** -- A solution of method analytes distributed by the Quality Assurance Research Division (QARD), Environmental Monitoring Systems Laboratory (EMSL-Cincinnati), U. S. Environmental Protection Agency, Cincinnati, Ohio, to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used by QARD to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.



**3.12 Quality Control Sample (QCS)** – A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

**Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

## IV. Interferences

4.1 For strong acid dissociable and weak acid dissociable cyanide, non-volatile interferences are eliminated or minimized by the distillation procedure.

4.2 Sulfides adversely affect the colorimetric procedure. If a drop of the sample on lead acetate test paper indicates the presence of sulfide, treat 25 mL more of the stabilized sample (pH >12) than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

4.3 Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiocyanide, thiosulfate, and sulfide. Multiple interferences may require the analysis of a series of laboratory fortified sample matrices (LFM) to verify the suitability of the chosen treatment. See Standard Methods section 412A and for details of preliminary sample treatment to remove volatile interferences see Standard Methods 18<sup>th</sup> ed. Sources Section 16.

## V. Safety:

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis.

5.3 The following chemicals have the potential to be highly toxic or hazardous. For detailed explanations consult the MSDS.

- 5.3.1 Cyanide
- 5.3.2 Hydrochloric acid
- 5.3.3 Pyridine

## VI. Equipment and Supplies:

6.1 Balance – analytical, capable of accurately weighing to the nearest 0.0001 g.



6.2 Glassware – Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

6.3 Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.4 Autosampler

6.5 Multichannel proportioning pump

6.6 Reaction unit or manifold

6.7 Colorimetric detector

6.8 Data system

6.9 Heating Unit

6.10 Macro Distillation Setup

## VII. Reagents and Standards:

### 7.1 REAGENTS

**Reagent Water:** Distilled or deionized water, free of the analyte of interest. ASTM Type II or equivalent.

**Ascorbic acid:** Crystal (CASRN-50-81-7)

**Sodium Hydroxide Solution, 1.25 N:** Dissolve 50 g NaOH (CASRN-1310-73-2) in about 600 mL of reagent water in a 1 L beaker. (**Caution:** this is an exothermic reaction producing heated fumes, therefore it must be performed in a hood.) Allow the solution to cool, transfer to a 1 liter volumetric flask, and dilute to the mark with reagent water.

**Sulfamic Acid (H<sub>2</sub>NSO<sub>3</sub>H) Solution, 0.4 N:** Dissolve 40 g H<sub>2</sub>NSO<sub>3</sub>H in reagent water in a 1-liter volumetric flask.

**Sulfuric Acid, 18 N:** Slowly add 500 mL of conc. H<sub>2</sub>SO<sub>4</sub> (CASRN-5329-14-6) to 500 mL of reagent water.

#### **Degassing with helium:**

To prevent bubble formation, degas all instrument reagents listed below except the standards with helium. Use He at 140kPa (20 lb/in<sup>2</sup>) through a clean pipet. Bubble He vigorously through the solution for one minute.

#### **Reagent 1. Carrier, 0.25 M Sodium Hydroxide**

In a 1-L plastic container dissolve **10.0 g NaOH** in **1.00 L** or **1.00 kg water**.



**Reagent 2. Phosphate Buffer, 0.71 M**

**By Volume:** In a 1 L volumetric flask, dissolve **97 g potassium phosphate, monobasic, anhydrous**, ( $\text{KH}_2\text{PO}_4$ ) in approximately **800 mL of water**. Dilute to the mark and invert to mix. Prepare fresh monthly.

**Reagent 3. Chloramine – T**

Dissolve 2.0 g Chloramine – T hydrate in 500 g water. Prepare fresh daily.

**Reagent 4. Pyridine-Barbituric Acid Reagent**

**By Volume:** In the fume hood, place **15.0 g barbituric acid** in a 1 L beaker and add **100 mL water**, rinsing down the sides of the beaker to wet the barbituric acid. Add **75 mL pyridine** ( $\text{C}_5\text{H}_5\text{N}$ ) while stirring and mix until the barbituric acid dissolves. Add **15 mL concentrated hydrochloric acid** (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert to mix. Prepare fresh weekly.

**7.3 PREPARATION OF STANDARDS**

**Standard 1. Stock Standard 1250 mg CN/L**

In a **1 L** volumetric flask, dissolve **2.0 g potassium hydroxide** (KOH) in approximately **500 mL water**. Add **3.129 g potassium cyanide** (KCN). CAUTION: KCN IS HIGHLY TOXIC. AVOID INHALATION OF DUST OR CONTACT WITH THE SOLID OR SOLUTIONS. Dilute to the mark with DI water and invert three times. Prepare fresh weekly or re- standardize weekly.

**DISTILLED STANDARDS**

(Standards that will be taken through the complete distillation procedure with the samples to demonstrate distillation efficiency.)

**Standard 2. Stock Standard 12.5 mg /L CN<sup>-</sup> in Deionized Water**

In a **100 mL** volumetric flask add **1 mL of Standard 1** (1250 mg /L). Dilute to the mark with DI water and invert to mix. Prepare fresh weekly.

These standards **will be** distilled

<b>Working Standards</b> (prepare daily)	<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>E</b>	<b>F</b>
Concentration mg /L CN <sup>-</sup>	0.500	0.300	0.100	0.050	0.010	0.005



**By Volume**

Volume (mL) of <b>stock standard 2</b> Diluted to 500 mL with DI water	20.0	12.0	4.0	2.0	---	---
Volume (mL) of <b>standard A</b> diluted to 500 mL with DI water	---	---	---	---	10.0	5.0

**NON-DISTILLED STANDARDS**

(Standards that are prepared at a 2:1 concentration, and analyzed directly to offset the concentration step performed during a distillation.)

**Distillation Matrix (0.25 M NaOH)**

**By Volume:** In a **1 L** volumetric flask containing approximately **600mL DI water**, add **10 g of sodium hydroxide (NaOH)**. Dilute to the mark and invert to mix.

**Standard 2. Stock Standard 12.5 mg /L CN<sup>-</sup> in Distillation Matrix**

In a **100 mL** volumetric flask add **1 mL of Standard 1** (1250 mg CN<sup>-</sup>/L). Dilute to the mark with **0.25 M NaOH** and invert to mix. Prepare fresh weekly.

These standards **will not** be distilled

<b>Working Standards</b> (prepare daily)	<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>E</b>	<b>F</b>
<i>Actual Concentration mg/L CN<sup>-</sup></i>	1.000	0.600	0.200	0.100	0.020	0.010
Label as: mg/L CN <sup>-</sup> See note below	0.500	0.300	0.100	0.050	0.010	0.005

**By Volume**

Volume (mL) of <b>standard 2</b> diluted to 250 mL with 0.25 M NaOH	20.0	12.0	4.0	2.0	---	---
Volume (mL) of <b>standard A</b> diluted to 250 mL with 0.25 M NaOH	---	---	---	---	5.0	2.5

Note: Following the macro distillation procedure, the cyanide ion present in the original samples is concentrated by a factor of two from the distillation procedure. The initial sample volume is 500 mL; however, the cyanide which has distilled over into the 1.25 M NaOH absorbing solution is diluted to a 250 mL final volume.



Because the non-distilled standards are not carried through the distillation procedure, they are not concentrated by factor of two. To compensate for this factor, the standard concentrations are doubled, but still labeled as the expected concentrations.

## VIII. Sample Collection, Preservation and Storage:

8.1 Samples should be collected in plastic 1-liter bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2 Samples must be preserved with 2 mL of 10 N sodium hydroxide per liter of sample (pH>12) at the time of collection.

8.3 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples must be stored at 4°C.

8.4 Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper; a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6g of ascorbic acid for each liter of sample volume.

8.5 Aldehydes convert cyanide to cyanohydrin. Longer contact times between cyanide and the aldehyde and the higher ratios of aldehyde to cyanide both result in increasing losses of cyanide that are not reversible during analysis. If the presence of aldehydes is suspected, stabilize with NaOH at time of collection and add 2 mL 3.5% ethylene diamine solution per 100 mL of sample.

## IX. Quality Control:

9.1 The minimum requirements for this method consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

### 9.2 Initial Demonstration of Performance:

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by 10%, linearity must be reestablished. If any portion



of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

**9.2.3 Quality Control Sample (QCS)** -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

**9.2.4 Method Detection Limit (MDL)** -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units.

Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]

S = standard deviation of the replicate analyses

MDLs should be determined every six months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 Assessing Laboratory Performance:

**9.3.1 Laboratory Reagent Blank (LRB)** -- The laboratory must analyze at least one LRB with each batch of 20 samples or less. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

**9.3.2 Laboratory Fortified Blank (LFB)** -- At least one LFB with each batch of 20 samples or less. Calculate accuracy as percent recovery (Section 9.4.2). If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

**9.3.3** The LFB analyses data must be used to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery ( $\bar{x}$ ) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \bar{x} + 3S$$

$$\text{LOWER CONTROL LIMIT} = \bar{x} - 3S$$

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to 10 new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to



establish an on-going precision statement for the level of concentrations included in the LFB. This data must be kept on file and be available for review.

At least quarterly, replicates of LFBs should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.

**9.3.4 Instrument Performance Check Solution (IPC)** -- For all determinations the IPC (a mid-range check standard) must be analyzed and a calibration blank immediately following daily calibration, and after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

#### 9.4 Assessing Analyte Recovery and Data Quality:

**9.4.1 Laboratory Fortified Sample Matrix (LFM)** -- The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples.

In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.4.1.1 If the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

9.4.2 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%.

Percent recovery may be calculated using the following equation:

$$R = \frac{C_s - C}{S} \times 100$$

where, R = percent recovery

C = fortified sample concentration

C<sub>s</sub> = sample background concentration

s = concentration equivalent of analyte added to sample

9.4.3 Until sufficient data becomes available (usually a minimum of 20-30 analysis), assess laboratory performance against recovery limits of 90-110%. When sufficient internal performance data becomes available, develop control limits from percent mean recovery and the standard deviation of the mean recovery.

9.4.4 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery



problem encountered with the LFM is judged to be either matrix or solution related, not system related.

9.4.5 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

**9.4.2 Sample Duplicate:** Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of  $\pm 20\%$  for RPD shall be used for sample values greater than 5 times the instrument detection limit. A difference of detection limit is to be used to evaluate samples below 5 times the detection limit.

## X. Calibration and Standardization:

10.1 Prepare a series of 6 standards, as found in Section 7.2, and a blank by diluting suitable volumes of standard solution.

10.2 It is not imperative that all standards be distilled in the same manner as the samples. At least two standards (a mid-level and low-level) and a blank be distilled and compared to similar values on the standard curve to insure that the distillation technique is reliable. If distilled standards do not agree within  $\pm 10\%$  of the undistilled standards the analyst should find the cause of the apparent error before proceeding.

10.3 Set up the manifold for cyanide analysis in a well-ventilated area.

10.4 Allow the instrument to warm up and begin pumping all reagents through the sample lines to establish a stable baseline.

10.5 Place the standards in the autosampler in order of increasing concentration and perform analysis.

10.6 Prepare standard curve by plotting instrument response against concentration values. A calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured value of the calibration solution and the "true value" concentration.

10.7 After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (QCS). If measurements exceed  $\pm 10\%$  of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as an additional continuing calibration check.



## **XI. Procedure**

### **11.1 SAMPLE PRETREATMENT**

#### **11.1.1 Sample Preparation**

Oxidized products of sulfide convert  $\text{CN}^-$  to  $\text{SCN}^-$  rapidly, especially at high pH. Sulfides adversely affect the colorimetric procedure. If a drop of the sample on lead acetate test paper indicates the presence of sulfide by turning black, treat 25 mL more of the stabilized sample (pH >12) than that required (i.e. 525 mL) for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

When particulate, metal cyanide complexes are suspected filter the sample before removing  $\text{S}_2^-$ . Reconstitute sample by returning filtered particulates to the sample after  $\text{S}_2^-$  removal. Homogenize particulates before distillation.

#### **11.1.2 Sample and Standard Distillation**

Samples and Standards are distilled per SM4500 CN C.

### **11.2 SYSTEM START-UP PROCEDURE**

11.2.1 Prepare reagent and standards as described in Section 7.

11.2.2 Set up manifold.

11.2.3 Input data system parameters.

11.2.4 Place samples and/or standards in the autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.

11.2.5 Calibrate the instrument by injecting the standards.

### **11.3 SYSTEM NOTES**

11.3.1 Allow 15 minutes for heating unit to warm up to 60° C.



11.3.2 If sample concentrations are greater than the high standard, the distilled sample should be diluted with 0.25 M sodium hydroxide (NaOH) diluent. When the automated diluter is used, 0.25 M NaOH should be used. **Do not dilute distilled samples or standards with DI water.**

## XII. Data Analysis and Calculations:

12.1 Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by any applicable dilution factor performed during analysis.

### **Important Notes:**

12.1.1 Any dilution of the original sample prior to distillation (i.e. 250 mL of sample + 250 mL of 0.25 N NaOH) must be recorded as a dilution during the distillation procedure, **not** during analysis.

12.1.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.2 Results are reported in mg/L CN<sup>-</sup>.

## XIII. Method Performance:

Current method performance information as presented by the instrument manufacturer is presented in Table 1.

## XIV. Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## XV. Waste Management:

15.1 All waste is handled in accordance with Premier Laboratory's Chemical Hygiene Plan, which is made available to all employees and interested parties.



**SW-846 METHOD 9060**  
**TOC**  
**(SOLID SAMPLES)**



**Standard Operating  
Procedure**

**Standard Procedure for the analysis  
of TOC on solid samples**

Reviewed by: *Stephen Perry* Reviewed Date: \_\_\_\_\_  
Department Analyst

Implemented by: *M. Shu* Date Implemented: 6/2/1999  
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STANDARD OPERATING PROCEDURE  
TOTAL ORGANIC CARBON IN SOLIDS  
SHIMADZU TOC-5000A Analyzer

TABLE OF CONTENTS

<u>Subject</u>	<u>Page</u>
1.0 Scope and Application .....	2
2.0 Summary of Method .....	2
3.0 Interferences .....	3
4.0 Safety .....	3
5.0 Equipment and Materials.....	4
6.0 Reagents and Standards .....	4
7.0 Quality Control .....	5
8.0 Sample Collection.....	8
9.0 Sample Preparation .....	8
10.0 Instrument Startup.....	8
11.0 Data Report Generation.....	11
12.0 Record keeping .....	11
13.0 Waste management.....	11

**STANDARD OPERATING PROCEDURE  
TOTAL ORGANIC CARBON IN SOLIDS  
SHIMADZU TOC-5000A Analyzer**

**1.0 SCOPE AND APPLICATION**

- 1.1 This method is used to determine the concentration of organic and/or inorganic carbon in particulated suspensions, water solutions and solids including samples such as soil, mud and sediments.
- 1.2 The official method detection limit (MDL) has been determined as 100ppm. The instrument measurement range is 0.1mgC-30mgC TC and 0.1mgC-20mgC IC. These values vary with sample type and analysis conditions.

**2.0 SUMMARY OF METHOD**

- 2.1 The solid sample is prepared by homogenizing by means of mixing, pulverizing or cutting in order to achieve a uniform sample. A maximum sample weight of 1 gram is placed in a clean dry sample boat. The boat is transferred into a heated, catalyzed reaction zone in which the carbonaceous matter is converted to carbon dioxide. A flowing gas stream carries the gaseous reaction products through a detector which measures the carbon dioxide content. The detector output is graphically displayed by a recorder. The weight of the peak is proportional to the carbon content of the sample. The proportionality is quantitated by a calibration curve prepared from known carbon content standards.
- 2.2 Since carbon dioxide is liberated from carbonates as well as from organic matter under the total carbon test conditions, carbonate carbon alone is determined in a separate reaction in which the temperature is too low to convert the organic matter to carbon dioxide. The organic carbon can thus be determined by difference. Alternately, the sample may be acidified to a pH of 2 or less with hydrochloric acid and heat treatment is applied to remove the inorganic carbon. The remaining material can then be analyzed for total carbon and thus organic carbon can be determined by virtue of acidification.
- 2.3 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO<sub>2</sub>) by catalytic combustion. The CO<sub>2</sub> formed is then measured directly by an infrared detector. The amount of CO<sub>2</sub> in a sample is directly proportional to the concentration of carbonaceous material in the sample.

### 3.0 INTERFERENCES

- 3.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.
- 3.2 This procedure is applicable only to homogeneous samples which can be introduced into the apparatus reproducibly by means of the boat transfer system.
- 3.3 Removal of carbonate and bicarbonate by acidification and subsequent heating can result in the loss of volatile organic substances.

### 4.0 SAFETY

- 4.1 Certain unknown samples may contain toxic constituents, therefore use the appropriate personnel safety equipment when doing this analysis.
- 4.2 DO NOT measure samples containing mercury, arsenic, or other substances which generate hazardous substances in the exhaust gas, unless the exhaust is properly vented or treated.

### 5.0 EQUIPMENT AND MATERIALS

- 5.1 Shimadzu 500A TOC Analyzer
- 5.2 Apparatus for homogenizing and/or pulverizing the sample.
- 5.3 A mixing spatula and tweezers.
- 5.4 analytical balance capable of measuring 0.100 mg
- 5.5 Sample boats
- 5.6 Drying oven capable of maintaining a temperature of 105°C.
- 5.7 Micropipettor w/ disposable pipettes (100ul to 1000 ul)
- 5.8 IC reaction acid pipetter.
- 5.9 Thermolyne type 30400 furnace.
- 5.10 Analytical Balance capable of four decimal place measurement.

## 6.0 REAGENTS AND STANDARDS

- 6.1 Distilled water used in preparation of standards for dilution of samples should be ultra pure to reduce the carbon concentration of the blank. Carbon dioxide-free, double distilled water is recommended. Ion exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.
- 6.2 Potassium hydrogen phthalate, stock solution, 1000mg carbon/liter: Dissolve 0.2128g of potassium hydrogen phthalate (Primary Standard Grade) in reagent water and dilute to 100ml.
- 6.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solutions by dilution with reagent water.
- 6.4 Carbonate-bicarbonate stock solution, 1000mg carbon/liter: Weigh 0.3500g of sodium bicarbonate and 0.4418g of sodium carbonate and transfer both to the same 100ml volumetric flask. dilute with reagent water.
- 6.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to step 6.3 (1.0%, 0.1%, 0.0% NaHCO<sub>3</sub>)
- 6.5 Dextrose, Anhydrous, Powder reagent.
- 6.6 Dextrose standard solution:  
(10%Carbon) (1.0%Carbon) (0.0%Carbon)
- 6.7 Blank solution: use the same reagent water (or similar quality water) used for the preparation of the standard solutions.
- 6.8 Purity of water - unless otherwise indicated, references to reagent water shall be understood to mean ASTM Type II. Where specified, carbon dioxide-free water is to be prepared by boiling reagent water in a conical flask for 20 min. The boiled water is cooled in the flask that is stoppered with a one-hole rubber stopper fitted to a soda lime-Ascarite drying tube. For larger (10 to 20L) volumes of carbon dioxide-free water, the absorbed carbon dioxide may be removed by inserting a fritted-glass gas-dispersion tube to the bottom of the container and bubbling nitrogen through the water for at least 1 hour. Carbon dioxide-free water may be stored if properly protected from a atmospheric contamination.
- 6.9 Phosphoric Acid, concentrated ACS Reagent Grade

- 6.10 Phosphoric Acid Reagent - make 1:2 dilution of conc  $H_3PO_4$ .
- 6.11 Hydrochloric Acid, concentrated ACS Reagent Grade.
- 6.12 Hydrochloric Acid Reagent - Dilute conc. HCl with reagent water to pH 2.

## 7.0 QUALITY CONTROL

### 7.1 Laboratory Capability

Method Detection Limit > To establish the ability to detect Organic and Inorganic Carbon, the laboratory shall determine the MDL using a blank solution fortified at two to three times the estimated detection limit. To generate an MDL, seven identical aliquots are processed through the analytical method. The results are then entered in the MDL chart for TOC, where the actual MDL is calculated. The MDL generated must be low enough to detect carbon at the levels outlined in compliance monitoring regulations. This MDL is determined annually, unless a change in instrument hardware or operating conditions occurs which would warrant re-evaluation.

### 7.2 Calibration Blanks

For all analyses, run a calibration blank following each calibration, after every 10 or fewer samples, and at the end of the sample run. Analysis of the calibration blank should be less than the MDL.

### 7.3 Initial Calibration Verification

For each analytical run, analyze one ICV immediately following each calibration, and the initial CB. Analysis of the ICV solution should verify the instrument is within  $\pm 5\%$  of calibration.

### 7.4 Continuing Calibration Verification

The CCV sample is analyzed after every 10 or fewer samples, immediately following the Calibration Blank, and at the end of the sample run after the final CB. Analysis of the CCV solution should be within  $\pm 10\%$  of calibration.

## 7.5 Matrix Spikes and Duplicates

To monitor interference caused by the sample matrix, a matrix spike, in duplicate, must be run on a minimum of 5% of all samples in the run. In the case of routine discharges, sites may be rotated so as to eventually spike every discharge. The two sample aliquots shall be spiked with the QC Stock solution.

(A) Calculate the % recovery (R) of carbon in each aliquot using the following equation:

$$R = \frac{(MS - A) \times 100}{S}$$

where;

*MS = Measured concentration of analyte after spiking*

*A = Measured background concentration of sample*

*S = True concentration of the spike*

(B) Compare the % recovery of the carbon spike with the control limit recovery range of 80-120% to determine if the analysis is in control. If it fails, there is either a matrix interference present, bad sampling technique, or a method deficiency, any of which must be corrected before repeating the analysis and declaring the results valid. If the instrument performance is shown to be in control, then the recovery problem is matrix related. The results for the unspiked sample should be labeled to inform the client that the results could be suspect due to matrix effects.

(C) Compare the relative % difference (RPD) between the duplicate results using the following equation:

$$RPD = \frac{|MS - DS|}{(MS + DS)/2} \times 100$$

where;

*MS = Concentration of analyte in the Matrix Spike sample*

*DS = Concentration of analyte in the Duplicate Spike sample*

(D) Compare the RPD results to the control limit ranges also. If the criteria are not met, the system is out of control and the problem must be identified and corrected before declaring the results valid.

## 8.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 8.1 All samples must be collected using a sampling plan that addresses the considerations discussed in this manual.
- 8.2 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.
- 8.3 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between sample collection and the start of analysis should be (4° C) and protected from sunlight and atmospheric oxygen.

## 9.0 SAMPLE and BOAT PREPARATION

- 9.1 For samples containing non-uniform particles, homogenization and blending is imperative.
- 9.2 The sample boats are to be treated (baked) in a heating furnace to oxidize and remove their carbon component. The treatment is performed for 20 minutes or longer at 900 degrees C. The boats are cooled and stored in a clean container.
- 9.3 The sample boats used for IC measurement only are pretreated by dipping in 2N or 3N hydrochloric acid for several minutes and then thoroughly washed and dried for use.
- 9.4 The tweezers and spatula used in the handling of the boats and the sample are heat treated to remove all carbon and stored separately.
- 9.5 TREATMENT FOR REMOVING IC

The following types of samples cannot be measured for IC content:

(a) Samples such as alkaline substances, in which a large amount of phosphoric acid is needed to acidify the sample for IC measurement.

(b) Samples having large particle size, etc., which are not rapidly or completely reacted with phosphoric acid in the IC reaction tube.

(c) Samples having a greater IC content than TOC content: If TOC is determined based on the difference between TC and IC, the margins of error in the TC and IC measurements will have a large impact on the TOC result. For this reason, TOC cannot be measured precisely.

One method of handling such cases is to first remove the IC via pretreatment, and then perform measurements assuming TC=TOC. In general, a reaction with acid is used even in cases where the IC is removed in pretreatment. Using a large amount of acid, it is possible to devote a sufficient amount of time to the reaction. In addition, it is also possible to select, relatively freely, conditions such as heating and agitation, which are used in accelerating reactions. For these reasons, this method can also be applied to the samples described in (a) and (b) above.

There are a variety of pretreatment methods, including one whereby a sufficient amount of hydrochloric acid is added to the sample and heat treatment is applied to remove the IC, after which the sample is dried to remove the excess hydrochloric acid; and one whereby the sample is set in hydrochloric acid gas to remove the IC. For further details, investigate the literature and testing methods in which such methods are described. Since the acid to be used must be removed from the sample during TC measurement with the SSM-5000A, be sure to include a step in which the excess hydrochloric acid is removed at the end of the pretreatment.

#### 9.6 LOADING OF SAMPLES INTO THE SAMPLE BOAT

A balance is used to weigh the sample directly into the sample boat. The weighed sample is set on the bottom of the sample boat toward the front (TC furnace side). In this procedure, the amount of the sample used is in a range close to the upper limit for carbon content which the system can measure in order to: 1) reduce the effects of poor distribution, which tends to occur with solid samples; 2) reduce the degree of weighing error; 3) reduce the impact of external contamination. However, samples which combust extremely rapidly tend to combust less completely as the amount of sample is increased due to lack of oxygen during combustion. For this reason, the amount of sample should be determined in consideration of factors such as the combustion characteristics of each individual sample.

The weighing precision of the balance used in gathering the sample has a direct effect on measurement precision. Thus, an analytical balance or micro-balance should be used, in consideration of factors such as the amount of sample to be weighed and the required measurement precision.

Depending on the type of sample, scattering may occur during combustion. Oxalic acid or glucose crystals, for example, tend to scatter when they are combusted. In such cases, if part of the sample scatters out of the sample boat and enters a low temperature area of the TC furnace, it may not be fully oxidized. Covering the sample boat eliminates such problems. A long, thin cover is fashioned from the supplied ceramic fibers, and is set on top of the sample so as to roughly cover the opening of the sample boat. The ceramic fiber cover prevents the sample from scattering, but also allows good ventilation. In addition, the cover itself is not changed by heat or oxygen.

In measuring the IC of a carbonate such as sodium carbonate, adding phosphoric acid will cause a violent reaction, which may cause part of the sample to scatter outside the sample boat. For this reason, this type of sample should be moistened beforehand with a small amount of water.

## 10.0 INSTRUMENT STARTUP

### 10.1 System Peripherals

This instrument should be operated by a technician who is completely familiar with the manuals supplied by Shimadzu and who is qualified in the operation and analysis of total organic carbon.

### 10.2 Daily Performance Checks for TOC instrument:

- A. Open valve to allow ultra high purity oxygen flow through the TOC analyzer. Verify flow through exhaust tube.
- B. Pressures (80 psi tank) (flow of 0.45 L/min)
- C. Monitor baseline - (adjust as necessary)
- D. Drain separator Water reservoir level - maintain
- E. Phosphoric acid pipetter level

### 10.3 Instrument Power-Up

- 5000A normally on
- in the rare event that the TOC 5000A is not on depress switch located on left side behind the auto sampler
- Self tests will be performed

Note: Be sure PC stand alone switch is in the PC

position.

#### 10.4 Computer Power-Up

- a. Turn on CPU - this will start the monitor and the printer. The computer will perform several self-tests and then autoboot Windows 95.
- b. After the Windows 95 screen comes up, double click the TOC PC icon to start program.
- c. User name box appears - push enter
- d. Expand TOC Control Screen and select measure on the toolbar - click on CONNECT to establish communication between PC and TOC 5050A.
- E. Select Options and click on instrument conditions
- F. Select the SSM tab and X SSM used, SSM TC and IC furnace on. Cell length is W-short. click OK.
- G. Initiate connection of PC to the TOC-5000A. Combustion and reaction furnaces will now heat to 900 degrees C and 200 degrees C respectively.
- H. Select Options and go into Display Settings choose the settings you want to appear in the sample table and press OK.
- I. Sample Table template:  
  
Select Edit from Toolbar and insert Standards, Controls and Samples into the table.
- J. Save information in the data file.

#### 10.5 MEASUREMENT

- A. Commence operation by clicking on Start on vertical toolbar.
- B. Open the sample port cover and place the sample boat containing the first standard in the sample boat holder, then shut the sample port cover firmly. Next, press the START/STOP key, and gently move the sample boat push rod knob to the MEASURING position (limit of push rod travel) to move the sample boat into the TC furnace. During IC measurement, close the sample port firmly, then activate the dispenser to inject the IC reaction acid into the sample boat before moving it.

into the IC furnace. The standard is measured according

to the conditions set on the CALIBRATION/CONDITIONS screen. When COMPLETED is displayed after measurement, return the sample boat push rod to the COOLING position following the instructions on the screen. During IC measurement, return it to the SAMPLE CHANGE position immediately.

- C. For additional measurement, set the weight (or volume) of the sample to be measured additionally in STD WEIGHT (VOLUME), and confirm by ENTER key, and push START/STOP key to show the MEASUREMENT START screen, replace the sample boat with the measured boat, and measure in the same way. After measurement is completed, press the NEXT key. IF a printout is needed, press the print (F5) key.
- B. Make TC Standard curve by introducing <sup>6.0</sup>2.0%, 1.0% and 0.0% Dextrose
- C. Make IC Standard curve by introducing <sup>1.0</sup>5.0%, <sup>0.1</sup>1.0% and 0.0% Sodium Carbonate
- D. Run TC and TC Quality Control samples @ 1000 ppm. (0.10%)
- E. Continue to run samples as outlined above checking calibration curve every ten samples.

## 11.0 CALCULATIONS

- 11.1 Sample introduced into the TOC is combusted (oxidized) to form CO<sub>2</sub>. The CO<sub>2</sub> is transported by the carrier gas into the sample cell of the NDIR detector, where CO<sub>2</sub> is detected. The NDIR detector sends out an analog signal, which generates a peak and is processed by the TOC Control software. The peak is proportional to the concentration the sample.

## 12.0 DATA REPORT GENERATION

- 12.1 To print reports close all windows except the Run Time Report. Click File and Print Table and select print parameters to be reported.
- 12.2 Review the report for QC and Data Guidelines compliance.
- 12.3 Save and Close out the sample file.

### 13.0 RECORDKEEPING

- 13.1 All data files generated by the software are stored on hard disk and hard copies of the runtime reports and results reports are stored in files for reference purposes in accordance with regulatory guidelines.
- 13.2 The NGS Laboratory maintains cumulative QC data in chart form to monitor Blanks, ICV recoveries, MS/DS RSDs, and MDLs, with trend analyses.
- 13.3 The 5000A has a maintenance log that is filled out periodically to keep the instruments in good working order, as well as a repair log kept for problems encountered that require non-routine maintenance or service calls.

### 14.0 WASTE MANAGEMENT

- 14.1 PAL complies with all federal, state, and local regulations pertaining to waste management and disposal, as well as maintaining conservation programs to minimize waste disposal and recycle solvents whenever possible.
- 14.2 Standards are prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed of.
- 14.3 The solvents and reagents used in this method present minimal threat to the environment when managed and disposed of properly.

## ALTERNATE TOC PROCEDURE UTILIZING SAMPLE PRETREATMENT FOR IC REMOVAL

1. Weigh 1.0 g of well mixed sample into a Pyrex beaker.
2. Add 10 ml of 2N HCl. Heat mixture to just below boiling while stirring.
3. Cool and Filter the mixed slurry through a Buchner funnel apparatus and rinse sample with 200 ml of deionized water.
4. Transfer the "dried" filter cake to a suitable container for drying in an oven @ 100 degrees C for 1 hr.
5. Transfer the dried sample into an instrument "boat" and record weight.
6. Run TOC analysis (SW 846 9060) on dried sample.  
Total Carbon remaining = Total Organic Carbon

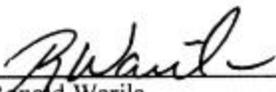


**PCB's**  
Method 8082  
Revision 1.1  
Effective: March 12, 2002

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## **Polychlorinated Biphenyls by Gas Chromatography SW-846 8082**

Prepared by:   
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Approved by:   
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Laboratory Director

Reviewed and  
Implemented by:   
Philip Rusconi  
Managing Member

### **REFERENCE:**

Test Methods for Evaluating Solid Waste, SW-846, Revision 0, December 1996, method 8082.

### **Applicability:**

Analyte: Polychlorinated biphenyls as Aroclors listed in Table 1.  
Matrix: Extracts from solid waste matrices, soils, and aqueous samples  
Regulation: RCRA

### **Important Notes:**

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross contamination.

### **Summary:**

- a) A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.
- b) Liquid samples are extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), or other appropriate technique.
- c) Solid samples are extracted with hexane, hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3545 (pressurized fluid extraction), Method 3550 (ultrasonic extraction), or other appropriate technique.



## PCB's

Method 8082

Revision 1.1

Effective: March 12, 2002

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- d) Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate clean up (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds. Instead, use Method 8081.

### Interferences:

- a) Refer to Methods 3500 (Sec. 3.0, in particular), 3600, and 8000, for a discussion of interferences
- b) Sources of interference in this method can be grouped into three broad categories
- i) Contaminated solvents, reagents, or sample processing hardware.
  - ii) Contaminated GC carrier gas, contaminated injection port, column surfaces, or detector surfaces.
  - iii) Compounds extracted from the sample matrix to which the detector will respond.
- c) Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.
- i) These materials may be removed prior to analysis using Method 3665 (Sulfuric acid/permanganate cleanup).
  - ii) Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations.
  - iii) Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.
  - iv) Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- d) Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment. Do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analysis due to the volatilization and spread of PCBs in the oven.
- e) The presence of elemental sulfur will result in broad peaks that interfere with the detection of early eluting compounds. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur.



## Apparatus And Materials:

- a) Gas chromatograph/dual electron capture detectors/data system.
- b) Chromatography columns: RTX-CLP Pesticides-1 (30m x 0.32 mm I.D. x 0.50  $\mu$ m)  
RTX-CLP Pesticides-2 (30m x 0.32 mm I.D. x 0.25  $\mu$ m)
- c) 10 $\mu$ L, 25 $\mu$ L, 100 $\mu$ L, and 1000 $\mu$ L syringes.

## Reagents:

- a) Stock standard solutions: 1000mg/L. Purchased from commercial suppliers of certified standards.
- b) Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10<sup>0</sup>C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.
- c) Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.
- d) Reagent grade or pesticide grade chemicals shall be used in all tests.
- e) Calibration standards for Aroclors: A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations is used to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors. In addition, the mixture is used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. A minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with isooctane or hexane. Initial Calibration standards are prepared by diluting commercial standards to provide concentrations of 0.2, 1.0, 2.0, 5.0, and 10.0 mg/L. All standards should be stored at -10<sup>0</sup>C to -20<sup>0</sup>C and should be freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard (2.0 mg/L) should be prepared weekly and stored at -10<sup>0</sup>C to -20<sup>0</sup>C.
- f) Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in the above section have been used to demonstrate the linearity of the detector, the single standards of the remaining five Aroclors are used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors at 2.0 mg/L, which corresponds typically to the mid-point of the linear range of the detector.
- g) Surrogate Standards -The performance of the method is monitored using the surrogate compounds: Decachlorobiphenyl and Tetrachlo-m-xylene. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. Control charts must be prepared and updated at least annually to define the surrogate acceptance ranges for all matrices.



## PCB's

Method 8082

Revision 1.1

Effective: March 12, 2002

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### Procedure:

#### SAMPLE PREPARATION :

a) Sample extraction

Refer to Chapter Two of SW-846 and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520), or other appropriate technique. Solid samples are extracted with hexane or hexane-acetone (1:1) using the pressurized fluid extraction (Method 3545), ultrasonic extraction (Method 3550), or other appropriate technique.

b) Extract cleanup- Refer to Methods 3660 ( if a sulfur interference is observed in the preliminary scan), 3665, and 3620.

#### GC OPERATING CONDITIONS:

Column temperature program:	180°C hold for 0 minutes Ramp to 240°C at 11°C/min, hold 0 min Ramp to 300°C at 20°C/min, hold 4.5 min
Detector temperature:	300°C
Injector temperature:	280°C
Injector:	Grob-type, splitless
Sample volume:	2.0 µL
Carrier gas:	Argon/Methane P-5 mix
Column 1	RTX-CLP Pesticides-1 (30m x 0.32 mm I.D. x 0.50 um)
Column 2	RTX-CLP Pesticides-2 (30m x 0.32 mm I.D. x 0.25 um)

#### INITIAL CALIBRATION:

- a) Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.
- b) Calibration standards: Initial Calibration standards containing a mixture of Aroclor 1016 and 1260 are prepared by diluting commercial standards to provide concentrations of 0.2, 1.0, 2.0, 5.0, and 10.0 mg/L. A standard concentration of 0.1 mg/L may also be included if required to meet specific project detection limits. All standards are stored at -10 °C to -20 °C and should be freshly prepared once a year, or sooner if check standards indicate a problem.
- c) A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260,



should they be present in a sample. Therefore, an initial five-point calibration is performed using the mixture of Aroclors 1016 and 1260.

- d) Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards.
- e) Unless otherwise necessary for a specific project, the analysis of the multi-component analytes employs a single point calibration. A single calibration standard near the mid point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column.
- f) A minimum of 3 peaks must be chosen for each Aroclor. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 peaks must include at least one peak that is unique to that Aroclor. Use 6 peaks (3 for 1016, 3 for 1260) for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.
- g) A 2  $\mu\text{L}$  injection volume of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.
- h) External standard calibration: Calculate the calibration factor for each characteristic Aroclor peak at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formula below.

Calculate the calibration factor for each characteristic peak at each concentration:

$$\text{CF} = \frac{\text{Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

Calculate the mean calibration factor for each peak.

Calculate the relative standard deviation (RSD) of the calibration factors for each peak.

Five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the 6 peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least three calibration factors, one for each selected peak.

If the RSD for each peak of the 1016/1260 mixture is <20%, then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a calibration curve or a non-linear calibration model (e.g. a polynomial equation) for quantitation.



The mean calibration factor is acceptable for quantitating sample results when meeting the quality objectives for specific projects if the following is met.

The mean of the RSDs for the 3 peaks of each compound are less than 20% while allowing 1 peak to exceed individually the 20% RSD criteria.

This criteria must be acceptable and directed by the data end user prior to its application.

## Continuing Calibration And Sample Analysis:

- The same GC operating conditions used for the initial calibration must be employed for sample analyses.
- Verify calibration each 12-hour shift by injecting a calibration verification standard of Aroclor 1016/1260 prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is *recommended* to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence.
- The calibration factor for each analyte must not exceed a  $\pm 15$  percent difference from the mean calibration factor calculated for the initial calibration.

$$\% \text{ Difference} = \frac{\left| \text{CF} - \text{mean CF} \right|}{\text{mean CF}} \times 100$$

- If the calibration does not meet the  $\pm 15\%$  limit, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within  $\pm 15\%$ , then a new initial calibration must be prepared.
- Inject a 2- $\mu\text{L}$  aliquot of the concentrated sample extract. Record the volume injected to the nearest 0.05  $\mu\text{L}$  and the resulting peak size in area units.

## QUANTITATION:

Calculate the sample concentration using the mean CF from the initial calibration.

- For aqueous samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_T)(D)}{(\overline{\text{CF}})(V_i)(V_s)}$$

where:



$A_x$  = Area of the peak for the analyte in the sample.

$V_T$  = Total volume of the concentrated extract ( $\mu\text{L}$ ).

$D$  = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made,  $D = 1$ . The dilution factor is always dimensionless.

$\overline{CF}$  = Mean calibration factor from the initial calibration (area/ng).

$V_i$  = Volume of the extract injected ( $\mu\text{L}$ ). The injection volume for samples and calibration standards must be the same.

$V_s$  = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to  $\mu\text{g/L}$ .

b) For non-aqueous samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_T)(D)}{(\overline{CF})(V_i)(W_s)(TS)}$$

where:  $A_x$ ,  $V_T$ ,  $D$ ,  $\overline{CF}$ , and  $V_i$  are the same as for aqueous samples, and

$W_s$  = Weight of sample extracted (g).

$TS$  = % Solids in sample (To report on a dry weight basis)

- c) If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.
- d) When simultaneous analyses are performed from a single injection, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.
- e) Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples.
- f) The results from these bracketing standards must meet the  $\pm 15\%$  continuing calibration verification criteria. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. More frequent analyses of



## PCB's

Method 8082

Revision 1.1

Effective: March 12, 2002

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standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

- g) Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.
- h) The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.
- i) Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.
- j) Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

### Quality Control:

Refer the Premier Laboratory Quality Manual for specific quality control procedures to demonstrate the ability to generate data of acceptable accuracy and precision for the method. This includes but is not limited to the following:

A method detection limit (MDL) study must be completed before samples can be analyzed. The MDL study must be repeated whenever a significant change in the procedure is made.

An initial demonstration of capability (IDC) must be completed by each qualified analyst before samples can be analyzed. The IDC must be repeated whenever a significant change in the procedure is made.

### Method Blank Analysis

- a) A method blank must be prepared and analyzed at a frequency of 1 per batch of samples extracted per matrix, not to exceed 20 samples.
- b) The concentration of each target compound found in the blank must be less than the required quantitation limit for the project.
- c) A solvent blank should be analyzed whenever a new lot of solvent is introduced to check for potential contamination.



### **Matrix Spike/ Laboratory Control Spike Analysis**

- a) A matrix spike and matrix spike duplicate pair must be extracted and analyzed at a frequency of 1 per 20 samples extracted per matrix.
- b) One MS/MSD pair must be extracted and analyzed at least every 30 days for each matrix.
- c) The laboratory must generate MS/MSD recovery data control charts at least annually for each matrix.
- d) The control charts will be used to define acceptable recovery ranges of spike compounds.
- e) If one or more compounds are outside of the control limits then an LCS (QC check standard) must be analyzed to check for matrix interference.
- f) The LCS must meet the laboratory generated acceptance criteria for those compound that failed acceptance criteria in the MS/MSD.

- g) Matrix spike recovery calculation:

$$\% \text{ Recovery} = \frac{\text{spiked sample result} - \text{sample result}}{\text{spike added}} \times 100$$

- h) LCS recovery calculation:

$$\% \text{ Recovery} = \frac{\text{LCS sample result}}{\text{spike added}} \times 100$$

### **Surrogate Recoveries**

- a) Surrogate spike recovery limits must be generated and updated at least annually for each matrix through the use of control charts.
- b) If the recovery of one or more compounds is outside of the control limits, the sample must be re-analyzed. If after re-analysis the recovery is still not within the limits the sample must be re-extracted and re-analyzed. If the re-extracted sample surrogates do not meet the criteria, then the matrix interference problem must be noted in the project case narrative or non-conformance summary.



**PCB's**  
Method 8082  
Revision 1.1  
Effective: March 12, 2002

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**TABLE 1**

CAS No.	Compound	Soil Estimated Quantitation Limits (ug/Kg)	Water Estimated Quantitation Limits (ug/L)
12674-11-2	Aroclor 1016	13.3	0.40
11104-28-2	Aroclor 1221	13.3	0.40
11141-16-5	Aroclor 1232	13.3	0.40
53469-21-9	Aroclor 1242	13.3	0.40
12672-29-6	Aroclor 1248	13.3	0.40
11097-69-1	Aroclor 1254	13.3	0.40
11096-82-5	Aroclor 1260	13.3	0.40



## APPENDIX F

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STANDARD OPERATING PROCEDURES FOR  
ANALYTICAL METHODS

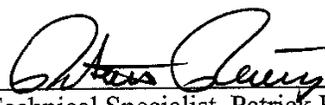
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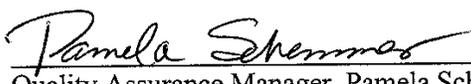
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Revision No. 6.0  
Revision Date: 12/15/02  
Page: 1 of 78

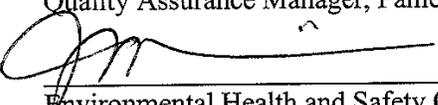
**STL SACRAMENTO STANDARD OPERATING PROCEDURE**  
**TITLE: METHOD 8290 AND TO-9A - POLYCHLORINATED DIOXINS & FURANS**  
**BY HRGC/HRMS**

**(SUPERSEDES: SAC-ID-0005, REVISION 5.0, 7/13/01)**

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<b>1.</b>	<b>SCOPE AND APPLICATION.....</b>	<b>3</b>
<b>2.</b>	<b>SUMMARY OF METHOD.....</b>	<b>3</b>
<b>3.</b>	<b>DEFINITIONS .....</b>	<b>4</b>
<b>4.</b>	<b>INTERFERENCES.....</b>	<b>7</b>
<b>5.</b>	<b>SAFETY .....</b>	<b>8</b>
<b>6.</b>	<b>EQUIPMENT AND SUPPLIES .....</b>	<b>9</b>
<b>7.</b>	<b>REAGENTS AND STANDARDS.....</b>	<b>13</b>
<b>8.</b>	<b>SAMPLE COLLECTION, PRESERVATION AND STORAGE .....</b>	<b>16</b>
<b>9.</b>	<b>QUALITY CONTROL.....</b>	<b>18</b>
<b>10.</b>	<b>CALIBRATION AND STANDARDIZATION .....</b>	<b>22</b>
<b>11.</b>	<b>PROCEDURE.....</b>	<b>31</b>
<b>12.</b>	<b>DATA ANALYSIS AND CALCULATIONS .....</b>	<b>47</b>
<b>13.</b>	<b>METHOD PERFORMANCE .....</b>	<b>52</b>
<b>14.</b>	<b>POLLUTION PREVENTION .....</b>	<b>53</b>
<b>15.</b>	<b>WASTE MANAGEMENT .....</b>	<b>53</b>
<b>16.</b>	<b>REFERENCES.....</b>	<b>54</b>
<b>17.</b>	<b>MISCELLANEOUS (TABLES, APPENDICES, ETC...) .....</b>	<b>54</b>

## 1. SCOPE AND APPLICATION

- 1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.
- 1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.
- 1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).
- 1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A.

## 2. SUMMARY OF METHOD

- 2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques.
- 2.2. If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination. A simplified analysis flow chart is show in Figure 1.
- 2.3. A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (<sup>13</sup>C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent) extraction, for soil, sediment, fly ash

samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction for water samples; c) dilution of a small sample aliquot in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent) extraction for fish tissue.

- 2.4. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20  $\mu\text{L}$  of a tetradecane solution containing 100  $\text{pg}/\mu\text{L}$  of each of the two recovery standards 13C-1,2,3,4-TCDD and 13C-1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF internal standards while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF internal standard percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.
- 2.5. One to two  $\mu\text{L}$  of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).
- 2.6. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a 13C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective internal or recovery standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. All other identified PCDD/PCDF congeners are identified by their relative retention times based on the daily CCV standard, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.
- 2.7. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

### 3. DEFINITIONS

- 3.1. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 2) that contain from one to eight chlorine atoms. The

seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

- 3.2. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.
- 3.3. Isomer: Defined by the arrangement of chlorine atoms within a homologous series. For example, 2,3,7,8-TCDD is a TCDD isomer.
- 3.4. Congener: Any isomer of any homologous series.
- 3.5. Surrogate Standards: A <sup>13</sup>C-labeled analog or mixture of analogs that are added to the sample collection media prior to shipment to the field.
- 3.6. Internal Standard: An internal standard is a <sup>13</sup>C-labeled analog of a congener chosen from the compounds listed in Table 3. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.
- 3.7. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The <sup>13</sup>C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while <sup>13</sup>C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. <sup>13</sup>C-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.
- 3.8. High-Resolution Concentration Calibration Solutions (Table 5): Tetradecane solutions containing known amounts of the 17 2,3,7,8-substituted PCDDs and PCDFs, a minimum of nine internal standards (<sup>13</sup>C-labeled PCDDs/PCDFs), and two carbon-labeled recovery standards; the set of five solutions is used to determine the instrument response of the unlabeled analytes relative to the internal standards and of the internal standards relative to the recovery standards.
- 3.9. Sample Fortification Solution (Table 2): A solution (isooctane or toluene) containing the nine internal standards, which is used to spike all samples before extraction and cleanup.

- 3.10. Recovery Standard Solution (Table 2): A tetradecane solution containing the two recovery standards, which is added to the final sample extract before HRGC/HRMS analysis.
- 3.11. Field Blank: A portion of a sample representative of the matrix under consideration, which is used to assess potential PCDD/F contribution from sampling and transport.
- 3.12. Rinsate: A portion of solvent used to rinse sampling equipment. The rinsate is analyzed to demonstrate that samples were not contaminated during sampling.
- 3.13. GC Column Performance Check Mixture: A tetradecane solution containing a mixture of selected PCDD/PCDF standards including the first and last eluters for each homologous series, which is used to demonstrate continued acceptable performance of the capillary column (i.e.,  $\leq 25$  percent valley separation of 2,3,7,8-TCDD from all the other 21 TCDD isomers) and to define the homologous PCDD/PCDF retention time windows.
- 3.14. Performance Evaluation Materials (PEM's): Representative sample portions containing known amounts of certain unlabeled PCDD/PCDF congeners (in particular the ones having a 2,3,7,8-substitution pattern). Representative interferences may be present. PEMs may be obtained from the EPA EMSL-LV or other sources and submitted to potential contract laboratories, which must analyze these and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). PEMs may also be included as unspecified ("blind") quality control (QC) samples in any sample batch submitted to a laboratory for analysis.
- 3.15. Relative Response Factor: Response of the mass spectrometer to a known amount of a native analyte relative to a known amount of an internal standard, or a known amount of internal standard to a known amount of a recovery standard.
- 3.16. Sample Re-extraction: Extraction of another portion of the sample followed by extract cleanup and extract analysis.
- 3.17. Extract Reanalysis: Instrument analysis by HRGC/HRMS of another aliquot of the final extract.
- 3.18. Tuning (Mass Resolution Check): Standard method used to demonstrate a static resolving power of 10,000 minimum (10 percent valley definition).
- 3.19. Method Calibration Limits: For a given sample size, a final extract volume, and the lowest and highest concentration calibration solutions, the lower and upper calibration limits delineate the region of quantification for which the HRGC/HRMS system was calibrated with standard.

- 3.19. Matrix Spike Fortification Solution: Solution used to prepare the laboratory control sample, matrix spike, and matrix spike duplicate samples. It contains all unlabeled analytes listed in Table 5. The solution also contains all internal standards used in the sample fortification solution per the method.
- 3.20. Definitions of other terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

#### 4. INTERFERENCES

- 4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.
- 4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.
- 4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.
- 4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.
- 4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when the quantitated value for 2,3,7,8-TCDD on the DB-5 column is greater than the Target Detection Limit (TDL), or 1/2 the lower calibration limit.

#### 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

- 5.1. Specific Safety Concerns or Requirements
  - 5.1.1. Hearing protection must be worn when using mechanical systems to grind fish or tissue samples.
  - 5.1.2. When dissecting crawfish abdomens with a scalpel, cut from the hand holding the abdomen toward the tail (away from you).
  - 5.1.3. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.
  - 5.1.4. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.
  - 5.1.5. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff

members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

- 5.1.6. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.
- 5.1.7. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.8. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

## 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Benzene	Flammable Toxic Carcinogen	PEL: 1 PPM TWA ; 5 PPM 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Potassium Hydroxide	Corrosive Poison	2 mg/m <sup>3</sup> ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on the severity of exposure. Symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m <sup>3</sup>	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m <sup>3</sup>	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

## 6. EQUIPMENT AND SUPPLIES

### 6.1. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

- 6.1.1. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2- $\mu$ L injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2  $\mu$ L). 1  $\mu$ L injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.
- 6.1.2. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause

peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

- 6.1.3. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.
- 6.1.4. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

## 6.2. GC Column

- 6.2.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when the quantitated value for 2,3,7,8-TCDF is greater than the TDL.
- 6.2.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

### 6.3. Miscellaneous Equipment and Materials

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

- 6.3.1. Nitrogen evaporation apparatus with variable flow rate.
- 6.3.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.
- 6.3.3. Centrifuge.
- 6.3.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within  $\pm 2^{\circ}\text{C}$ .
- 6.3.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.
- 6.3.6. Drying oven.
- 6.3.7. Stainless steel spoons and spatulas.
- 6.3.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.
- 6.3.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.
- 6.3.10. Reacti-vial, 2 mL, silanized clear glass.
- 6.3.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.
- 6.3.12. Separatory funnels, 250 mL.
- 6.3.13. Separatory funnels, 1000 mL.
- 6.3.14. Teflon® boiling chips (or equivalent) washed with DCM before use.
- 6.3.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.
- 6.3.16. Adapters for concentrator tubes.
- 6.3.17. Glass fiber filters.

- 6.3.18. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.
- 6.3.19. Continuous liquid-liquid extractor.
- 6.3.20. All-glass Soxhlet apparatus, 500 mL flask.
- 6.3.21. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.
- 6.3.22. Glass funnels, sized to hold 170 mL of liquid.
- 6.3.23. Desiccator.
- 6.3.24. Turbo evaporator
- 6.3.25. Rotary evaporator with a temperature controlled water bath.
- 6.3.26. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.
- 6.3.27. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

***Note:** Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:*

- 6.4. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.
  - 6.4.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.
  - 6.4.2. After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.
  - 6.4.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of

active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

- 6.4.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

## 7. REAGENTS AND STANDARDS

### 7.1. Column Chromatography Reagents

- 7.1.1. Silica Gel - Kieselgel 60 or equivalent, activate for 1 hour at 184°C before use. Store at 130°C in covered flask.
- 7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.
- 7.1.3. Basic Alumina - ICN or equivalent. No activation required.
- 7.1.4. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon<sup>®</sup> lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.
- 7.1.5. 44% H<sub>2</sub>SO<sub>4</sub>/silica gel - Mix 24 mL conc. H<sub>2</sub>SO<sub>4</sub> and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.
- 7.1.6. 33% NaOH/silica gel - Mix 34 mL 1N NaOH and 67 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

### 7.2. Reagents

- 7.2.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.
- 7.2.2. Potassium hydroxide, ACS grade, 20 percent (w/v) in distilled water.
- 7.2.3. Distilled water demonstrated to be free of interferences
- 7.2.4. Potassium carbonate, anhydrous, analytical reagent.
- 7.2.5. Silica gel.

- 7.2.6. Solution for breaking emulsions: Slowly add 1.0L of reagent grade NaOH solution to a 2.0L NaOH container, containing 1.0L of DI H<sub>2</sub>O, and leave the container in secondary containment with the lid off. **Warning: *The solution will begin to heat so let the solution stand until equilibrium is met and the solution is at room temperature.*** When this process is complete, the solution will then be ready for use in the samples.
- 7.2.7. Precleaned Ottawa sand (minimum 4 hours toluene Soxhlet Extracted).
- 7.2.8. Canola Oil (for tissue extraction only).
- 7.3. Desiccating Agent
- 7.3.1. Sodium sulfate, granular, anhydrous.
- 7.4. Solvents
- 7.4.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, benzene, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.
- 7.5. All calibration, daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.
- 7.5.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.
- 7.6. Calibration Solutions
- 7.6.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/μL) and the highest for the octachlorinated congeners (2000 pg/μL).
- 7.6.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

- 7.6.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

#### 7.7. GC Column Performance Check Solution

- 7.7.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The 13C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/μL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.
- 7.7.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of  $\leq 25\%$ . Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

#### 7.8. Field Surrogate Solution (air matrices)

- 7.8.1. This solution contains one <sup>37</sup>Cl labeled analog (for Method TO-9/TO-9A) or one <sup>37</sup>C and four <sup>13</sup>C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

#### 7.9. Sample Fortification Solution (Internal Standard)

- 7.9.1. This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that <sup>13</sup>C-OCDF is not present in the solution.)

#### 7.10. Recovery Standard Solution

- 7.10.1. This tetradecane solution contains two recovery standards (13C-1,2,3,4-TCDD and 13C-1,2,3,7,8,HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and

HRGC/HRMS analysis.

7.11. Preparation and QC of PUF material

- 7.11.1. The PUF material is purchased pre-cut.
- 7.11.2. The PUFs are rinsed by Soxhlet with toluene (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.
- 7.11.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.
- 7.11.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.
- 7.11.5. The 1613/8290 daily internal standard solution is spiked into the PUF and it is extracted for a minimum of 16 hours.
- 7.11.6. The Soxhlet extract is recovered and processed according to section 11.4.
- 7.11.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

**8. SAMPLE COLLECTION, PRESERVATION AND STORAGE**

- 8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.
- 8.2. Grab and composite samples must be collected in glass containers.
- 8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.
- 8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See SAC-ID-0009 for sample preparation procedures).

- 8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.
- 8.6. Grinding or blending of fish samples.
- 8.6.1. If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3- to 5-mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded.
- 8.6.2. **Warning: *Hearing protection must be worn when grinding samples.***
- 8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.
- 8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C).
- 8.9. Soil, Sediment or Paper Sludge (Pulp) Percent Moisture Determination.  
The percent moisture of soil or sediment samples showing detectable levels (see note below) of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined according to the following recommended procedure.

Generally, depending on sample availability, a 5-10 g sample, weighed to three significant figures, is used for % solids determination. The sample is then dried to constant weight at 110°C ± 10 in an adequately ventilated oven. Weigh the dried solid to three significant figures. Calculate and report the percent moisture on the appropriate form. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

$$\text{Percent Moisture} = \frac{\text{Weight of wet soil} - \text{Weight of dry soil}}{\text{Weight of wet soil}} \times 100$$

- 8.10. Fish Tissue Lipid Content Determination

The percent lipid of fish samples is determined as follows:

Concentrate the extract from Section 11.3.5 on a rotary evaporator until constant weight is attained. The percent lipid is calculated using the following expression:

$$\text{Percent lipid} = \frac{\text{Weight of residue from extraction (in g)}}{\text{Weight of fish tissue portion (in g)}} \times 100$$

## 9. QUALITY CONTROL

- 9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

- 9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.
- 9.1.2. Use Ottawa sand as the method laboratory matrix when solids are extracted. Use a mixture of Ottawa sand and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.
- 9.1.3. The method blank must be spiked prior to extraction with the same amount of <sup>13</sup>C-labeled internal standards as added to samples.
- 9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.
- 9.1.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data

appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples  $>10x$  the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy QA-003-SAC for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. An MS/MSD pair

are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

- 9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.
- 9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.
- 9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").
- 9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.
- 9.3.5. Analyze the MS and MSD samples as described in Section 11.
- 9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.
- 9.3.7. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

#### 9.4. Duplicates

- 9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative

samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

## 9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of internal standard to yield 100 pg/ $\mu$ L in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11.3. As applicable, add the appropriate amount of recovery standard to yield 100 pg/ $\mu$ L in the final extract. Analyze a 1-2  $\mu$ L aliquot of the concentrated extract.

9.5.1.3. Calculate the concentration of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards.

## 9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

9.6.2.1. Take a 100-mL ( $\pm$  0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of internal standard to yield 100 pg/ $\mu$ L in the final extract.

- 9.6.3. Using appropriate methods, concentrate to approximately 10 mL.
  - 9.6.4. Just before analysis, add the appropriate amount of recovery standard to yield 100 pg/ $\mu$ L in the final extract. Reduce the volume to a final volume of 20  $\mu$ L, as necessary. No column chromatography is required.
  - 9.6.5. Analyze an aliquot following the same procedures used to analyze samples.
  - 9.6.6. Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in pg/mL of rinsate solvent.
- 9.7. Surrogate/Clean Up Recovery Standard
- 9.7.1. A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.
- 9.8. Internal Standards
- 9.8.1. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.
  - 9.8.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.
  - 9.8.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.
- 9.9. Recommended Corrective Actions and Troubleshooting Steps
- Verify satisfactory instrument performance.
  - If possible, verify that no error was made while weighing the sample portions.
  - Review the analytical procedures with the performing laboratory personnel.

## 10. CALIBRATION AND STANDARDIZATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

## 10.1. Tuning (Mass Resolution Check)

- 10.1.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.
- 10.1.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

*NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.*

- 10.1.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at  $m/z$  304.9824 (PFK) or any other reference signal close to  $m/z$  303.9016 (from TCDF). Verify that the exact mass of  $m/z$  380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).
- 10.1.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal ( $m/z$  380.9760). The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-

mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 5) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at  $m/z$  380.9760 (or 0.038 amu at that particular mass).

## 10.2. Performance Checks

10.2.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 3 (HRCC-3) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-2 or HRCC-4 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.2.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.2.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for  $^{13}\text{C}$ -HxCDF and  $^{13}\text{C}$ -HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

- 10.2.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

### 10.3. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

- 10.3.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.
- 10.3.2. Tune the instrument with PFK.
- 10.3.3. Inject 1 or 2  $\mu\text{L}$  of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be  $\leq 1$  second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.
  - 10.3.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.
  - 10.3.3.2. The same injection volume must be used for all samples, QC, and standards.
- 10.3.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or 2- $\mu\text{L}$  portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.
  - 10.3.4.1. The total cycle time for data acquisition must be  $\leq 1$  second. The total cycle time includes the sum of all dwell times and voltage reset times.

- 10.3.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.
- 10.3.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).
- 10.3.4.4. The ratio of integrated ion current for the ions belonging to the <sup>13</sup>C labeled internal and recovery standards must be within the control limits stipulated in Table 9.

*NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.*

- 10.3.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

- 10.3.5.1. Referring to Table 10, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate internal standards (Table 5) and the nine RRFs for the labeled <sup>13</sup>C internal standards [RRF(m); m=18 to 26] relative to the two recovery standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

$$RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

Where:

$A_x$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for unlabeled PCDDs/PCDFs,

$A_{is}$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

$A_{rs}$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled recovery standards,

$Q_{is}$  = quantity of the internal standard injected (pg),

$Q_{rs}$  = quantity of the recovery standard injected (pg), and

$Q_x$  = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express  $Q_{is}$ ,  $Q_{rs}$ , and  $Q_x$  must be the same.

- 10.3.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = \left(\frac{1}{5}\right) \sum_{j=1}^5 RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 10), and j is the injection number (or calibration solution number; j = 1 to 5).

- 10.3.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 10) are calculated as follows:

- 10.3.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain 13C-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the  $[M+6]^+$  ion of 13C-OCDF from the  $[M+2]^+$  ion of OCDD (and  $[M+4]^+$  from 13C-OCDF with  $[M]^+$  of OCDD). Therefore, the RRF for OCDF is calculated relative to 13C-OCDD.

10.3.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = \left(\frac{1}{t}\right) \sum_{n=1}^t RRF_n$$

Where:

k = 27 to 30 (Table 10), with 27 = PeCDF;  
28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,  
t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.3.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RRF(m) = \frac{A_{is}^m \times Q_{rs}}{Q_{is}^m \times A_{rs}}$$

$$\overline{RRF}(m) = \left(\frac{1}{5}\right) \sum_{j=1}^5 RRF_j(m)$$

Where:

m = 18 to 26 (congener type)  
j = 1 to 5 (injection number),  
A<sub>is</sub><sup>m</sup> = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),

- $A_{rs}$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard ( $m = 18$  to 26),
- $Q_{rs}$  &  $Q_{is}^m$  = quantities of, respectively, the recovery standard (rs) and a particular internal standard (m) injected (pg),
- $RRF(m)$  = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and
- $\overline{RRF(m)}$  = calculated mean relative response factor of a particular internal standard, as determined from the five initial calibration injections (j).

#### 10.4. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

- 10.4.1. The percent relative standard deviations for the mean response factors [ $RRF(n)$  and  $RRF(m)$ ] from the 17 unlabeled standards must be  $\leq 20$  percent, and those for the nine labeled reference compounds must be  $\leq 30$  percent.
- 10.4.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be  $\geq 10$ .
- 10.4.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

#### 10.5. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall

be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.5.1. Inject 1 or 2  $\mu\text{L}$  of the concentration calibration solution HRCC-3 containing 10  $\text{pg}/\mu\text{L}$  of tetrachlorinated congeners, 50  $\text{pg}/\mu\text{L}$  of penta-, hexa-, and heptachlorinated congeners, 100  $\text{pg}/\mu\text{L}$  of octachlorinated congeners, and the respective internal and recovery standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

#### 10.6. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.6.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be  $\pm 20$  percent of the mean values established during the initial calibration (Section 10.3.5.)

10.6.1.1. The bracketing continuing calibration must be  $\pm 20\%$  of the average RRF calculated from the initial calibration.

10.6.1.1.1. If the target compounds in the ending standard are less than or equal to  $\pm 20\%$  of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.6.1.1.2. If the target analytes are greater than  $\pm 20\%$  but less or equal to  $\pm 25\%$  and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than  $\pm 20\%$  but less or equal to  $\pm 25\%$  and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.6.1.1.3. If the percent deviation of unlabeled compounds exceeds  $\pm 25\%$ , a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.6.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the

opening continuing calibration must be less than or equal to  $\pm 30$  percent of the mean values established during the initial calibration (Section 10.1.5).

10.6.2.1. The bracketing continuing calibration must be  $\pm 30\%$  of the average RRF calculated from the initial calibration.

10.6.2.1.1. If the labelled compounds in the ending standard are less than or equal to  $\pm 30\%$  of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.6.2.1.2. If the internal standard analytes are greater than  $\pm 30\%$  but less or equal to  $\pm 35\%$ , an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.6.2.1.3. If the percent deviation of labeled compounds exceeds  $\pm 35\%$ , reanalyze samples if adversely impacted.

10.6.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.6.4. If either criteria in Sections 10.6.1 or 10.6.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.3.4.4 for resolution.

10.6.5. If either one of the above criteria (Sections 10.6.1 and 10.6.2) cannot be satisfied, the entire initial calibration process (Section 10.3) must be repeated.

## 11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

### 11.3. Extraction

11.3.1. Internal Standard Addition. A 2000 pg aliquot of the internal standard mixture is added to all samples, regardless of sample size. As an example, for 13C-2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of 13C-2,3,7,8-TCDD to give the requisite fortification level.

11.3.2. Sludges. Paper Pulp Sludges are generally air-dried and ground. Because of the drying procedure, a Dean-Stark water separator is optional for extraction. Extraction is generally done by Soxhlet with 200-300 mL of toluene (150mL of toluene using the Soxtherm or equivalent).

11.3.2.1. Non-Paper Pulp Sludges are extracted with 200-300 mL of toluene (150mL of toluene using the Soxtherm or equivalent).

11.3.2.2. Extract the sample for a minimum of 16 hours (2 hours using the Soxtherm or equivalent). Cool the sample, filter the toluene extract, if needed, through a glass-fiber filter or equivalent into a round-bottom flask. Rinse the filter with 10 mL toluene, and combine the extract and rinsate. Concentrate the combined solutions to near dryness on a rotary evaporator at 50°C. Use of an inert gas to concentrate the extract is also permitted. Proceed with Section 11.6 as necessary.

11.3.3. Still-Bottom/Fuel Oil. All organic liquids, fuel oils, and solids that will dissolve in a solvent and are treated as a solvent dilution will be dissolved in 1-2 mL of an appropriate solvent; then diluted with 5-10 mL of hexane if no acid wash is required. Spike with appropriate Internal Standards and proceed with Section 11.6 as necessary.

11.3.4. Fly Ash. Extract fly ash samples by jar shake with hydrochloric acid before Soxhlet (or Soxtherm or equivalent) extraction. Weigh 2-10g of sample aliquot into a clean glass jar. Add 1.0mL of the internal standard mixture with 2 mL of acetone. Add 150 mL of 1N hydrochloric acid and shake for 4 hours. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking. The contents of the jar are then filtered through a glass fiber filter. The solids are Soxhlet extracted for 16 hours (2 hours using the Soxtherm or equivalent). The aqueous filtrate is extracted with 100 mL of toluene and twice with 100 mL of hexane. Concentrate the combined toluene

solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.6 as necessary.

*Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.*

- 11.3.5. Solids/Tissues. Add anhydrous sodium sulfate to the soil sample portion in a ratio of 2 to 1 (e.g. 20 g sodium sulfate to 10 g of sample) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass-wool plug (the use of an extraction thimble is optional). Add 200 to 250 mL toluene to the Soxhlet apparatus and reflux for 16 hours (150mL of toluene and 2 hour extraction using the Soxtherm or equivalent). The solvent must cycle completely through the system at least five times per hour. Proceed with Section 11.6.

Note: The matrix for the tissue method blank consists of 9g of pre-cleaned Ottawa sand and 1g of canola oil.

- 11.3.6. Preparation of stationary source samples. See SOP SAC-ID-0009.

#### 11.4. Preparation of crawfish samples

- 11.4.1. Prior to dissecting the crawfish, rinse the animal with reagent water.
- 11.4.2. Isolate the crawfish abdomen by manually pulling the abdomen away from the cephalothorax.
- 11.4.3. Using forceps, hold the abdomen down and make a ventral incision of the cuticle along the full length of the abdomen midline using either dissection scissors or a scalpel blade. If using a scalpel blade, cut from the head toward the tail.
- 11.4.4. Hold open the abdomen midline incision and remove the abdominal flexor muscle (tail meat) with forceps and place in a weighing boat.
- 11.4.5. If any of the anterior portion of the abdominal flexor muscle remained in the cephalothorax, remove it with forceps and combine with the muscle portion from 11.4.4. The combined portion should yield 3-6 grams of meat.
- 11.4.6. With a spatula, remove a portion of the hepatopancreous from the cephalothorax. An attempt should be made to recover as much of the hepatopancreous as possible. This should yield a few hundred milligrams of tissue. (Test yielded approximately 0.5-0.8 g wet weight of hepatopancreous from medium sized crawfish.)

- 11.4.7. Combine the hepatopancreous with the abdominal flexor muscle tissue. (Tests yielded approximately 5 g total wet weight of combined tissue per crawfish specimen using 12-18 g animals.)
- 11.4.8. Repeat steps 11.4.1 through 11.4.6 with additional crawfish specimens and composite the tissue until sufficient tissue mass is acquired for sample preparation and analysis as specified in the appropriate laboratory SOPs. (The composited tissue may be required for more than just the Dioxin analysis described in this SOP).
- 11.4.9. Record the composited tissue mass collected.

Note: The laboratory may elect to perform a combination of Dioxin/Furans and PCBs, in which case a 20 gram aliquot of the sample will be extracted and split prior to extract cleanup.

- 11.4.10. Homogenize the composited tissue and proceed with removal of a sample aliquot for extraction and analysis as directed in Section 11.4.11.
- 11.4.11. Weigh approximately a 10 gram aliquot into an extraction thimble and record the sample weight on the extraction sheet.
- 11.4.12. Place the extraction thimble containing the sample in a pre-cleaned Soxhlet extractor charged with 200-220 mL of fresh toluene and boiling chips (150mL toluene and 2 hour extraction using the Soxtherm or equivalent).
- 11.4.13. Proceed with the extraction according to section 11.3.5.

Note: If a % lipid determination is required, a tared round bottom flask is used. There is no solvent keeper added prior to extract concentration by rotary evaporation and the sample is reduced to dryness and reweighed according to section 8.11. The sample is then redissolved in hexane and process to section 11.6.2.

- 11.4.14. Aqueous Samples. Weigh the sample bottle. Also, mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Pour the entire sample (approximately 1-L) into a 2-L separatory funnel.
  - 11.4.14.1. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel. The internal standard mixture is

first mixed in 2 mL of acetone then it is added to the sample in the separatory funnel. Each aliquot of spike mixture is added similarly. Extract the sample by shaking the funnel for two minutes with periodic venting. **Warning: DCM will rapidly build up pressure. The funnel should vented initially after a few shakes.** Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. Extraction is repeated two additional times with methylene chloride. **Warning: Separatory funnel extraction is a high risk activity. Analyst will wear a face shield over safety glasses/goggles for this extraction. Alternatively, the extraction can take place behind a closed hood sash.**

- 11.4.14.2. Determine the original sample volume by re-weighing the sample bottle, or filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.
- 11.4.14.3. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with DCM and load funnel with DCM-rinsed  $\text{Na}_2\text{SO}_4$ . Pour extract through  $\text{Na}_2\text{SO}_4$  to remove water. Rinse  $\text{Na}_2\text{SO}_4$  with fresh DCM and collect in round bottom flask.
- 11.4.14.4. Transfer the extract to a 500-mL round-bottom, add approximately 100  $\mu\text{L}$  of tetradecane and concentrate on a rotary evaporator or TurboVap.
- 11.4.15. A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel.
  - 11.4.15.1. Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface.
  - 11.4.15.2. Transfer the solvent to the extractor.
  - 11.4.15.3. Repeat the sample bottle rinse with an additional 50- to 100-mL portion of methylene chloride and add the rinsate to the extractor.

11.4.15.4. Add 200 to 500 mL methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 hours.

11.4.15.5. Allow to cool, then detach the distilling flask.

#### 11.4.16. Filter/PUF Samples

11.4.16.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.4.16.2. Add 2 mL (4000 pg) of 1613/8290 Internal Standard solution to all samples and QC.

11.4.16.3. Add 50 mL of 1613/8290 Native Spike to the LCS.

11.4.16.4. Extract the samples and QC for a minimum of 16 hours.

11.4.16.5. Concentrate the extract from the round bottom flask with hexane and adjust the volume.

11.4.16.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.4.16.7. Split the extract 50:50 for analysis and archive.

11.4.16.8. Proceed to Section 11.6.

#### 11.4.17. Wipe Extractions

##### 11.4.17.1. Jar Shake Method

11.4.17.1.1. Place a pre-cleaned wipe in a French Square jar. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another French Square. This will be the LCS aliquot.

11.4.17.1.2. Transfer each wipe sample and all accompanying liquid into separate French Square jars or appropriate sized containers (Note: If the container used to deliver the wipe to the laboratory can contain 100 ml of solvent, then the container can be used for the extraction).

11.4.17.1.3. Spike all samples, method blank and LCS with an appropriate amount of internal standard. Additionally, spike the LCS with the appropriate amount of native standard.

11.4.17.1.4. Add 100 ml of toluene to each jar and secure Teflon lined cap. Place the closed container onto the flatbed shaker and secure in place. Turn the shaker on such that the shaking motion is aggressive enough to move the liquid through the wipe. Shake for 4 hours.

11.4.17.1.5. Filter each sample through a filter funnel with a glasswool plug. Capture the sample into a 500 ml round bottom flask. Add approximately 100  $\mu$ L of tetradecane and concentrate to approximately 100  $\mu$ L on a rotary evaporator or TurboVap.

11.4.17.1.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.4.17.1.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take  $\frac{1}{2}$  of each sample (or an appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.4.17.1.7.1. If only one analysis is required, then  $\frac{1}{2}$  of the sample is archived and the other half is analyzed.

11.4.17.1.7.2. If three analyses are required, then  $\frac{1}{3}$  is archived  $\frac{1}{3}$  is used for one test and  $\frac{1}{3}$  is used for the third test. For additional analyses, adjust the fractions accordingly.

#### 11.4.17.2. Soxhlet Method

- 11.4.17.2.1. Place a pre-cleaned wipe in a precleaned Soxhlet extraction apparatus. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another precleaned Soxhlet extraction apparatus. This will be the LCS aliquot.
- 11.4.17.2.2. Transfer each wipe sample and all accompanying liquid into separate precleaned Soxhlet extraction apparatus.
- 11.4.17.2.3. Spike all samples, method blank and LCS with an appropriate amount of internal standard. Additionally, spike the LCS with the appropriate amount of native standard.
- 11.4.17.2.4. Charge the Soxhlet with approximately 300 mL toluene and completely assemble the Soxhlet apparatus. Turn the temperature to the appropriate setting to cycle the toluene. Cycle for 16 hours.
- 11.4.17.2.5. After cycling is complete, turn heat off and allow glassware to cool. Remove round bottom flask. Add approximately 100  $\mu$ L of tetradecane and concentrate to approximately 100  $\mu$ L on a rotary evaporator or TurboVap.
- 11.4.17.2.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.
- 11.4.17.2.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take  $\frac{1}{2}$  of each sample (or an appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.4.17.2.7.1. If only one analysis is required, then  $\frac{1}{2}$  of the sample is archived and the other half is analyzed.

11.4.17.2.7.2. If three analyses are required, then  $\frac{1}{3}$  is archived  $\frac{1}{3}$  is used for one test and  $\frac{1}{3}$  is used for the third test. For additional analyses, adjust the fractions accordingly.

11.5. There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with DCM. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 1:1 NaOH/H<sub>2</sub>O solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. See section 7.2 for reagent preparation.

#### 11.5.1. Breaking Emulsions:

- 11.5.1.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.
- 11.5.1.2. Pour approximately 100 mL of the 1:1 NaOH:H<sub>2</sub>O into a 1.0L AGB.
- 11.5.1.3. Drain the sample with the emulsion from the 2.0L separatory funnel into the 1L AGB and let it stand.
- 11.5.1.4. Empty the aqueous waste into the LLE waste drum.
- 11.5.1.5. Pour the solution with DCM back into the same 2.0L separatory funnel and drain the DCM phase through Na<sub>2</sub>SO<sub>4</sub> into a 500mL round-bottom flask.
- 11.5.1.6. Empty the aqueous waste into the LLE waste drum.
- 11.5.1.7. Proceed with section 11.6.

## 11.6. Optional Extract Clean-Ups

For all samples which are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC Extracts (See also Section 9.71)

### 11.6.1. Acid/Base Partitioning

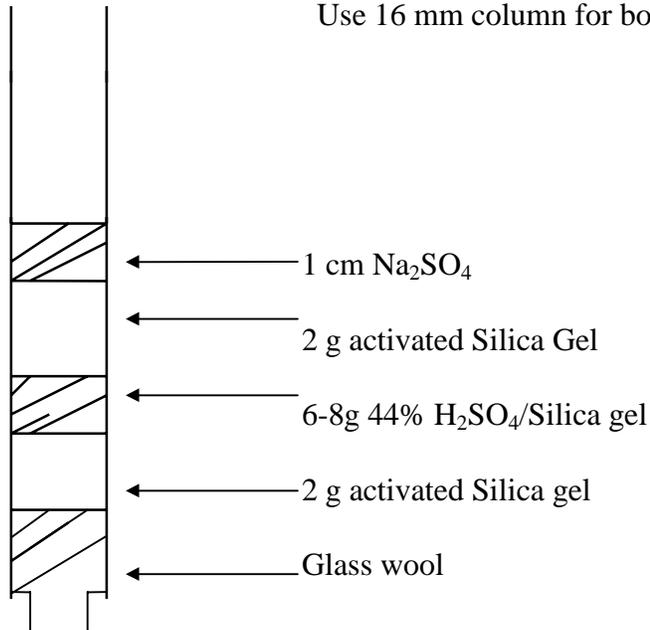
- 11.6.1.1. Partition the extract in 50-125 mL of hexane against 40 mL concentrated H<sub>2</sub>SO<sub>4</sub> in a separatory funnel. Shake for two minutes. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings). **Warning: *Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.***
- 11.6.1.2. Partition the extract against 50 mL distilled H<sub>2</sub>O. Shake for two minutes. Remove and discard the aqueous layer (bottom). If further cleanup is required, proceed to section 11.6.1.3
- 11.6.1.3. Partition the extract using 50 mL of 10 N NaOH. Shake for two minutes. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base is known to degrade certain PCDDs/PCDFs, contact time must be minimized. The NaOH partition is applied only as samples warrant it at the discretion of the analyst. **Warning: *Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.***
- 11.6.1.4. Partition the extract against 50 mL of distilled H<sub>2</sub>O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15-mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.) The DI H<sub>2</sub>O partition is applied only as samples warrant it at the discretion of the analyst.

## 11.6.2. Silica Column Clean-Up “Exhibit A”

EXHIBIT A  
IFB Column Clean-up

Use 20 mm column for top column

Use 16 mm column for bottom column\*



\*Upper and lower columns are piggy backed – See Exhibit B for lower column packing

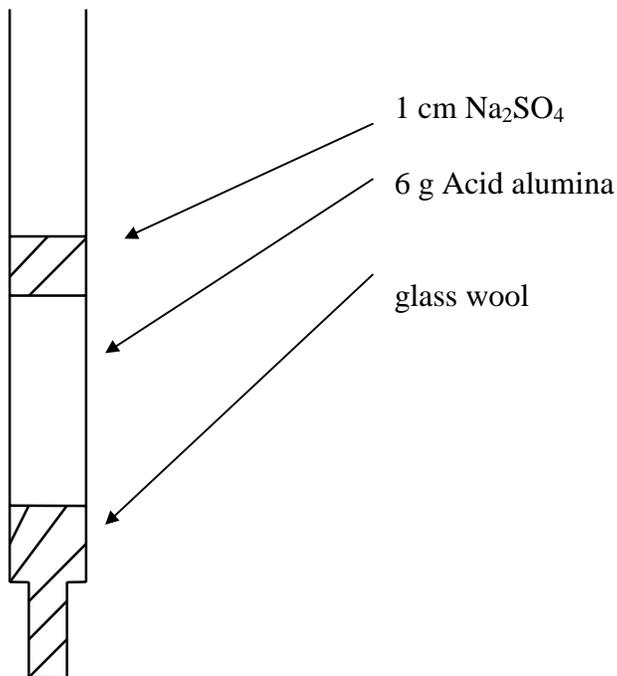
- 11.6.2.1. Pre-rinse both columns with hexane - 20 mL Top and 20 mL Bottom.
- 11.6.2.2. Put one column above the other.
- 11.6.2.3. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.
- 11.6.2.4. Elute 60 mL hexane directly onto acid silica column (upper piggy backed columns).
- 11.6.2.5. Discard upper column.
- 11.6.2.6. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.6.2.7. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.6.2.8. Proceed with additional cleanups as necessary.

### 11.6.3. Acid Alumina Column Clean-Up “Exhibit B”

#### Exhibit B Acid Alumina Column Clean-up



11.6.3.1. Alumina activity may vary with the matrix or environmental conditions. Monitor internal standard and cleanup recovery standard recoveries in extract analysis. Low recoveries of cleanup recovery standard (CRS) may indicate loss of alumina activity. Assess stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile). Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified (see section 11.6.3.2 for profile technique).

- 11.6.3.2. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:
  - 11.6.3.2.1. Set up and label 3 acid alumina columns.
  - 11.6.3.2.2. Pre-rinse with 20 mL hexane.
  - 11.6.3.2.3. Add 2 mL hexane spiked with internal standards and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.
  - 11.6.3.2.4. Elute each column with 20 mL hexane. Collect and label these fractions.
  - 11.6.3.2.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.
  - 11.6.3.2.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.
- 11.6.3.3. Review data and select an elution scheme. Group the fraction from each solvent system as follows:
  - 11.6.3.3.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.
  - 11.6.3.3.2. Analyte fraction - consists of all that contain detectable levels of target analytes.
  - 11.6.3.3.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.
- 11.6.3.4. Select the solvent system which best meets the following two conditions:
  - 11.6.3.4.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.
  - 11.6.3.4.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

11.6.3.5. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

11.6.3.6. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

11.6.4. Carbon Column Clean-up “Exhibit C”

11.6.4.1. Prepare an activated Carbon & Silica Gel column as described in EXHIBIT C, below.

Exhibit C  
Carbon Column Clean-up  
Special D2

“A”

0
1
2 mL
3 mL
4 mL
5 mL
6 mL
7 mL
8 mL
9 mL
10 mL

- Cut off both ends of a 10 mL pipette, or use pre-cut column.
- Push a glasswool plug down to the mark.
- Add 1g of 5% activated carbon/silica and top with another glasswool plug.
- Pre-elute with 5 mL 1:1 MeCl<sub>2</sub>:cyclohexane. Direction “A”
- Turn over and pre-elute with 5 mL 1:1 MeCl<sub>2</sub>:cyclohexane in direction “B”.
- Discard pre-eluates.
- Dilute extract to 1 mL with hexane and transfer to the column in the “B” direction .
- Rinse sample vial onto the column with 2 X 2 mL 1:1 MeCl<sub>2</sub>:cyclohexane.
- Elute with 6mL 1:1 MeCl<sub>2</sub>:cyclohexane  
5mL 75:20:5 MeCl<sub>2</sub>:MeOH:Benzene
- Discard Evaluates
- After flipping column back to the “A” direction, the column is eluted with 30 mL of toluene and collected

“B”

- N<sub>2</sub> or roto-vap to NEAR dryness and proceed to 11.8.

## 11.7. Sample Dilution Procedure

- 11.7.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 50X dilution of original 10 g/20  $\mu$ L sample  
(10 g/20  $\mu$ L) x (2  $\mu$ L aliquot + 98  $\mu$ L keeper) = 1 g/100  $\mu$ L FV

Record the final sample concentration on the extract label.

- 11.7.2. Complex dilution requiring respiking of IS and RS: Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20  $\mu$ L final volume)

Take a 2  $\mu$ L aliquot (1/10 of original sample) and add 18  $\mu$ L of solvent keeper. Take a 2  $\mu$ L aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20  $\mu$ L RS, reduced to 20  $\mu$ L FV.

Record the final sample concentration of the extract label.

## 11.8. Analytical Procedures

- 11.8.1. With a stream of dry, purified nitrogen, reduce the extract volume to approximately 100  $\mu$ L. Add 20  $\mu$ L of the recovery standard solution (Table 2). With a stream of dry, purified nitrogen, reduce the extract to volume to 20  $\mu$ L.

11.8.1.1. Transfer the extract to an autoinjection vial and store in the dark at room temperature.

11.8.1.2. A smaller final volume can be used to decrease the detection limit upon client approval.

11.8.1.3. A larger final volume can be use to decrease potential matrix interferences of which cleanups were unsuccessful.

- 11.8.2. Inject a 1 or 2  $\mu\text{L}$  aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.
- 11.8.3. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.
- 11.8.4. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

11.8.4.1. Retention Times

- 11.8.4.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled internal or recovery standard at  $m/z$  corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.
- 11.8.4.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract, the relative retention time (relative to the appropriate internal standard) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to  $^{13}\text{C}$ -OCDD as determined from the daily routine calibration results.
- 11.8.4.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

11.8.4.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously ( $\pm 2$  seconds).

11.8.4.1.5. The ion current responses for both ions used for the labeled standards (e.g., for  $^{13}\text{C}$ -TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously ( $\pm 2$  seconds).

11.8.4.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

11.8.4.3. Signal-To-Noise Ratio

All ion current intensities must be  $>2.5$  times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 6 describes the procedure to be followed for the determination of the S/N.

11.8.4.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N  $>2.5$  is detected, at the same retention time ( $\pm 2$  seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

## 12. DATA ANALYSIS AND CALCULATIONS

12.1. For gas chromatographic peaks that have met the criteria outlined in Section 11.8.4 calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times RRF(n)}$$

Where:

$C_x$  = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

$A_x$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

$A_{is}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

$Q_{is}$  = quantity, in pg, of the internal standard added to the sample before extraction,

$W$  = sample size in g (if solid) or L (if liquid).

$RRF(n)$  = Calculated mean relative response factor for the analyte

[ $RRF(n)$  with  $n = 1$  to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs,  $RRF(n)$  is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the  $RRF(k)$  value is the one calculated using the equation in Section 10.3.5.3.2 [ $RRF(k)$  with  $k = 27$  to 30].

- 12.2. Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Percent Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times RRF(m)} \times 100$$

Where:

$A_{is}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

$A_{rs}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

$Q_{is}$  = Quantity, in pg, of the internal standard added to the sample before extraction,

$Q_{rs}$  = Quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$RRF(m)$  = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Section 10.3.5.4 [ $RRF(m)$  with  $m = 18$  to 26].

- 12.3. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

- 12.3.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately.
- 12.3.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately.
- 12.4. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.
  - 12.4.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.
  - 12.4.2. Extraction of an aliquot large enough to be representative with an increased concentration of internal standard and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.
  - 12.4.3. Dilution of the original extract. Internal standard components are re-spiked at an appropriate level prior to analysis. In this case, the internal standard recoveries are taken from the original analysis.
- 12.5. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.
- 12.6. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.
- 12.7. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.7.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

12.7.1.1. Use the expression for EDL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a <sup>13</sup>C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their internal standard must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific2,3,7,8-subst.PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times RRF(n)}$$

$$EDL(\text{specific } 2,3,7,8\text{-subst. PCDD/PCDF}) = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

$H_x$  = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

$H_{is}$  = height of one of the quantitation ions (Table 6) for the labeled internal standards.

W, RRF (n), and Q<sub>is</sub> retain the same meanings as defined in Section 12.1

12.7.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

12.7.2.1. When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 11.8.4, calculate the “Estimated Maximum Possible Concentration” (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDL can be calculated using the above formula and the height of one of the ions as appropriate.

12.8. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2) / 2} \times 100$$

S<sub>1</sub> and S<sub>2</sub> represent sample and duplicate sample results.

12.9. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 11). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 11.

12.9.1. Two-GC Column TEF Determination

12.9.1.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be  $\leq 25\%$  valley.

- 12.9.1.2. For samples that have a presumptive positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be  $\leq 25\%$  valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.
- 12.9.1.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.
- 12.9.1.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 12 and the results from the routine calibration run on the DB-5 column.

### 13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 3 provides a typical 12-hour analysis sequence. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

#### 13.1. GC Column Performance

- 13.1.1. Inject 1 or 2  $\mu\text{L}$  of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of  $< 1$  second.
- 13.1.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of  $\leq 25$  percent (Figure 4),

Where:

$$\text{Valley Percent} = \left( \frac{x}{y} \right) \times 100$$

x = measured as in Figure 4 from the 2,3,7,8-closest TCDD eluting isomer,  
y = the peak height of 2,3,7,8-TCDD

- 13.1.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The

chromatograms showing the first and last eluters of a homologous series must be included.

- 13.1.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

## 14. POLLUTION PREVENTION

- 14.1. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.
- 14.2. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.
- 14.3. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.
- 14.4. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.
- 14.5. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

## 15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

- 15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.
- 15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials contaminated with methylene chloride. Silica gel, alumina, carbon and sodium sulfate, from column clean-ups, contaminated with various solvents and eluates. Dump the materials into a contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.3. Flammable solvent waste generated during glassware and sodium sulfate cleaning. Flammable solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Keep waste flammable solvents separated from waste methylene chloride. Collect the waste flammable solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.4. Waste methylene chloride generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing. Waste methylene chloride collected during roto-rap/turbo-vap reduction of extracted samples. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel

methylene chloride drum in the H3 closet. When the drum is full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

- 15.5. Contaminated mercury used during extract cleanup. Pour the contaminated mercury into a 250-ml plastic bottle labeled for contaminated mercury. When full or after one year, whichever comes first, transfer this jar to the waste collection area for shipment.
- 15.6. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.
- 15.7. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

## 16. REFERENCES

- 16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.
- 16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.
- 16.3. Compendium Method TO-9A "Determination of Polychlorinated, Polybrominated, and Brominated, Chlorinated Dibenzo-p-dioxins and Dibenzofurans in Ambient Air", EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.
- 16.4. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.
- 16.5. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)
- 16.6. "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.
- 16.7. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

## 17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

- 17.1. Deviations from reference method.
  - 17.1.1. The method specifies that 2  $\mu$ L injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1  $\mu$ L injections for all performance checks, standards, QC samples, and samples.
  - 17.1.2. In Section 2.7 of Method 8290, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled internal standards. All available labeled internal standards are used; therefore, a retention time window of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290.

- 17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.
- 17.1.4. Extract clean-ups are performed at the discretion of the analyst when interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.
- 17.1.5. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.
- 17.1.6. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any other mechanical means to break up an emulsion.
- 17.1.7. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.
- 17.1.8. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2).
- 17.1.9. Modifications from TO-9A method
  - 17.1.9.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.
  - 17.1.9.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.
  - 17.1.9.3. The 37CL-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/  $\mu$ L).
  - 17.1.9.4. Samples are extracted with toluene not benzene.
  - 17.1.9.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

- 17.1.9.6. All cleanup procedures are optional and applied based on the analyst's discretion.
- 17.1.9.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.
- 17.1.9.8. The final volume is adjusted to 20  $\mu$ L in tetradecane.
- 17.1.9.9. Calibration and quantitation are performed in accordance to this SOP.

17.2. Summary of modifications to SOP from previous revisions

- 17.2.1. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

17.3. Tables or figures referenced in body of SOP.

- 17.3.1. Table 1 - Types of Matrices
- 17.3.2. Table 2 - Composition of The Sample Fortification and Recovery Standard Solutions.
- 17.3.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners
- 17.3.4. Table 4 - Isomers of Chlorinated Dioxins and Furans
- 17.3.5. Table 5 - Concentrations of Calibration Solutions
- 17.3.6. Table 6 - Ions Monitored for PCDDs/PCDFs
- 17.3.7. Table 7 - Recommended GC Operating Conditions
- 17.3.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)
- 17.3.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits
- 17.3.10. Table 10 - Relative Response Factor Attributes
- 17.3.11. Table 11 - 2,3,7,8-TCDD Equivalent Factors
- 17.3.12. Table 12 - TEF: Analyte Relative Retention Time Reference Attributes

17.3.13. Figure 1 - Analysis Flowchart

17.3.14. Figure 2 - Compound Structure

17.3.15. Figure 3 - Analysis Scheme

17.3.16. Figure 4 - GC Performance Check Chromatogram on the DB-5 Column

17.3.17. Figure 5 - PFK Peak Profile

17.3.18. Figure 6 - Manual Determination of Signal-to-Noise

17.3.19. Appendix A - Periodic Wipe Test Performance

**TABLE 1**

**Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based  
Method Calibration Limits (Parts per Trillion)**

	<b>Water</b>	<b>Soil Sediment Paper Pulp</b>	<b>Fly Ash</b>	<b>Human/ Fish Tissue</b>	<b>Adipose Tissue</b>	<b>Sludges, Fuel Oil</b>	<b>Still- Bottom</b>	<b>Ambient or Source Samples</b>
Lower MCL(a)	0.02	2.0	2.0	2.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (µL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 2.5 for PeCDD/PeCDF/HxCDD/HxCDF/ HpCDD/HpCDF, and by 5 for OCDD/OCDF.

**TABLE 2**  
**Composition of the Sample Fortification  
and Recovery Standard Solutions**

Analyte	Sample Fortification Solution Concentration pg/ $\mu$ L; Solvent: Isooctane	Recovery Standard Solution Concentration pg/ $\mu$ L; Solvent: Tetradecane
13C-2,3,7,8-TCDD	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-2,3,7,8-TCDF	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,4-TCDD	--	100
13C-1,2,3,7,8-PeCDD	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,7,8-PeCDF	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,6,7,8-HxCDD	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,4,7,8-HxCDF <sup>(d)</sup>	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,7,8,9-HxCDD	--	100
37Cl-2,3,7,8-TCDD <sup>(b)(c)</sup>	0.8 <sup>(b)</sup> , 100 <sup>(c)</sup>	
	100 <sup>(c)</sup>	
13C-2,3,4,7,8-PeCDF <sup>(c)</sup>	100 <sup>(c)</sup>	
13C-1,2,3,6,7,8-HxCDF <sup>(c)(d)</sup>	100 <sup>(c)</sup>	
13C-1,2,3,4,7,8-HxCDD <sup>(c)</sup>	100 <sup>(c)</sup>	
13C-1,2,3,4,7,8,9-HpCDD <sup>(c)</sup>	100 <sup>(c)</sup>	
13C-1,2,3,4,6,7,8-HpCDD	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-1,2,3,4,6,7,8-HpCDF	2 <sup>(a)</sup> , 100 <sup>(c)</sup>	--
13C-OCDD	4 <sup>(a)</sup> , 200 <sup>(c)</sup>	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations

(b) Method TO9 and TO9A surrogate concentrations

(c) Method 23 and Method 0023A surrogate concentrations

(d) 13C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and 13C-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

**TABLE 3**

**The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners**

<b>PCDD</b>	<b>PCDF</b>
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(\*)The <sup>13</sup>C-labeled analog is used as an internal standard.

(+)The <sup>13</sup>C-labeled analog is used as a recovery standard.

**TABLE 4**

**Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms**

<b># of Chlorine Atoms</b>	<b># of Dioxin Isomers</b>	<b># of 2,3,7,8 Isomers</b>	<b># of Furan Isomers</b>	<b># of 2,3,7,8 Isomers</b>
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
<b>Total</b>	<b>75</b>	<b>7</b>	<b>135</b>	<b>10</b>

**TABLE 5**

**High Resolution Concentration Calibration Solutions**

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3 (VER(6))	CS4	CS5
Native CDDs and CDFs					
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8-TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
Labeled CDDs and CDFs					
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -2,3,4,6,7,8-HxCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	100	100	100	100	100

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3 (VER(6))	CS4	CS5
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -OCDD	200	200	200	200	200
Cleanup Standard/ FS					
<sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	0.5	2	10	40	200
Recovery Standards					
<sup>13</sup> C <sub>12</sub> -1,2,3,4-TCDD	100	100	100	100	100
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	100	100	100	100	100

**TABLE 6\***  
Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate <sup>(a)</sup> Mass	Ion ID	Elemental Composition	Analyte
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK
2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDF
	341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF
	351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDF (S)
	353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)
	355.8546	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD
	357.8516	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD
	367.8949	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)
	369.8919	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)
	409.7974	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDFE
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK
3	373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF
	375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF
	383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> O	HxCDF (S)
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)

**TABLE 6 (cont.)\***

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate <sup>(a)</sup> Mass	Ion ID	Elemental Composition	Analyte
4	407.7818	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF
	409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF
	417.8250	M	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> O	HpCDF (S)
	419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF
	423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD
	425.7737	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD
	435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)
	437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)
	479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDPE
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK
5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
	443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
	457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
	459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
	469.7780	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)
	471.7750	M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)
	513.6775	M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE
	[442.9728]	LOCK	C <sub>10</sub> F <sub>17</sub>	PFK

<sup>(a)</sup> The following nuclidic masses were used:

H = 1.007825	O = 15.994915
C = 12.000000	<sup>35</sup> Cl = 34.968853
<sup>13</sup> C = 13.003355	<sup>37</sup> Cl = 36.965903
F = 18.9984	

S = Internal/recovery standard

\*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

**TABLE 7**

**Recommended GC Operating Conditions**

The GC Operating Conditions (Temperatures (°C), and Times (minutes))  
Are as Follows:

Injector Temperature: 280°C

Interface Temperature: 280°C

Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

**TABLE 8**

**PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column<sup>(b)</sup>**

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4 <sup>(a)</sup>	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

<sup>(a)</sup> In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, <sup>13</sup>C<sub>12</sub>-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- <sup>13</sup>C<sub>12</sub>-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are ≤ 25%.

**TABLE 9**

**Theoretical Ion Abundance Ratios and Their  
Control Limits for PCDDs and PCDFs**

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6 <sup>(a)</sup>	M / M+2	0.51	0.43	0.59
7 <sup>(b)</sup>	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

<sup>(a)</sup> Used only for <sup>13</sup>C-HxCDF (IS)

<sup>(b)</sup> Used only for <sup>13</sup>C-HpCDF (IS)

**TABLE 10**

**Relative Response Factor [RRF (number)] Attributes**

<u>Number</u>	<u>Specific Congener Name</u>
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCdf
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD
19	<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF
20	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD
21	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF
22	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD
23	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
24	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD
25	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF
26	<sup>13</sup> C <sub>12</sub> . OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

**TABLE 11**

**2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated  
Dibenzodioxins and Dibenzofurans**

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCdd	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

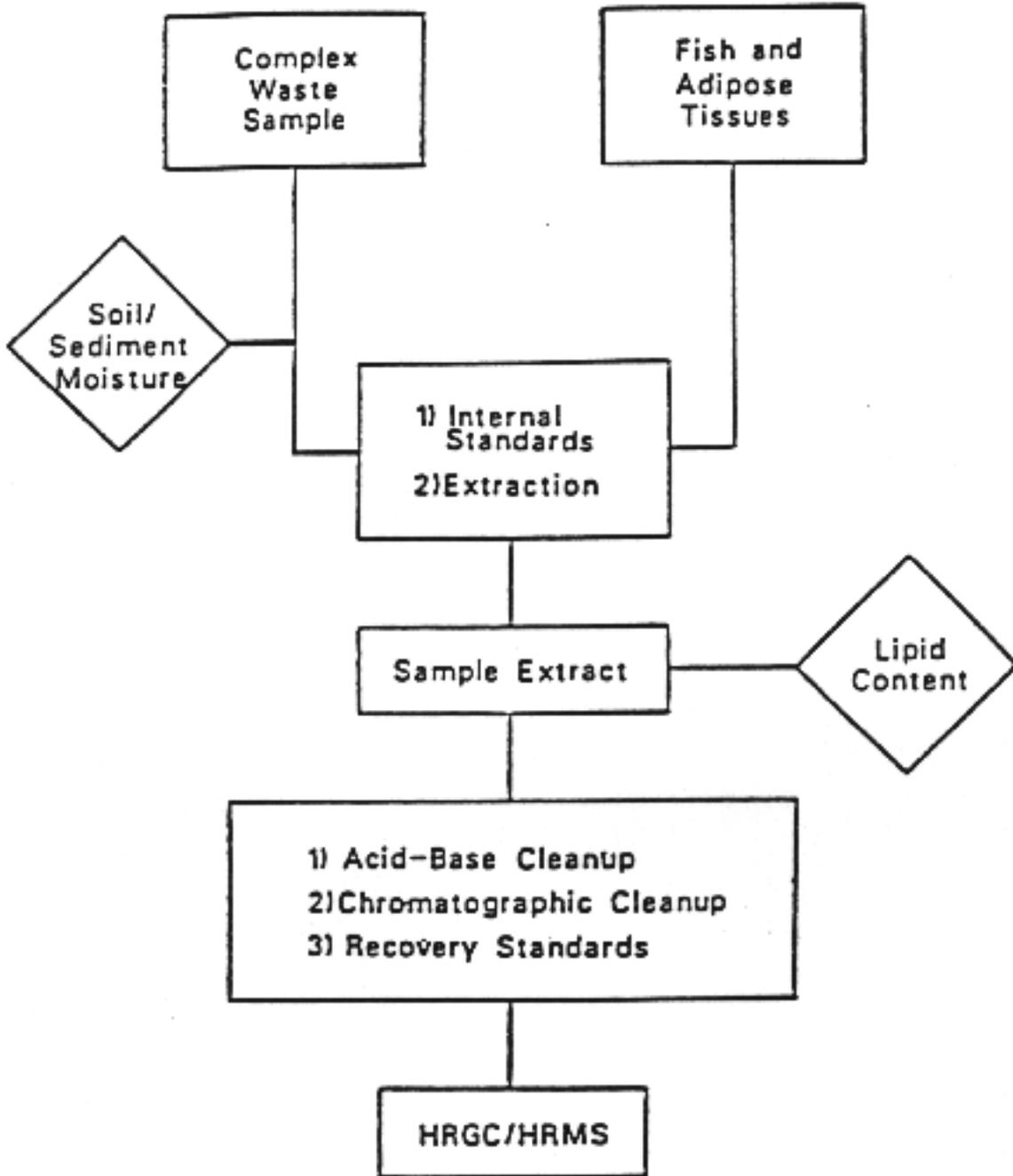
**TABLE 12**

**Toxicity Equivalency Factor:  
Analyte Relative Retention Time Reference Attributes**

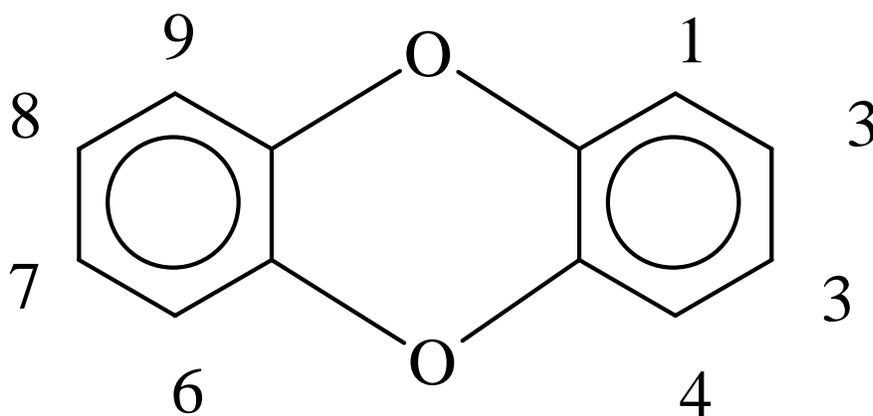
Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to <sup>13</sup>C<sub>12</sub>-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to <sup>13</sup>C<sub>12</sub>-1,2,3,4,6,7,8-HpCDF

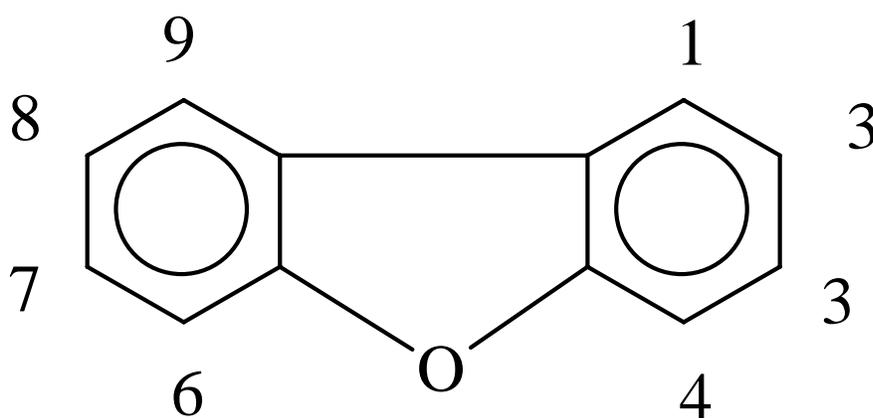
FIGURE 1



**FIGURE 2**  
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

**FIGURE 3**  
**Analytical Procedure**

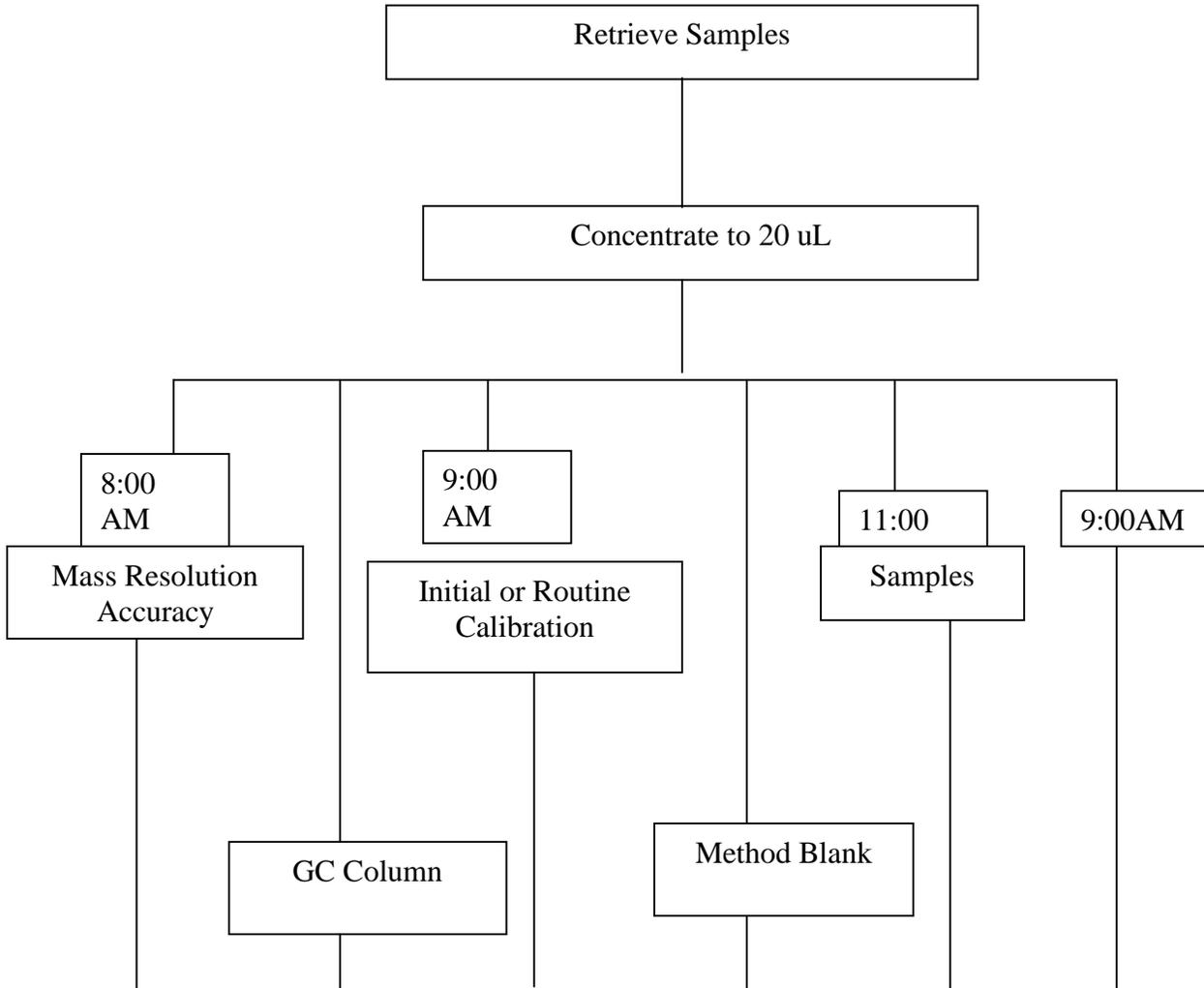


FIGURE 4

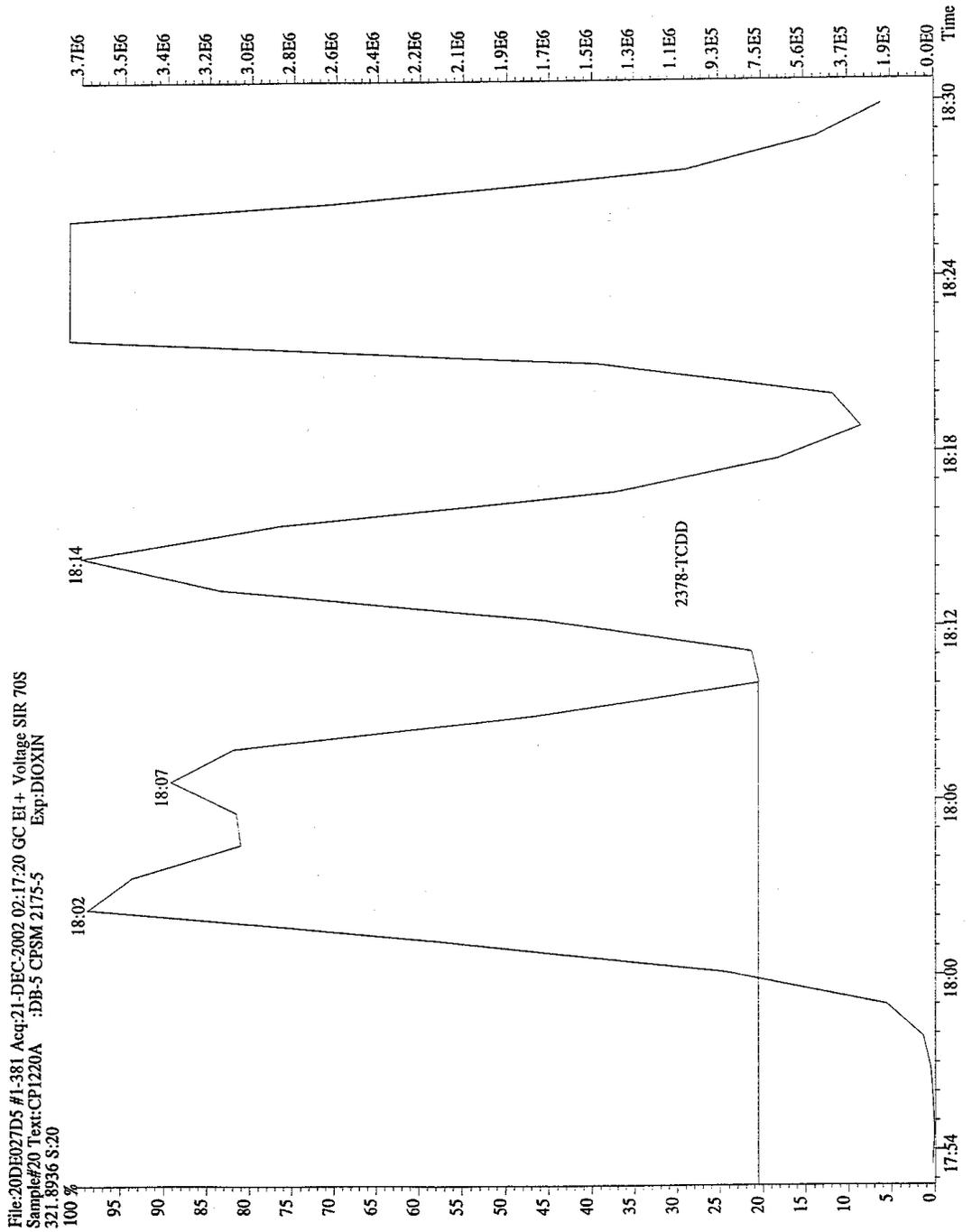


Figure 5

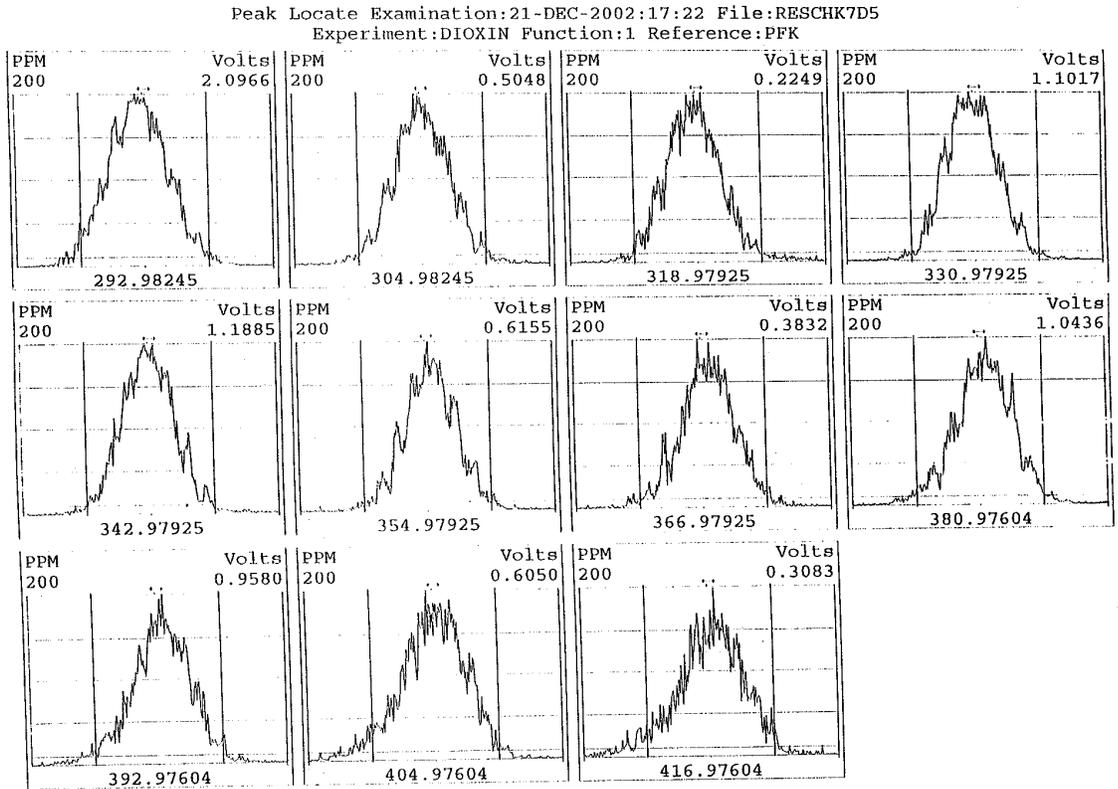
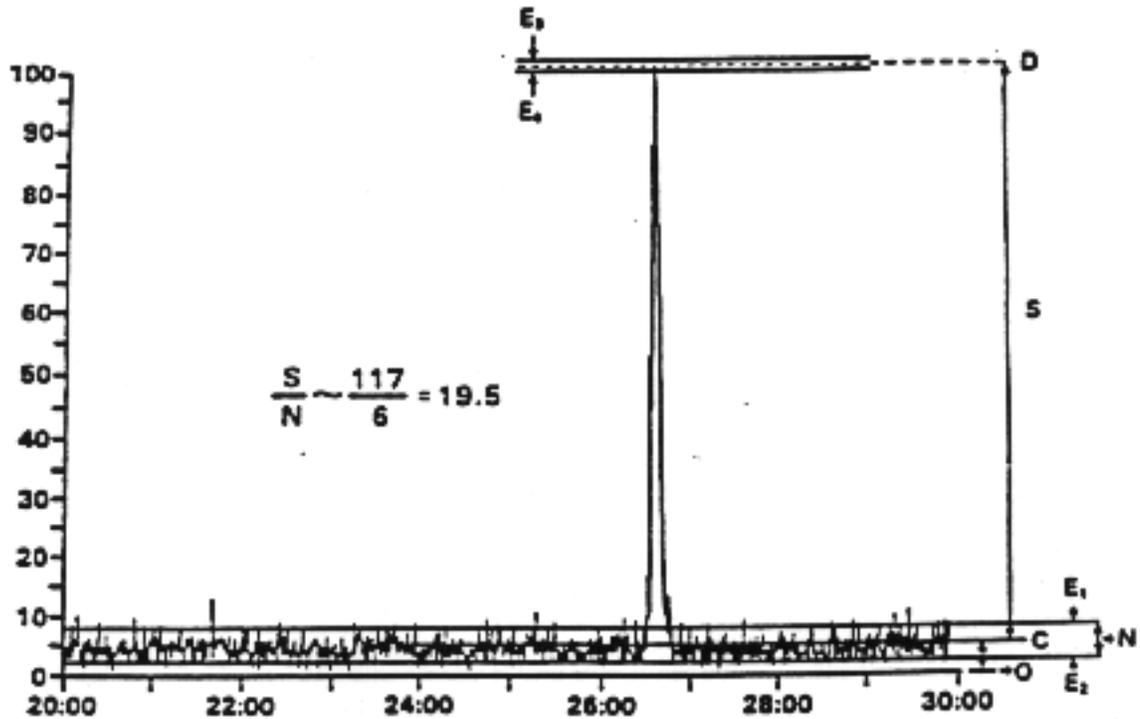


FIGURE 6



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

**NOTE:** It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

## APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

### PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

### SAMPLE PREPARATION

Close the jar containing the wipers and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

### EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20  $\mu$ L (either in a minivial or in a capillary tube). Inject 2  $\mu$ L of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

### REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is  $25 \times 5 = 125$  pg/WTE and the positive response for the blank would be  $8 \times 5 = 40$  pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

### FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

### CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 1
<b>SOP Title:</b>	METHOD 8290 AND TO-9A - POLYCHLORINATED DIOXINS & FURANS BY HRGC/HRMS	
<b>SOP Sections Affected by Change:</b>	Sections 10.6; 11.6	
<b>Reason for Addition or Change:</b>	<p>Section 10.6: Amend the ending calibration check criteria to be method compliant.</p> <p>Section 11.6: Add the practice of adding CRS (Cleanup Recovery Standard) to samples prior to sample cleanups.</p>	
<b>Change Effective From [Date]:</b>	11/19/03	
<b>Change or Addition (Specific Section; use additional sheets if necessary.):</b>	<p>Section 10.6.1, Change to read:</p> <p>The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be <math>\pm 20</math> percent of the mean values established during the initial calibration (Section 10.3.5.)</p> <p>Section 10.6.1.1, Change to read:</p> <p>The bracketing continuing calibration must be less than or equal to <math>\pm 20\%</math> of the average RRF calculated from the initial calibration.</p> <p>Section 10.6.1.1.1, Change to read:</p> <p>If the target compounds in the ending standard are less than or equal to <math>\pm 20\%</math> of the average RRF from the initial calibration, the average RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.</p> <p>Add Section 10.6.1.1.2:</p> <p>If the target analytes are greater than <math>\pm 20\%</math> but less or equal to <math>\pm 25\%</math> and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than <math>\pm 20\%</math> but less or equal to <math>\pm 25\%</math> and are positive, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.</p> <p>Add Section 10.6.1.1.3:</p> <p>If the percent deviation of unlabeled compounds exceeds <math>\pm 25\%</math>, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.</p> <p>Section 10.6.2, Change to read:</p> <p>The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to <math>\pm 30</math> percent of the mean values established during the initial calibration (Section 10.1.5).</p> <p>Section 10.6.2.1, Change to read:</p> <p>The bracketing continuing calibration must be <math>\pm 30\%</math> of the average RRF calculated from the initial calibration.</p> <p>Section 10.6.2.1.1, Change to read:</p> <p>If the labelled compounds in the ending standard are less than or equal to <math>\pm 30\%</math> of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.</p>	

<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 1
	<p>Add Section 10.6.2.1.2: If the internal standard analytes are greater than <math>\pm 30\%</math> but less than or equal to <math>\pm 35\%</math>, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.</p> <p>Add Section 10.6.2.1.3: If the percent deviation of labeled compounds exceeds <math>\pm 35\%</math>, reanalyze samples if adversely impacted.</p> <p>Section 11.6, append paragraph: For all samples which are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC extracts. (See also section 9.7.1)</p>	

Submitted by: Lisa Stafford

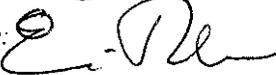
Date: 11/19/2003

Approved by: \*

Technical Reviewer:  Date: 11-21-03

EH&S Signature: NA Date:

QA Signature:  Date: 11/21/03

Management Signature:  Date 11/24/03

\*Must be same signature authorities of SOP being revised.

<b>SOP Number:</b>	SAC-ID-0005    Rev. 6.0	Change Form Number: 2
<b>SOP Title:</b>	METHOD 8290 AND TO-9A - POLYCHLORINATED DIOXINS AND FURANS HRGC/HRMS	
<b>SOP Sections Affected by Change:</b>	Section 7.2.7 (added) Section 7.2.8 (added) Section 9.1.2 Section 11.3.5	
<b>Reason for Addition or Change:</b>	Adding a matrix for QC samples during tissue analysis. Changing the laboratory matrix for soil samples from sodium sulfate to precleaned Ottawa sand.	
<b>Change Effective From [Date]:</b>	2/24/04	
<b>Change or Addition (Specific Section; use additional sheets if necessary.):</b>	<p><b>All changes within the section have been underlined.</b></p> <p>Section 7.2.7 should read as follows: <u>Precleaned Ottawa sand (minimum 4 hours toluene Soxhlet Extracted).</u></p> <p>Section 7.2.8 should read as follows: <u>Canola Oil (for tissue extraction only).</u></p> <p>Section 9.1.2 should read as follows: Use <u>Ottawa sand</u> as the method laboratory matrix when solids are extracted. <u>Use a mixture of Ottawa sand and canola oil as the matrix when tissues are extracted.</u> Take the method blank through all steps detailed in the analytical procedure.</p> <p>Section 11.3.5 should be followed by a Note that read as follows: Note: <u>The matrix for the tissue method blank consists of 9g of pre-cleaned Ottawa sand and 1g of canola oil.</u></p>	

Submitted by: Eugene Mathis

Date: 2/24/04

Approved by: \*

Technical Reviewer: 

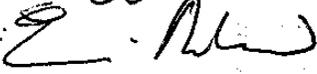
Date: 3/19/04

EH&S Signature: 

Date: 3/19/04

QA Signature: 

Date: 3/4/04

Management Signature: 

Date: 3/28/04

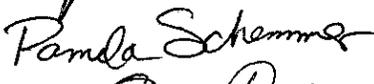
\*Must be same signature authorities of SOP being revised.

<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 3
<b>SOP Title:</b>	METHOD 8290 AND TO-9A - POLYCHLORINATED DIOXINS & FURANS BY HRGC/HRMS	
<b>SOP Sections Affected by Change:</b>	9.1	
<b>Reason for Addition or Change:</b>	Add clarification for compliance with DOD QSM.	
<b>Change Effective From [Date]:</b>	9/20/04	
<b>Change or Addition (Specific Section; use additional sheets if necessary.):</b>	Section 9.1, add paragraph to read: Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.	

Submitted by: Lisa Stafford

Date: 9/20/04

**Approved by: \***

Technical Reviewer:  Date: 9-22-04  
 EH&S Signature:  Date: 9/22/04  
 QA Signature:  Date: 9/22/04  
 Management Signature:  Date: 9/24/04

**\*Must be same signature authorities of SOP being revised.**



<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 4
<b>SOP Title:</b>	METHOD 8290 AND TO-9A - POLYCHLORINATED DIOXINS & FURANS BY HRGC/HRMS	
<b>SOP Sections Affected by Change:</b>	5, 14, and 15	
<b>Reason for Addition or Change:</b>	Replaced and updated to comply with current EH&S requirements.	
<b>Change Effective From [Date]:</b>	8/29/05	
<b>Change or Addition (Specific Section; use additional sheets if necessary.):</b>	<p>Section 5, SAFETY, change to read:</p> <p>Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&amp;S Staff, or a senior manager.</p> <p>5.1      SPECIFIC SAFETY CONCERNS OR REQUIREMENTS</p> <p>5.1.1    Hearing protection must be worn when using mechanical systems to grind fish or tissue samples.</p> <p>5.1.2    When dissecting crawfish abdomens with a scalpel, cut from the hand holding the abdomen toward the tail (away from you).</p> <p>5.1.3    Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.</p> <p>5.1.4    The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.</p> <p>5.1.5    Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.</p> <p>5.1.6    Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.</p> <p>5.1.7    Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.</p> <p>5.1.8    Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&amp;S staff. The task will be analyzed to determine a better means of accomplishing it.</p> <p>5.2      PRIMARY MATERIALS USED</p> <p>The following is a list of the materials used in this method, which have a serious or significant</p>	

<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 4																																				
	<p>hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.</p> <p>(Note: The following material to be presented in table form:)</p> <table border="0"> <thead> <tr> <th data-bbox="480 554 888 583">Material Hazards</th> <th data-bbox="964 554 1326 583">Exposure Limit (2)</th> <th data-bbox="964 554 1326 583">Signs and symptoms of exposure</th> </tr> </thead> <tbody> <tr> <td data-bbox="480 600 1526 659">Acetone; Flammable;</td> <td data-bbox="480 600 1526 659">1000 ppm-TWA;</td> <td data-bbox="480 600 1526 659">Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.</td> </tr> <tr> <td data-bbox="480 676 1571 884">Benzene; Flammable, Toxic, Carcinogen;</td> <td data-bbox="480 676 1571 884">PEL: 1 PPM, TWA ; 5 PPM, 15 MIN. STEL;</td> <td data-bbox="480 676 1571 884">Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.</td> </tr> <tr> <td data-bbox="480 905 1562 993">Cyclohexane; Flammable, Irritant;</td> <td data-bbox="480 905 1562 993">300 ppm TWA;</td> <td data-bbox="480 905 1562 993">Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.</td> </tr> <tr> <td data-bbox="480 1014 1562 1102">Hexane; Flammable, Irritant;</td> <td data-bbox="480 1014 1562 1102">500 ppm-TWA;</td> <td data-bbox="480 1014 1562 1102">Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.</td> </tr> <tr> <td data-bbox="480 1123 1533 1211">Iso-octane; Flammable, Irritant;</td> <td data-bbox="480 1123 1533 1211">None established;</td> <td data-bbox="480 1123 1533 1211">Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.</td> </tr> <tr> <td data-bbox="480 1232 1559 1375">Methanol; Flammable, Poison, Irritant;</td> <td data-bbox="480 1232 1559 1375">200 ppm-TWA;</td> <td data-bbox="480 1232 1559 1375">A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.</td> </tr> <tr> <td data-bbox="480 1396 1567 1518">Methylene Chloride; Carcinogen, Irritant;</td> <td data-bbox="480 1396 1567 1518">25 ppm-TWA, 125 ppm-STEEL;</td> <td data-bbox="480 1396 1567 1518">Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.</td> </tr> <tr> <td data-bbox="480 1539 1576 1682">Potassium Hydroxide; Corrosive, Poison;</td> <td data-bbox="480 1539 1576 1682">2 mg/m<sup>3</sup> ceiling;</td> <td data-bbox="480 1539 1576 1682">Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on the severity of exposure. Symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.</td> </tr> <tr> <td data-bbox="480 1703 1559 1791">Sodium Hydroxide; Corrosive, Poison;</td> <td data-bbox="480 1703 1559 1791">2 ppm, 5 mg/m<sup>3</sup>;</td> <td data-bbox="480 1703 1559 1791">This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.</td> </tr> <tr> <td data-bbox="480 1812 1567 1900">Sulfuric Acid (1); Corrosive, Oxidizer, Dehydrator;</td> <td data-bbox="480 1812 1567 1900">1 mg/m<sup>3</sup>;</td> <td data-bbox="480 1812 1567 1900">This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.</td> </tr> <tr> <td data-bbox="480 1921 1508 1971">Tetradecane; Irritant;</td> <td data-bbox="480 1921 1508 1971">None established;</td> <td data-bbox="480 1921 1508 1971">Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.</td> </tr> </tbody> </table>		Material Hazards	Exposure Limit (2)	Signs and symptoms of exposure	Acetone; Flammable;	1000 ppm-TWA;	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.	Benzene; Flammable, Toxic, Carcinogen;	PEL: 1 PPM, TWA ; 5 PPM, 15 MIN. STEL;	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.	Cyclohexane; Flammable, Irritant;	300 ppm TWA;	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.	Hexane; Flammable, Irritant;	500 ppm-TWA;	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.	Iso-octane; Flammable, Irritant;	None established;	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.	Methanol; Flammable, Poison, Irritant;	200 ppm-TWA;	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.	Methylene Chloride; Carcinogen, Irritant;	25 ppm-TWA, 125 ppm-STEEL;	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.	Potassium Hydroxide; Corrosive, Poison;	2 mg/m <sup>3</sup> ceiling;	Severe irritant. 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Tetradecane; Irritant;	None established;	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Material Hazards	Exposure Limit (2)	Signs and symptoms of exposure																																				
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<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 4
	<p>Toluene; Flammable, Poison, Irritant; 200 ppm-TWA, 300 ppm-Ceiling; Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.</p> <p>1 – Always add acid to water to prevent violent reactions.          2 – Exposure limit refers to the OSHA regulatory exposure limit.</p> <p>Section 14, POLLUTION PREVENTION, change to read:</p> <p>14.1 Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.</p> <p>14.2 The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.</p> <p>14.3 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.</p> <p>14.4 Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.</p> <p>14.5 Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.</p> <p>Section 15, WASTE MANAGEMENT, change to read:</p> <p>The following waste streams are produced when this method is carried out.</p> <p>15.1 Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.</p> <p>15.2 Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials contaminated with methylene chloride. Silica gel, alumina, carbon and sodium sulfate, from column clean-ups, contaminated with various solvents and eluates. Dump the materials into a contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.</p> <p>15.3 Flammable solvent waste generated during glassware and sodium sulfate cleaning. Flammable solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Keep waste flammable solvents separated from waste methylene chloride. Collect the waste flammable solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.</p> <p>15.4 Waste methylene chloride generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing.. Waste methylene chloride collected during roto-rap/turbo-vap</p>	

<b>SOP Number:</b>	SAC-ID-0005 Rev. 6	Change Form Number: 4
	<p>reduction of extracted samples. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride drum in the H3 closet. When the drum is full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.</p> <p>15.5 Contaminated mercury used during extract cleanup. Pour the contaminated mercury into a 250-ml plastic bottle labeled for contaminated mercury. When full or after one year, whichever comes first, transfer this jar to the waste collection area for shipment.</p> <p>15.6 Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.</p> <p>15.7 Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.</p>	

Submitted by: Lisa Stafford

Date: 8/29/2005

Approved by: \*

Technical Reviewer: 

Date: 8/30/05

EH&S Signature: 

Date: 8/30/05

QA Signature: 

Date: 8/30/05

Management Signature: 

Date: 8/31/05

\*Must be same signature authorities of SOP being revised.

revised reports must be annotated in accordance with Policy QA-005-SAC, "Results and Report Revision".

#### **8.8.6. Client Confidentiality**

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence, unless such information is generally available to the public or is in the public domain. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of our clients and are not released to a third party without written consent from the client. Data confidentiality is also discussed Section 3.6.

#### **8.9. Data Validation**

Data validation for STL refers to data reviews conducted in accordance with the USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is usually performed by the client or a third party, one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

#### **8.10. Preventive Maintenance and Service**

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within STL Sacramento, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

##### **8.10.1. Analytical Instrumentation and Equipment**

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. The laboratory maintains the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory

- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name and serial number of the item or equipment
- Details of maintenance performed
- Dates and results of recalibrations/reverifications indicating return to control
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or piece of equipment that does not perform to specifications or is defective shall be taken out of service, and tagged as out of service until it has been repaired and shown by calibration/verification to perform satisfactorily.

### ***8.10.2. Frequency of Equipment Maintenance***

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in Table 8.10-1 for analytical instrumentation and equipment or defined in operation specific routine maintenance SOPs. Frequency of maintenance for the facility systems is documented in the STL Corporate Safety Manual.

### ***8.10.3. Facilities***

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power. The facility floor plan is provided in Figure 8.10-1.

The maintenance and use of these facilities and proper operations are described in the STL Corporate Safety Manual. The Laboratory Director has responsibility for ensuring a properly maintained facility. The Laboratory Director also has the responsibility for ensuring that facilities are available to store samples properly without contamination, work areas are equipped with adequate bench, hood and operational space, and that procedures are in place to ensure the areas are free from chemical and radiological contamination that may affect analytical results.

**TABLE 8.10-1  
 Instrument Maintenance Schedule**

<b>APCI/ESI LC/MS/MS</b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.
<b>Daily<sup>(2)</sup></b>	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature. Verify vaporizer heater temperature. Verify rough pump and turbo-pump oil levels. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.
<b>Semi-Annually</b>	Replace rough-pump oil (4-6 months). Vacuum/clean inlet foam. Replace oil mist filter. Replace activated alumina filter.
<b>Annually</b>	Vacuum system components including fans and fan covers.

**TABLE 8.10-2  
 Instrument Maintenance Schedule**

<b>High Pressure Liquid Chromatograph<sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.
<b>Daily<sup>(2)</sup></b>	Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.
<b>Every 6-9 Months</b>	Change pump seals.

**TABLE 8.10-3**

<b>Gas Chromatograph<sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Replace septum. Clean injector port Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Perform gas purity check (if high baseline indicates that impure carrier gas may be in use). Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. PID: clean lamp window or replace as needed, replace seals. ECD: follow manufacturers suggested maintenance schedule Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.
<b>Daily<sup>(2)</sup></b>	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.
<b>Semi-Annually</b>	ECD: perform wipe test.

**TABLE 8.10-4  
 Instrument Maintenance Schedule**

<b>Purge and Trap Systems</b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Change trap. Check purge flow. Flush lines (after foaming sample). Periodic leak checks quarterly, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean glassware. Check purge flow. Bake trap as needed to correct for high background. Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed. Purge & trap autosamplers: leak check system, clean sample lines, valves.
<b>Daily<sup>(2)</sup></b>	Bake out trap prior to commencing analysis.

**TABLE 8.10-5  
 Instrument Maintenance Schedule**

<b>Gas Chromatography/Low-Resolution Mass Spectrometer<sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	<p>Replace septum.            Clean injector port.            Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.            Replace injection port liner when front portion of capillary column is removed.            Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed.            Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.            Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.            Repair/replace jet separator.            Replace filaments when both filaments burn out or performance indicates need for replacement.            Check mass calibration (PFTBA or FC-43).            Check ion source and analyzer (clean, replace parts as needed).            Check vacuum, relays, gas pressures and flows.            Change oil in the mechanical rough pump.            Relubricate the turbomolecular pump-bearing wick.            HP 7673 Autosampler: Replace syringe.</p>
<b>Daily<sup>(2)</sup></b>	<p>Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.            Check temperatures of injector, detector.            Verify temperature programs.            Check inlets, septa.            Check baseline level.            Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.            Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.            HP 7673 Autosampler: fill wash bottle, dispose of waste bottle contents.</p>
<b>Annually</b>	<p>Replace the exhaust filters on the mechanical rough pump every 1-2 years.</p>

**TABLE 8.10-6**

<b>Gas Chromatography/High-Resolution Mass Spectrometer<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	<p>Full Bake-Out.            Change oil in rotary pump.            Change oil in diffusion pump. Replace o-rings.            Solvent rinse the flight tube.            Clean the first field free region.            Check the data system ventilation (fans and filters).            Check detector voltages.            Clean and dust connectors, etc on the outside of the instrument.            Check the vacuum: <math>5 \times 10^{-8}</math> MBAR on both analyzer ion gauges, and <math>5 \times 10^{-7}</math> MBAR on the source, with no helium flowing.            Check isolation valve for leaks, correct if needed.            Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable.            Replace septum.            Clean injector port.            Break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.            Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed.            Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.            Repair/replace jet separator.            Replace filaments when both filaments burn out or performance indicates need for replacement.</p>
<b>Daily<sup>(2)</sup></b>	<p>Check resolution sensitivity.            Check stability.            Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.            Check temperatures of injector, detector.            Verify temperature programs.            Check inlets, septa.            Check baseline level.            Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.            Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.</p>

**TABLE 8.10-7  
 Instrument Maintenance Schedule**

<b>Cold Vapor Atomic Absorption (Leeman PS 200) <sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Change pump tubing. Check/change Hg lamp. Clean optical cell. Change drying tube. Grease pump.
<b>Daily<sup>(2)</sup></b>	Check sample tip for clogs. Check drying tube. Check pump tubing/drain tubing. Check gas pressure. Check liquid/gas separator. Check tubing.

**TABLE 8.10-8  
 Instrument Maintenance Schedule**

<b>Inductively Coupled Argon Plasma/Mass Spectrometry (ICAP/MS) <sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.
<b>Daily<sup>(2)</sup></b>	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.
<b>Every 2-3 Months</b>	Replace oil in roughing pumps.

**TABLE 8.10-9**

<b>ICP<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>Daily<sup>(2)</sup></b>	Check that argon feed pressure is 80-120 psi. Check that chiller coolant pressure is 45-80 psig, no leaks. Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig. Check radial purge and axial windows for deposits. Check that nebulizer is not clogged. Check that capillary tubing is clean and in good condition. Check that peristaltic pump windings are secure. Check that exhaust vent is operational Check that torch, glassware, aerosol injector tube are clean.
<b>Monthly or As Needed</b>	Clean plasma torch assembly to remove accumulated deposits. Check RF coil. Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance. Clean filters on back of power unit to remove dust. Replace when needed: peristaltic pump tubing. sample capillary tubing. autosampler sipper probe. Check performance with yttrium or sodium. Check O-rings. Clean/lubricate pump rollers.
<b>Semi-Annually</b>	Check chiller coolant filter. (may require more or less frequently)
<b>Annually</b>	Notify manufacturer service engineer for scheduled preventive maintenance service.

**TABLE 8.10-10**

<b>Ion Chromatograph<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Clean micromembrane suppressor when decreases in sensitivity are observed. Check fuses when power problems occur. Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. De-gas pump head when flow is erratic. Check all air and liquid lines for discoloration and crimping, if indicated. Check/change bed supports guard and analytical columns, if indicated.
<b>Daily<sup>(2)</sup></b>	Check plumbing/leaks. Check gases. Check pump pressure. Check conductivity meter.
<b>Weekly</b>	Check pump heads for leaks. Check filter (inlet).
<b>Annually</b>	Change pump seals. Change injection valve. Clean conductivity cell. Check conductivity cell for calibration.

**TABLE 8.10-11  
 Instrument Maintenance Schedule**

<b>Alpkem Colorimetric Auto Analyzer<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Prepare fresh reagents. Replace tubing. (About every 100 hours of use)
<b>Daily<sup>(2)</sup></b>	Check detector. Make sure there are no trapped bubbles in detector cell. Check Valves Check peristaltic tubing. Check sampler. Check auto diluter.
<b>Weekly</b>	Clean pump, diluter, and XYZ Sampler.
<b>Monthly</b>	Lubricate pump roller.
<b>Semi-Annually</b>	Clean pump rollers with steel wool and lubricate.

**TABLE 8.10-12  
 Instrument Maintenance Schedule**

<b>Chemical Oxygen Demand (COD) Reactor<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Electronics serviced.
<b>Annually</b>	Check temperature with NIST reference thermometer.

**TABLE 8.10-13**

<b>Auto Titrator<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Electronics serviced.
<b>Daily<sup>(2)</sup> (When Used)</b>	Calibrate with check standards. Inspect electrodes daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use. Prime buret Check rinse water reservoir.

**TABLE 8.10-14  
 Instrument Maintenance Schedule**

<b>Conductance Meter<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Electronics serviced. Replace batteries
<b>Daily<sup>(2)</sup> (When Used)</b>	Check probe and cables. Check calibration. Inspect conductivity cell

**TABLE 8.10-15  
 Instrument Maintenance Schedule**

<b>Spectrophotometer<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Replace lamp. Replace fuse.
<b>Annually</b>	Check instrument manual. Perform wavelength calibration. Replace lamp annually or when erratic response is observed.

**TABLE 8.10-16  
 Instrument Maintenance Schedule**

<b>pH Meter<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Clean electrode. Refill reference electrode.
<b>Daily<sup>(2)</sup></b>	Inspect electrode. Verify electrodes are properly connected and filled. Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.

**TABLE 8.10-17  
 Instrument Maintenance Schedule**

<b>Total Organic Carbon Analyzer (OI 1010 and Solids)</b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Check injection port septum after 50-200 runs. Perform leak test. Calibrate reagent pumps. Change sample loops. Adjust flow. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.
<b>Daily<sup>(2)</sup></b>	Check: Oxygen supply, (nitrogen supply for solids). Persulfate supply (1010 unit). Acid supply (1010 unit). Rinse water reservoir supply (1010 unit). IR millivolts for stability (after 30 min. warm-up).

**TABLE 8.10-18  
 Instrument Maintenance Schedule**

<b>Turbidimeter<sup>(1)</sup></b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>As Needed</b>	Electronics serviced.
<b>Daily<sup>(2)</sup> (When Used)</b>	Adjust linearity on varying levels of NTU standards. Standardize with NTU standards. Inspect cells.
<b>Monthly</b>	Clean instrument housing.

**TABLE 8.10-19  
 Instrument Maintenance Schedule**

<b>Digestion Block</b>	
<b>Frequency</b>	<b>Maintenance Items</b>
<b>Annually</b>	Check temperature with NIST thermometer.

**TABLE 8.10-20  
 Instrument Maintenance Schedule**

<b>Sonicator<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Replace probe tip. Disassemble and clean sonicator probe tips. Tune sonicator assembly.
<b>Daily<sup>(2)</sup> (When Used)</b>	Inspect probe tips for inconsistencies (etching/pitting).

**TABLE 8.10-21  
 Instrument Maintenance Schedule**

<b>Analytical/Top Loading Balances<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>Daily<sup>(2)</sup></b>	Check using ASTM Class 3 weights once daily or before use. Clean pan and weighing compartment.
<b>Annually</b>	Manufacturer cleaning and calibration.

**TABLE 8.10-22  
 Instrument Maintenance Schedule**

<b>Refrigerators/Walk-in Coolers<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Refrigerant system and electronics serviced.
<b>Daily<sup>(2)</sup></b>	Temperatures checked and logged.

**TABLE 8.10-23  
 Instrument Maintenance Schedule**

<b>Ovens<sup>(1)</sup></b>	
Frequency	Maintenance Items
<b>As Needed</b>	Electronics serviced.
<b>Daily<sup>(2)</sup></b>	Temperatures checked and logged.

***Footnotes to Preventive Maintenance Tables***

- 
- (1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.
  - (2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

## **STL SW8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC/HRMS)**

This method provides instrument and extraction procedures for the detection and quantitation of PCDDs (tetra through octa-chlorinated homologues) and PCDFs (tetra through octa-chlorinated homologues) in a variety of sample matrices in part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations.

Method SW8290 is used to detect dioxins and furans in variety of matrices and uses additional quality controls to allow more sophisticated determinations of detection limits and matrix spike recoveries than other routine GC and GC/MS methods.

SW8290 requires isotopically labeled analogs of target analytes to be spiked into each sample before extraction, and uses nine  $^{13}\text{C}$  labeled analogs, one furan and one dioxin at each chlorination level (13C-OCDF is not used). By adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the CDD/CDF can be made because the CDD/CDF and its labeled analog exhibit similar effects upon extraction, cleanup, concentration, and gas chromatography. Target analytes are quantitated relative to the labeled analog and therefore their calculated concentration compensates for extraction and cleanup efficiencies.

STL Method SW8290 provides a three tiered approach to reporting and detection limits. In the absence of target analytes, a sample specific estimated detection limit (EDL) is calculated based on signal-to-noise (S/N) ratios at the retention time of the analyte. The target analyte is then reported as “not detected” at the EDL. When target analytes that meet method identification criteria and are free of interferences (e.g. DPE interference) are found, they are reported down to the lowest calibration standard concentration (see reporting limits in table 1). Below the lower calibration limit (LCL) (Table 1), qualitatively confirmed analytes are reported as “estimated” down to the target detection limit

(TDL) to denote the less certain quantitation and the value is “J” flagged. The TDL is a value set by the laboratory at which there is no significant chance of false positives (usually set at one-half the LCL value). If there is a peak below the TDL, a detection limit based on the ion peaks is calculated and the target analyte is reported as “not detected” at the calculated detection limit. Second column confirmation will be performed only for 2,3,7,8-TCDF presumptive positives greater than the TDL.

A batch specific LCS (Laboratory Control Sample) is not required by Method 8290, however, STL still analyzes an LCS at a frequency of 1 per batch of 20 samples as an ongoing system and standard check. The target analyte concentrations for the LCS are given in Table 3. Sample matrix spikes and/or spike duplicates are performed only at client request. The spike concentrations are nominal values based on a full volume sample preparation (1000 mls for liquids and 10 grams for solids). If less than a full volume of sample is prepared due to sample matrix, sample availability, or method requirements, the spike amount will remain constant and therefore the spike concentrations will vary. See Table 4 and Table 5 for specific QC control and corrective action measures.

## Toxicity Equivalence Factors (TEFs)

As per client request, the 2,3,7,8-TCDD toxicity equivalence will be calculated in accordance with the procedures given in U.S. EPA "Update of Toxicity Equivalence Factors (TEFs) for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxins and Dibenzofurans (CDDs/CDFs)" March 1989 (EPA 625/3-89/016) and as described in the U.S. EPA Contract Laboratory Program Statement of Work, DFLM01.0. TEFs are assigned to each 2,3,7,8-substituted PCDDs/PCDFs in order to relate their toxicity to that of 2,3,7,8-TCDD. See Table 7 for the factors used to calculate TEFs. Note that EDL and detection limit values are not normally included in the TEQ adjusted concentration.

**TABLE 1 REPORTING LIMITS**  
**Method 8290 – STL, Sacramento**

Parameter	Method	Analyte	Water <sup>1</sup> pg/L	Soil <sup>2</sup> pg/g
Dioxins/Furans	SW8290	Dioxins		
		2,3,7,8-TCDD	10	1.0
		1,2,3,7,8-PeCDD	50	5.0
		1,2,3,4,7,8-HxCDD	50	5.0
		1,2,3,6,7,8-HxCDD	50	5.0
		1,2,3,7,8,9-HxCDD	50	5.0
		1,2,3,4,6,7,8-HpCDD	50	5.0
		OCDD	100	10
		Furans		
		2,3,7,8-TCDF	10	1.0
		1,2,3,7,8-PeCDF	50	5.0
		2,3,4,7,8-PeCDF	50	5.0
		1,2,3,4,7,8-HxCDF	50	5.0
		1,2,3,6,7,8-HxCDF	50	5.0
		1,2,3,7,8,9-HxCDF	50	5.0
		2,3,4,6,7,8-HxCDF	50	5.0
		1,2,3,4,6,7,8-HpCDF	50	5.0
		1,2,3,4,7,8,9-HpCDF	50	5.0
		OCDF	100	10

Note: "Totals" values are available upon client request.

<sup>1</sup> Based upon a 1.0 liter sample aliquot. Sensitivity of the method depends on the level of interferences rather than instrumental limitations. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

<sup>2</sup> Based upon a 10.0 gram sample aliquot. Maximum RL for samples "as received". Correction for moisture content may raise reporting limits above these levels. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

**TABLE 2 CONTROL LIMITS FOR MATRIX SPIKES and MATRIX SPIKE DUPLICATES  
Method 8290 – STL, Sacramento**

Analytical Method	Spiking Compounds	Spike Amount Water (pg/L)	Spike Amount Soil/Sediment (pg/g)	% Recovery Water <sup>(c)</sup>	% Recovery Soil/Sediment <sup>(c)</sup>	Relative Percent Difference (RPD) Water	Relative Percent Difference (RPD) Soil/Sediment	
STL SW8290	2,3,7,8-TCDF	200	20	67-140	65-134	23	25	
	2,3,7,8-TCDD	200	20	72-137	61-138	20	25	
	1,2,3,7,8-PeCDF	1000	100	77-131	70-136	20	25	
	2,3,4,7,8-PeCDF	1000	100	65-137	63-138	32	25	
	1,2,3,7,8-PeCDD	1000	100	65-141	64-139	20	25	
	1,2,3,4,7,8-HxCDF	1000	100	75-131	68-137	24	25	
	1,2,3,6,7,8-HxCDF	1000	100	69-141	62-147	32	25	
	2,3,4,6,7,8-HxCDF	1000	100	68-170	65-157	49	25	
	1,2,3,7,8,9-HxCDF	1000	100	65-158	56-151	54	25	
	1,2,3,4,7,8-HxCDD	1000	100	54-147	56-144	20	25	
	1,2,3,6,7,8-HxCDD	1000	100	70-133	68-134	20	25	
	1,2,3,7,8,9-HxCDD	1000	100	68-147	62-143	33	25	
	1,2,3,4,6,7,8-HpCDF	1000	100	77-129	70-133	20	25	
	1,2,3,4,7,8,9-HpCDF	1000	100	69-139	58-140	21	25	
	1,2,3,4,6,7,8-HpCDD	1000	100	75-130	70-132	20	25	
	OCDF	2000	200	67-140	64-142	23	25	
	OCDD	2000	200	78-127	70-140	20	25	
	(d)	<sup>13</sup> C-2,3,7,8-TCDF	2000	200	40-135	40-135		
	(d)	<sup>13</sup> C-2,3,7,8-TCDD	2000	200	40-135	40-135		
	(d)	<sup>13</sup> C-1,2,3,7,8-PeCDF	2000	200	40-135	40-135		
	(d)	<sup>13</sup> C-1,2,3,7,8-PeCDD	2000	200	40-135	40-135		
(d)	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-OCDD	4000	400	40-135	40-135			

(c) Native compound limits are STL historical limits and are subject to change.  
(d) Method default control limits. Signal-to-noise is also evaluated for data acceptability.

**TABLE 3 CONTROL LIMITS FOR LABORATORY CONTROL SAMPLES (LCS)  
Method 8290 – STL, Sacramento**

Analytical Method	Spiking Compounds	Spike Amount Water (pg/L)	Spike Amount Soil/Sediment (pg/g)	% Recovery Water <sup>(c)</sup>	% Recovery Soil/Sediment <sup>(c)</sup>	Relative Percent Difference (RPD) Water	Relative Percent Difference (RPD) Soil/Sediment	
STL SW8290	2,3,7,8-TCDF	200	20	67-140	65-134	23	25	
	2,3,7,8-TCDD	200	20	72-137	61-138	20	25	
	1,2,3,7,8-PeCDF	1000	100	77-131	70-136	20	25	
	2,3,4,7,8-PeCDF	1000	100	65-137	63-138	32	25	
	1,2,3,7,8-PeCDD	1000	100	65-141	64-139	20	25	
	1,2,3,4,7,8-HxCDF	1000	100	75-131	68-137	24	25	
	1,2,3,6,7,8-HxCDF	1000	100	69-141	62-147	32	25	
	2,3,4,6,7,8-HxCDF	1000	100	68-170	65-157	49	25	
	1,2,3,7,8,9-HxCDF	1000	100	65-158	56-151	54	25	
	1,2,3,4,7,8-HxCDD	1000	100	54-147	56-144	20	25	
	1,2,3,6,7,8-HxCDD	1000	100	70-133	68-134	20	25	
	1,2,3,7,8,9-HxCDD	1000	100	68-147	62-143	33	25	
	1,2,3,4,6,7,8-HpCDF	1000	100	77-129	70-133	20	25	
	1,2,3,4,7,8,9-HpCDF	1000	100	69-139	58-140	21	25	
	1,2,3,4,6,7,8-HpCDD	1000	100	75-130	70-132	20	25	
	OCDF	2000	200	67-140	64-142	23	25	
	OCDD	2000	200	78-127	70-140	20	25	
	(d)	<sup>13</sup> C-2,3,7,8-TCDF	2000	200	40-135	40-135		
	(d)	<sup>13</sup> C-2,3,7,8-TCDD	2000	200	40-135	40-135		
	(d)	<sup>13</sup> C-1,2,3,7,8-PeCDF	2000	200	40-135	40-135		
(d)	<sup>13</sup> C-1,2,3,7,8-PeCDD	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	2000	200	40-135	40-135			
(d)	<sup>13</sup> C-OCDD	4000	400	40-135	40-135			

- (c) Native compound limits are STL historical limits and are subject to change.  
(d) Method default control limits. Signal-to-noise is also evaluated for data acceptability.

**TABLE 4 SUMMARY OF CALIBRATION PROCEDURES**  
**Method 8290 – STL, Sacramento**

Method	Parameter	Calibration	Frequency	Acceptance Criteria	Corrective Action
SW8290 (GC/HRMS)	Dioxins/Furans	Tune using PFK.	Prior to sample analysis and at the end of the analytical sequence (no time limit for the ending PFK analysis).	Resolving power $\geq 10,000$ at $m/z=304.9824$ & $m/z=380.9760 \pm 5$ ppm of expected mass.	<ol style="list-style-type: none"> <li>1) Retune instrument.</li> <li>2) Reanalyze PFK.</li> <li>3) End resolution acceptable "as is" – assess data for impact if resolution is less than 10,000 and narrate or reinject as necessary.</li> </ol>
		Column Performance Check Solution (CPSM). Solution includes the Window Defining Mix.	Prior to 12 hrs of sample analysis.	Used to set retention times of first and last eluters. CPSM must have $\leq 25\%$ valley resolution for 2,3,7,8-TCDD	<ol style="list-style-type: none"> <li>1) Readjust windows.</li> <li>2) Evaluate system.</li> <li>3) Perform maintenance.</li> <li>4) Reanalyze CPSM.</li> <li>5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.</li> </ol>
		(5 point ICAL) Multipoint calibration.	Initially and as required.	<ol style="list-style-type: none"> <li>1) Int. std. = %RSD &lt; 30%</li> <li>2) Natives = %RSD &lt; 20%</li> <li>3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units.</li> <li>4) Ion ratios within Table 6 limits, and S/N <math>\geq 2.5</math></li> </ol>	<ol style="list-style-type: none"> <li>1) Evaluate system.</li> <li>2) Recalibrate.</li> <li>3) If all criteria are met except #4 (ratio), evaluate impact, narrate and report if no impact is found.</li> </ol>
	Daily Continuing Calibration Verification standard (CCV).	Once per 12 hours, prior to sample analysis and at the end of the analytical sequence (no time limit for the ending CCV).	<ol style="list-style-type: none"> <li>1) %D of I.S. <math>\leq 30\%</math> from avg. RRF (ICAL). (Ending %D of I.S. <math>\leq 35\%</math> from avg. RRF).</li> <li>2) %D of natives <math>\leq 20\%</math> from avg. RRF (ICAL). (Ending %D of natives <math>\leq 25\%</math> from avg. RRF).</li> <li>3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units.</li> <li>4) Ion ratios within Table 6 limits, and S/N <math>\geq 2.5</math></li> </ol>	<ol style="list-style-type: none"> <li>1) Evaluate system.</li> <li>2) Evaluate data for usability.</li> <li>3) Reanalyze (CCAL).</li> <li>4) Recalibrate (ICAL) as necessary.</li> </ol>	

**TABLE 5 SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES**  
**Method 8290 – STL, Sacramento**

Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW8290 (GC/HRMS)	Dioxins/Furans	Internal Standards	Every sample, method blank, and LCS.	1) Internal standard recovery within limits stated in Tables 2 and 3.	<ol style="list-style-type: none"> <li>1) Check chromatography for interferences. If found, flag data.</li> <li>2) Check S/N. If &lt; 10:1, re-extract sample.</li> <li>3) If S/N &gt; 10:1, evaluate data usability, flag, narrate and report.</li> <li>4) Check instrument and re-analyze the extract if a problem is found and corrected.</li> <li>5) Re-extract and re-analyze adversely affected samples.</li> </ol>
		Method blank	1 per analytical batch, not to exceed 20 field samples per matrix.	If target analyte is detected above the lower calibration limit (Table 1) or reporting limit (CRQL). For OCDD 5X the LCL. [Note 1: a "J" value positive in the method blank, or OCDD <5 X the LCL requires no corrective action – flag and report.] [Note 2: "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for positive totals in the method blank.]	<ol style="list-style-type: none"> <li>1) Re-analyze method blank if instrument carryover is suspected.</li> <li>2) If still exceeds and analyte concentration in sample &lt; CRQL or &gt; 10X blank concentration, narrate and report results.</li> <li>3) If non-compliant and analyte concentration in sample is between CRQL and 10X blank concentration, re-extract and re-analyze affected samples.</li> </ol>
		LCS (include natives)	At a frequency of 1 per 20 field samples extracted.	Refer to Table 3	<ol style="list-style-type: none"> <li>1) Review Internal Standards, as above.</li> <li>2) Evaluate data for usability.</li> <li>3) If sample results are ND and CRQL are met, no action is required – narrate and report.</li> <li>4) If samples have positives &gt; CRQL, re-extract and re-analyze affected samples for analytes outside the acceptance criteria.</li> </ol>
		Duplicates	As per client request.	Refer to Table 2 for internal standard RPD criteria.	<ol style="list-style-type: none"> <li>1) Review Internal Standards as above.</li> <li>2) If RPD exceeds, re-inject extract.</li> <li>3) Narrate any outliers.</li> </ol>
		Matrix Spike	As per client request.	Refer to Table 2.	<ol style="list-style-type: none"> <li>1) Review data for usability.</li> <li>2) Narrate outliers.</li> </ol>
		Matrix Spike Duplicate	As per client request.	Refer to Table 2.	<ol style="list-style-type: none"> <li>1) Review data for usability.</li> <li>2) Narrate outliers.</li> </ol>

**TABLE 6 CRITERIA FOR ISOTOPIC RATIO MEASUREMENT FOR PCDDs AND PCDFs**  
**Method 8290 – STL, Sacramento**

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits ( ± 15%)
4	M/(M+2)	0.77	0.65-0.89
5	(M+2)/(M+4)	1.55	1.32-1.78
6	(M+2)/(M+4)	1.24	1.05-1.43
6 <sup>a</sup>	M/(M+2)	0.51	0.43-0.59
7 <sup>b</sup>	M/(M+2)	0.44	0.37-0.51
7	(M+2)/(M+4)	1.04	0.88-1.20
8	(M+2)/(M+4)	0.89	0.76-1.02

<sup>a</sup> Used only for <sup>13</sup>C-HxCDF (Internal Standard)

<sup>b</sup> Used only for <sup>13</sup>C-HpCDF (Internal Standard)

**TABLE 7 PCDDs/PCDFs TOXICITY EQUIVALENCE FACTORS (TEF)  
Method 8290 – STL, Sacramento**

Analyte	TEF March 1989 (EPA 62/5-89/016)
2,3,7,8-TCDD	1.0
2,3,7,8-TCDF	0.1
1,2,3,7,8-PeCDF	0.05
1,2,3,7,8-PeCDD	0.5
2,3,4,7,8-PeCDF	0.5
1,2,3,4,7,8-HxCDF	0.1
1,2,3,6,7,8-HxCDF	0.1
1,2,3,4,7,8-HxCDD	0.1
1,2,3,6,7,8-HxCDD	0.1
1,2,3,7,8,9-HxCDD	0.1
2,3,4,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDF	0.1
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,6,7,8-HpCDD	0.01
1,2,3,4,7,8,9-HpCDF	0.01
OCDD	0.001
OCDF	0.001



## APPENDIX G

SAMPLE LABEL FORMAT, FIELD DATA SHEETS,  
CHAIN-OF-CUSTODY FORMS

# Analytical Parameter Request

Project #:	Date Sampled:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Name:	Date Submitted:	
Laboratory	Submittor: Fuss & O'Neill, Inc.	

Report To:	Attention:
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Invoice to:	Attention:
Mailing Address:	
City, State, Zip:	
Special Instructions:	
	Phone #:

COC #	Sample ID	COC #	Sample ID	COC #	Sample ID

Comments:	
	Blank(s) included in sample
	Duplicate(s) included in sample

## Requested Parameters

# Analytical Parameter Request

Project #: 95-079 K03	Date Sampled: _____	<b>Environmental Field Services</b>
Project Name: Willington Transfer Station	Date Submitted: _____	
Laboratory: Phoenix Environmental Labs	Submitter: <u>Fuss &amp; O'Neill, Inc.</u>	
Report to: Fuss & O'Neill, Inc.		Attention: Lynne Matteson
Copy to: _____		

Invoice to: Fuss & O'Neill, Inc.	Attention: Lynne Matteson
Mailing Address: 146 Hartford Road	Phone #: 646-2469
City, State, Zip: Manchester, CT 06040	
Special Instructions:	

COC #	Sample ID	COC #	Sample ID	COC #	Sample ID

Comments:		Blank(s) included in sample
		Duplicate(s) included in sample

Organic Method 8010/8020







# Soil Sampling Field Data

Client/Project Name:		 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<b>PROJECT #:</b>	
<b>Sample#:</b>	Sampling Location	

Sample Location Info (sketch map including location of stockpile)

## Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____  Sampling Device: Auger / Geoprobe / Shovel / Split Spoon / Trowel / Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____  Sample Depth: _____ PetroFLAG / OVM _____			

Comments:

# Soil Sampling Field Data

Client/Project Name: _____ Project Location: _____ Date: _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
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<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Geoprobe / Core Sampler / Shovel / Split Spoon / Scoop/ Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Soil Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Geoprobe / Core Sampler / Shovel / Split Spoon / Scoop/ Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Soil Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Geoprobe / Core Sampler / Shovel / Split Spoon / Scoop/ Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Soil Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			

<b>Sample #:</b>	Container	Quantity	Preservative
<b>Sample Location:</b>			
Sampler: _____ Time: _____ Sampling Device: Auger / Geoprobe / Core Sampler / Shovel / Split Spoon / Scoop/ Other ____ Field decon: Yes / No / Dedicated Type of Sample: Grab / Composite / Other _____ Generic Soil Description: _____ Sample Depth: _____ PetroFLAG / OVM _____			



## Stormwater Field Data Sheet

Client/Project Name:	Project #:
Project Location:	<u>Location ID</u>
Sample #:	

### Sample Location Info

Special Instructions:

### Sample Data

	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____	32 oz. Amber	1	H2SO4
	250 mL Plastic	1	HNO3
	500 mL Plastic	1	H2SO4
Estimated Flow Rate(GPM) _____ Stagnant / Dry / Other _____	250 mL Plastic	1	As Is
	1 L Plastic	1	As Is
Appearance:			
Comments:			

### Field Parameters

Parameter	Instrument	Value
pH		
Temperature		
rain pH		

Comments:

# Surface Water Field Data

Client/Project Name:	Project #:	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
Project Location:	<u>Monitoring Location</u>	
Sample #:		

## Sample Location Info

## Sample Data

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____			
Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____			
Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: <input type="text"/> Filter: Disposable / Other _____ Field Decon: Tubing / Other _____ Appearance:  Comments:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

Comments:

# Surface Water Field Data

Client/Project Name: _____ Project Location: _____ Date: _____ Weather: _____	<b>PROJECT #:</b> _____	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
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<b>Sample #:</b> _____ <b>Sample Location</b>	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____ Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____ Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: _____ Filter: Disposable / Other _____ Field Decon: Tubing / Other _____ Appearance:  Comments:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

<b>Sample #:</b> _____ <b>Sample Location</b>	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____ Estimated Flow Rate (GPM) _____ Stagnant / Dry / Other _____ Filtered in Field: No / @ Well Method of Filtration: Pressure / Vacuum / Syringe Pump ID#: _____ Filter: Disposable / Other _____ Field Decon: Tubing / Other _____ Appearance:  Comments:			

## Field Parameters

Parameter	Instrument ID	Value
pH		
Temp		
Spec. Cond.		

# Trip Blank Field Data

Client/Project Name:  Project Location:	<b>PROJECT #:</b>	 <b>FUSS &amp; O'NEILL</b> <i>Disciplines to Deliver</i>
<b>Sample#:</b>	<u>Well ID</u> Trip Blank	

Sample Data	Container	Quantity	Preservative
Date: _____ Time: _____ Sampler: _____ Weather: _____	Voa	2	HCl
Blank Water Supplied By:    Lab / F&O / Other _____          Comments:			

Comments: