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US ARMY CORPS OF ENGINEERS
New England District

Contract No. DACW33-96-0005
Delivery Order No. 59
November 8, 2000

QUALITY ASSURANCE PROJECT PLAN

CENTREDALE MANOR RESTORATION PROJECT SUPERFUND SITE BASELINE RISK ASSESSMENT, INITIAL PROJECT PLANNING AND SUPPORT

Task 15
Tissue Chemistry QAPP

1.0 TITLE AND APPROVAL PAGE (EPA WORKSHEET #1)

Site Name/Project Name: *Centredale Manor Restoration Project Superfund Site Baseline Risk Assessment, Initial Project Planning and Support*
Site Location: *Greystone Pond, Allendale Pond and Woonasquatucket River, North Providence, Rhode Island*

Document Title: *Centredale Manor Task 15 Quality Assurance Project Plan*

Lead Organization (Agency, State, Tribe, Federal Facility, PRP, or Grantee): *Battelle Duxbury Operations*

Preparer's Name and Organizational Affiliation: *Deirdre Dahlen/Battelle Duxbury Operations*

Preparer's Address and Telephone Number: *397 Washington Street, Duxbury, MA 02332 (781) 934-5253*

Preparation Date (Day/Month/Year): *11/8/00*

Investigative Organization's Project Manager: Donald Gunster 11/9/00
Signature/Date
Don Gunster/Battelle Duxbury Operations 11/8/00
Printed Name/Organization

Investigative Organization's Project QA Officer: Rosanna Buhl 11-9-00
Signature/Date
Rosanna Buhl/Battelle Duxbury Operations 11/8/00
Printed Name/Organization

Lead Organization's Project Manager: Donald Gunster 11/9/00
Signature/Date
Don Gunster/Battelle Duxbury Operations 11/8/00
Printed Name/Organization

Approval Signature:
Signature/Date
Andy Beliveau/ QA Officer
Printed Name/Title
EPA Region 1
Approval Authority

Other Approval Signatures:
Signature/Date
Laureen Borochaner/USACE NAE Project Officer
Printed Name/Title

Document Control Number: *Not applicable.*

2.0 OF CONTENTS AND DOCUMENTATION FORMAT

2.1 Table of Contents

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ATTACHMENTS

FIGURES

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This is a Project-Specific Assurance Project Plan.

2.2 Documentation Control Format

Document control format follows Region I, EPA-New England Compendium of Quality Assurance Project Plan Guidance. The format of this QAPP includes a synopsis of QAPP elements followed by all required EPA-NE QAPP Worksheets provided in an Attachment (Attachment A). Other relevant documents/forms are included in Attachments B through E.

2.3 Document Control Numbering System

A document control numbering system is not required for smaller projects and will not be used for the Centredale Manor Task 15 QAPP.

2.4 EPA-NE QAPP Worksheet #2

EPA-NE QAPP Worksheet #2 is included in Attachment A. This QAPP is intended to solely document analytical activities conducted under Task 15 of Delivery Order #59, *Centredale Manor Restoration Project Superfund Site Baseline Risk Assessment, Initial Project Planning and Support*. As a result, the following field-related, EPA-NE QAPP Worksheets are not applicable to this QAPP and are circled (and bolded) on EPA-NEQAPP Worksheet #2 to indicate that these worksheets are not included in this QAPP:

- EPA-NE QAPP Worksheet #7
- EPA-NE QAPP Worksheet #12a
- EPA-NE QAPP Worksheet #12b
- EPA-NE QAPP Worksheet #13
- EPA-NE QAPP Worksheet #14
- EPA-NE QAPP Worksheet #15
- EPA-NE QAPP Worksheet #17
- EPA-NE QAPP Worksheet #18
- EPA-NE QAPP Worksheet #19
- EPA-NE QAPP Worksheet #22a
- EPA-NE QAPP Worksheet #22b
- EPA-NE QAPP Worksheet #23a
- EPA-NE QAPP Worksheet #23b
- EPA-NE QAPP Worksheet #25

3.0 DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET

EPA-NE QAPP Worksheet #3 and #4 are included in Attachment A. Project personnel identified on EPA-NE QAPP Worksheet #4 are representative of trained staff at all participating laboratories. If the specified project personnel are not available to conduct project tasks at the time of sample receipt, then alternate staff with a similar level of training will be assigned to the project team. Alternate staff will receive a copy of the QAPP and complete the sign-off sheet.

4.0 PROJECT ORGANIZATION

4.1 Project Organizational Chart

The project organizational chart is shown in Figure 1 (EPA-NE QAPP Worksheet #5a, Attachment A).

4.2 Communication Pathways

Communication pathways are described in EPA-NE QAPP Worksheet #5b (Attachment A).

4.2.1 Modifications to Approved QAPP

Out of scope work will not be conducted without notification and approval (verbal/written) from USACE NAE. All deviations from protocols described in this QAPP will be documented and approved by the Project Manager and discussed in the final report. Notification and approval of modifications will adhere to the communication pathways described in EPA-NE QAPP Worksheet #5b (Attachment A)

4.3 Personnel Responsibilities and Qualifications

Personnel responsibilities and qualifications are described in EPA-NE QAPP Worksheet #6 (Attachment A). Personnel resumes for project personnel are included in Attachment B. Field sampling and risk assessment activities are outside the scope of Task 15 (Tissue Chemistry) and no project personnel are listed in this capacity.

4.4 Special Training Requirements/Certification

Not applicable.

5.0 PROJECT PLANNING/PROJECT DEFINITION

Case team members responsible for planning the project included Laureen Borochaner (USACE NAE), Cornell Rosiu (EPA Region I), Andy Beliveau (EPA Region I), Karen Foster, Don Gunster, Deirdre Dahlen and Karen Lesniak.

5.1 Project Planning Meetings

One formal project-planning meeting was held on July 17, 2000, though this meeting did not pertain to the tissue chemistry task. Even so, EPA-NE QAPP Worksheet #8a was completed for this meeting (Attachment A). Several informal meetings were held between case team members (*e.g.*, Project Manager, chemistry leaders) to discuss scheduling activities and availability of personnel to perform project tasks. These informal meetings are not itemized in EPA-NE QAPP Worksheet #8a.

5.2 Problem Definition/Site History and Background

Problem definition, site history and background are outlined in EPA-NE QAPP Worksheet #8b (Attachment A).

Field sampling activities and a synopsis of non-direct measurement data/information from all site reports is outside the scope of this task (Task 15 conducted under Delivery Order #59).

6.0 PROJECT DESCRIPTION AND SCHEDULE

Battelle has been contracted by USACE NAE to provide analytical support to EPA-NE. Biological tissue samples (*i.e.*, tree swallow egg, nestling, and diet) that were collected by EPA-NE on May 26, 2000, June 12, 2000, and June 19, 2000 will be analyzed by Battelle for dioxins/furans, 1,2,4,5,7,8-hexachloroxanthene (HCX), and PCB congeners. Samples have been held (frozen – personnel communication from Cornell Rosiu and Andy Beliveau) in EPA-NE's custody since collection. Battelle will submit a letter data report to USACE NAE by December 22, 2000. The letter data report will

include summary report tables and QA/QC narratives. One copy of each analytical data package (*e.g.*, dioxins/furans/HCX) will be provided to USACE NAE for potential third party validation. The data packages (raw data) will be submitted by January 5, 2001.

6.1 Project Overview

An overview of the project is provided in EPA-NE QAPP Worksheets #9a, 9b, 9c and 9d (Attachment A).

6.2 Project Schedule

A tentative project schedule, outlining project tasks and deliverables is provided in EPA-NE QAPP Worksheet #10 (Attachment A). The procedure for notifying project participants concerning project schedule delays is described in Section 4.2. The Battelle Duxbury laboratory holds weekly scheduling meetings to discuss and resolve resource and/or time constraints.

7.0 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA

7.1 Project Quality Objectives

Project quality objectives are discussed in EPA-NE QAPP Worksheet #11a (Attachment A).

7.2 Measurement Performance Criteria

Measurement performance criteria (MPC) used to assess data quality and project quality objectives are discussed EPA-NE QAPP Worksheet #11b (Attachment A). All work conducted under Task 15 of this Delivery Order pertains strictly to fixed laboratory activities, and as a result oversight split sampling, field-screening analyses, and field QC activities will not be discussed in this QAPP.

8.0 SAMPLING PROCESS DESIGN

All field sampling activities (*e.g.*, design) were the responsibility of EPA-NE and followed methods described in Custer *et al.*, (2000). This QAPP is solely intended to discuss fixed laboratory analytical activities and as a result field sampling design activities will not be discussed in this document. EPA-NE QAPP Worksheets applicable solely to sampling design activities (*i.e.*, EPA-NE QAPP Worksheets #12a and 12b) will not be completed or included in this document.

8.1 Sampling Design

Not applicable.

9.0 SAMPLING PROCEDURES AND REQUIREMENTS

Field sampling was conducted by EPA-NE in May and June 2000 and samples remained in EPA-NE's custody until October 17, 2000 when EPA-NE shipped samples to Battelle for tissue processing. Sampling procedures and requirements followed Custer (2000). EPA-NE QAPP Worksheets, applicable solely to field sampling activities (*i.e.*, EPA-NE QAPP Worksheets #12b, 13, 14, 15), will not be completed or included in this document.

9.1 Sampling Procedures

Not applicable.

9.2 Sampling SOP Modifications

Not applicable.

9.3 Cleaning and Decontamination of Equipment/Sample Containers

Not applicable.

9.4 Field Equipment Calibration

Not applicable.

9.5 Field Equipment Maintenance, Testing and Inspection Requirements

Not applicable.

9.6 Inspection and Acceptance Requirements for Supplies/Sample Containers

Containers used to stored samples after collection were provided by EPA-NE. Battelle will be responsible for providing sample containers for tissue samples after processing (freeze-drying, homogenization). Sample containers used to store biological samples for chemical analysis will be pre-cleaned glass jars with Teflon-lined hard caps purchased from Environmental Sampling Supply (ESS). A Certificate of Compliance accompanies sample containers and certifies that sample containers are contaminant free (organics, metals). Additional containers will be provided as contingency in case any of the containers are deemed unacceptable.

10.0 SAMPLE HANDLING, TRACKING AND CUSTODY REQUIREMENTS

Field sampling and tracking were the responsibility of EPA-NE. Battelle's laboratory custody requirement procedures are discussed in Section 10.3 of this document.

10.1 Sample Collection Documentation

Documentation of sample collection data (*i.e.*, field notes, field documentation management system) was the responsibility of EPA-NE and is not discussed in this QAPP.

10.2 Sample Handling and Tracking System

Sample handling and tracking was the responsibility of EPA-NE.

10.3 Laboratory Sample Custody

The custody of samples, and therefore the sample tracking and integrity, are assured through the following standard procedures, which are defined in Battelle SOPs ASAT.II-007 (Columbus), 6-010 (Duxbury), and MSL-A-002 (MSL). Highlights of the procedures include:

- Trained laboratory sample custodians are designated at each analytical laboratory.
- Upon receipt, samples are inspected to verify that (1) integrity is intact (containers are sealed and intact), (2) the sample label and custody forms agree, (3) all shipped samples have been received, and (4) holding temperatures were maintained.
- Sample receipt and the receipt conditions are documented, as are any discrepancies, which are also communicated to the Project Manager immediately.
- Custody forms are signed by the sample custodian and samples are logged into a formal sample receipt system to provide a permanent laboratory record and laboratory sample IDs are assigned.
- Samples are stored frozen (–20 °C) in a limited access area.
- Sample receipt and holding times are communicated to the laboratory manager who adds the samples to the laboratory schedule.
- The sample custodian retains custody of the samples until they are transferred from the holding location to the laboratory for analysis. The relinquishing of samples by the custodian and the receipt of sample by the analyst are documented.
- Internal laboratory documentation tracks sample custody location and storage conditions throughout processing and analysis.
- Sample archival and disposal are documented according to SOPs.

11.0 FIELD ANALYTICAL METHOD REQUIREMENTS

Field sampling and associated analyses (*i.e.*, screening; on-site mobile laboratory) were the responsibility of EPA-NE. EPA-NE QAPP Worksheets (*i.e.*, EPA-NE QAPP Worksheets #17, #18, and #19) that document field related procedures and requirements are not applicable to this QAPP.

12.0 FIXED LABORATORY ANALYTICAL METHOD REQUIREMENTS

12.1 Fixed Laboratory Analytical Methods and SOPs

Analytical methods and SOPs used to analyze tissue samples for required parameters are defined in EPA-NE QAPP Worksheet #20 (Attachment A). Copies of relevant SOPs are provided in Attachment E.

12.2 Fixed Laboratory Analytical Method/SOP Modifications

Pertinent laboratory SOPs are defined in EPA-NE QAPP Worksheet #20 (Attachment A). Three of the SOPs listed were recently revised and copies of the updated SOPs are provided in Attachment E. Four of the SOPs defined in EPA-NE QAPP Worksheet #20 were being developed at the time the draft QAPP was submitted (10/24/00). Copies of these new SOPs are now provided in Attachment E. SOP L-20 (Battelle Duxbury SOP 5-157) also defined in EPA-NE QAPP Worksheet #20 will be modified solely to include mass ions for PCB congeners; modifications to this SOP are now detailed in Attachemnt E. Otherwise, no modifications to the laboratory SOPs are planned. However, if modifications are necessary to meet PQOs, then the notification, approval and documentation process will follow methods described in Section 4.2.

12.3 Fixed Laboratory Instrument Calibration

Instrument maintenance and calibration information is provided in EPA-NE QAPP Worksheet #21 (Attachment A).

12.4 Fixed Laboratory Instrument/Equipment Maintenance, Testing and Inspection Requirements

Instrument maintenance, testing and inspection requirements are detailed in EPA-NE QAPP Worksheet #21 (Attachment A).

12.5 Fixed Laboratory Inspection and Acceptance Requirements for Supplies

The Laboratory Manager or his/her designee is responsible for tracking laboratory supplies to ensure that sufficient quantities are available to meet project/laboratory needs. Supplies used in the preparation of samples, and which may contribute to laboratory contamination, are checked and approved by the Laboratory Manager prior to use. Reagent/Receipt logbooks are maintained by the laboratory and document inspection and acceptance of laboratory supplies.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Sampling Quality Control

Not applicable.

13.2 Analytical Quality Control

A routine set of QC samples will be analyzed for each analytical parameter to monitor and measure data quality against a set of project quality objectives.

13.2.1 Field Analytical QC

Not applicable.

13.2.2 Fixed Laboratory QC

Fixed laboratory QC is identified for each analytical parameter in EPA-NE QAPP Worksheet#24a and #24b (Attachment A).

14.0 DATA ACQUISITION REQUIREMENTS

It is outside the scope of this work to perform an evaluation of historical data and other background information. Consequently, EPA-NE QAPP Worksheet #25 will not be completed or included in this QAPP.

15.0 DOCUMENTATION, RECORDS AND DATA MANAGEMENT

All documentation will conform to Battelle SOP 6-017 (*e.g.*, all original data are recorded in ink, corrections are made by placing a single line through the incorrect entry with a date, initials, and explanation). Data acquisition and reduction procedures, as well as the formulas applied to produce final data are detailed in the SOPs cited in EPA-NE QAPP Worksheet #20 (Attachment A). Statistical evaluations will be performed on all quality control samples. Equations for calculating quality control statistical evaluations are provided in EPA-NE QAPP Worksheet #30 (Attachment A). Data are reported as spreadsheet tables following standard reporting formats.

15.1 Project Documentation Records

Project documentation records are identified in EPA-NE QAPP Worksheet #26 (Attachment A).

15.2 Field Analysis Data Package Deliverables

Not applicable.

15.3 Fixed Laboratory Data Package Deliverables

Contents of each data package are described in EPA-NE QAPP Worksheet #9a (Attachment A). Battelle will submit a letter data report and one copy of each analytical data package (*e.g.*, dioxin/furans/HCX) to USACE NAE.

The letter data report will include summary report tables (validated data) and QA/QC narratives. The QA/QC narratives provide a discussion of the quality control results and a description of any MPC exceedences including the impact, if any, they have on the overall data.

Analytical data packages will include raw data (see Attachment C) that will be used for potential third party validation.

The schedule for these deliverables is outlined in EPA-NE QAPP Worksheet #10 (Attachment A).

15.4 Data Reporting Formats

Data are reported as spreadsheet tables following standard reporting formats. The final verified data will include summary Excel tables in hardcopy format for all field and QC data. Data reporting format and content is discussed in detail in EPA-NE QAPP Worksheet #9a (Attachment A).

15.5 Data Handling and Management

Upon receipt of samples at Battelle, samples are logged into the tracking system and assigned unique Ids. A copy of the sample custody log-in information is provided to the Project Manager and Task Leader(s). The laboratory QAPP is prepared, distributed to the project team, and a kick-off meeting held to review project tasks, deliverables and schedules. The Sample Preparation chemist prepares the samples in the laboratory for organic analysis and assigns each batch of samples with a unique batch ID. Sample preparation information pertaining to sample Ids and weights are either recorded manually or stored in electronic spreadsheets located on the Battelle network and are electronically accessible to the Analysts. Data handling and management procedures related to data acquisition and reduction are further described in EPA-NE QAPP Worksheet #9a (Attachment A).

15.6 Data Tracking and Control

Data acquired from Battelle's GC/MS and GC/HRMS systems are backed up each month, and the data operating systems provide a software package for quick and easy data retrieval.

Final excel tables are named according to the sample matrix, batch ID and analysis parameter. For example, PCB data for tissue batch 00-001 would be named "t00-001ms.xls". A copy of the final excel file(s) is electronically transferred to the Project Manager for the project files. The GC/MS and GC/HRMS Analyst also archive a copy of the final files in the GC facility.

16.0 ASSESSMENTS AND RESPONSE ACTIONS

Each participating laboratory is responsible for performing a QA audit on the analytical data to ensure adherence to project quality objectives and the QAPP. Assessment and response actions are documented in EPA-NE QAPP Worksheet #27a and #27b (Attachment A).

16.1 Planned Assessments

Planned assessments are documented in EPA-NE QAPP Worksheet #27c (Attachment A)

16.2 Assessment Findings and Corrective Action Responses

Results of QA audits will be reported to the analytical Task Leader and the Project Manager (EPA-NE QAPP Worksheet #27b, Attachment A). The audit reports will define any errors, deficiencies, or deviations from the QAPP. The responsible analyst documents the corrective action on the audit report and submits the audit report to the Project Manager for review and approval. Battelle audit reports are available for review at Battelle Duxbury.

16.3 Additional QAPP Non-Conformances

Project personnel are responsible for documenting corrective action procedures taken outside of the formal QA audit. Documentation is maintained in the analytical data packages and communicated to the Project Manager for approval.

17.0 QA MANAGEMENT REPORTS

Management is copied on QA audit findings. The project personnel responsible for reviewing and approving audit reports are outlined in EPA-NE QAPP Worksheet #28 (Attachment A). The QA Officer communicates the status of each project and any programmatic issues to the Duxbury Operations Manager during Quarterly briefings.

18.0 VERIFICATION AND VALIDATION REQUIREMENTS

Data validation described in this QAPP describes what Battelle and the other participating laboratories will perform internally. One copy of each data package (*e.g.*, dioxins/furans/HCX) will be submitted to USACE NAE for potential third party validation purposes.

Participating laboratories are responsible for verification and validation of data packages. Verification and validation requirements at Battelle Duxbury will follow internal laboratory SOPs and will encompass the verification and validation steps described in detail in EPA-NE QAPP Worksheet #9a (Attachment A).

19.0 VERIFICATION AND VALIDATION PROCEDURES

Verification and validation procedures are discussed in EPA-NE QAPP Worksheets #29a, b and c (Attachment A). Data validation is the responsibility of those immediately responsible for overseeing and/or performing analyses, data entry, data reduction, and data reporting. Data validation procedures conducted at Battelle Duxbury will follow SOPs 6-027. Similarly, data validation procedures conducted at Battelle Columbus will follow SOPs ASAT.II-003 and ASAT.II-010. An example of a data validation checklist used at Battelle Duxbury is provided in Attachment D. A series of reviews by technical personnel will be implemented to ensure that the data generated for this work assignment meet the data quality objectives. These reviews are further described in EPA-NE QAPP Worksheet #9a (Attachment A).

20.0 DATA USABILITY/RECONCILIATION WITH PROJECT QUALITY OBJECTIVES

Data usability assessment activities are documented in EPA-NE QAPP Worksheets #9a and #30 (Attachment A)

21.0 REFERENCES

Custer, C.M. and T.W. Custer. 2000. Pilot Study of Eco Risks to Tree Swallows. USEPA/USGS IAG#DW14 940228-01-1.

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ATTACHMENT A

EPA-NE QAPP Worksheets

EPA-NE QAPP Worksheet #2

Site Name/Project Name: Centredale Manor
Site Location: Greystone Pond, Allendale Pond, and Woonasquatucket River, North Providence, Rhode Island
Site Number/Code: 016P
Operable Unit:
Contractor Name: Battelle Duxbury Operations
Contractor Number: DACW33-96-D-0005
Contract Title: Centredale Manor Restoration Project Superfund Site Baseline Risk Assessment, Initial Project Planning and Support
Work Assignment Number: Delivery Order #59
Anticipated date of QAPP Implementation:10/25/00

1. Identify Guidance used to prepare QAPP: *Region I, EPA-NE Compendium QAPP Guidance, Draft Final September 1998*

2. Identify EPA Program: *Superfund*

3. Identify approval entity: *EPA-NE or State: U.S. Army Corps of Engineers, New England Division and EPA-NE* or other entity:

4. Indicate whether the QAPP is a generic program QAPP or a project-specific QAPP. (underline one)

5. List dates of scoping meetings that were held: July 17, 2000

6. List title of QAPP documents and approval dates written for previous site work, if applicable:

Title	Approval Date
Sampling and Analysis Plan Woonasquatucket River Sediment Investigation, Centredale Manor Site, North Providence, Rhode Island. (prepared by Tetra Tech NUS, Inc.)	September 1999

7. List organizational partners (stakeholders) and connection with EPA and/or State:

U.S. Army Corps of Engineers, New England District

EPA Region I

State of Rhode Island

8. List data users:

U.S. Army Corps of Engineers, New England District

EPA Region I

9. If any required QAPP Elements (1-20), Worksheets and/or Required Information are not applicable the project, then circle the omitted QAPP Elements, Worksheets and Required Information on the attached Table. Provide an explanation for their exclusion below:
EPA-NE QAPP Worksheet #7 - No special training required.

This QAPP pertains strictly to Fixed Laboratory Analytical activities – field sampling is not discussed. Therefore all worksheets that pertain to field sampling activities are not included, as follows EPA-NE QAPP Worksheets #12 (a and b), #13 through #15, #17 through #19, #22 (a and b), #23 (a and b), and #25.

EPA-NE QAPP Worksheet # 2a

Bold QAPP Elements, Worksheets and/or Required Information that are not applicable to the project and provide an explanation on EPA-NE QAPP Worksheet #2, Item 9.

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA-NE QAPP Worksheet #	REQUIRED INFORMATION
Project Management and Objectives			
A1	1.0 Title and Approval Page	1	- Title and Approval Page
A2	2.0 Table of Contents and Document Format 2.1 Table of Contents 2.2 Document Control Format 2.3 Document Control Numbering System 2.4 EPA-NE QAPP Worksheet #2	2	- Table of Contents - EPA-NE QAPP Worksheet
A3	3.0 Distribution List and Project Personnel Sign-off Sheet	3 4	- Distribution List - Project Personnel Sign-off Sheet
A4, A8	4.0 Project Organization 4.1 Project Organizational Chart 4.2 Communication Pathways 4.2.1 Modifications to Approved QAPP 4.3 Personnel Responsibilities and Qualifications 4.4 Special Training Requirements/ Certification	5a 5b 6 7	- Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - **Special Personnel Training Requirements Table**
A5	5.0 Project Planning/Project Definition 5.1 Project Planning Meetings 5.2 Problem Definition/Site History and Background	8a 8b	- Project Scoping Meeting Attendance Sheet with Agenda and other Project Planning Meeting Documentation - Problem Definition/Site History and Background - EPA-NE DQO Summary Form - Site Maps (historical and present)
A6	6.0 Project Description and Schedule 6.1 Project Overview 6.2 Project Schedule	9a 9b 9c 9d 10	- Project Description - Contaminants of Concern and Other Target Analytes Table - Field and Quality Control Sample Summary Table - Analytical Services Table - System Designs - Project Schedule Timeline Table
A7	7.0 Project Quality Objectives and Measurement Performance Criteria 7.1 Project Quality Objectives 7.2 Measurement Performance Criteria	11a 11b	- Project Quality Objectives/Decision Statements - Measurement Performance Criteria Table

EPA-NE QAPP Worksheet # 2a (continued)

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA-NE QAPP Worksheet #	REQUIRED INFORMATION
Measurement/Data Acquisition			
B1	8.0 Sampling Process Design 8.1 Sampling Design Rationale	**12a** **12b**	- **Sampling Design and Rationale** - **Sampling Locations, Sampling and Analysis Method/SOP Requirements Table** - **Sample Location Map**
B2, B6, B7, B8	9.0 Sampling Procedures and Requirements 9.1 Sampling Procedures 9.2 Sampling SOP Modifications 9.3 Cleaning and Decontamination of Equipment/Sample Containers 9.4 Field Equipment Calibration 9.5 Field Equipment Maintenance, Testing and Inspection Requirements 9.6 Inspection and Acceptance Requirements for Supplies/Sample Containers	**13** **12b** **14** **15**	- **Sampling SOPs** - **Project Sampling SOP Reference Table** - **Sampling Container, Volumes and Preservation Table** - **Field Sampling Equipment Calibration Table** - **Cleaning and Decontamination SOPs** - **Field Equipment Maintenance, Testing and Inspection Table**
B3	10.0 Sample Handling, Tracking and Custody Requirements 10.1 Sample Collection Documentation 10.1.1 Field Notes 10.1.2 Field Documentation Management System 10.2 Sample Handling and Tracking System 10.3 Sample Custody	16	- Sample Handling, Tracking and Custody SOPs - Sample Handling Flow Diagram - Sample Container Label (Sample Tag) - Chain-of-Custody Form and Seal
B4, B6, B7, B8	11.0 Field Analytical Method Requirements 11.1 Field Analytical Methods and SOPs 11.2 Field Analytical Method/SOP Modifications 11.3 Field Analytical Instrument Calibration 11.4 Field Analytical Instrument/ Equipment Maintenance, Testing and Inspection Requirements 11.5 Field Analytical Inspection and Acceptance Requirements for Supplies	**17** **18** **19**	- **Field Analytical Methods/SOPs** - **Field Analytical Method/SOP Reference Table** - **Field Analytical Instrument Calibration Table** - **Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Table**

EPA-NE QAPP Worksheet # 2a (continued)

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA-NE QAPP Worksheet #	REQUIRED INFORMATION
B4, B6, B7, B8	12.0 Fixed Laboratory Analytical Method Requirements 12.1 Fixed Laboratory Analytical Methods and SOPs 12.2 Fixed Laboratory Analytical Method/SOP Modifications 12.3 Fixed Laboratory Instrument Calibration 12.4 Fixed Laboratory Instrument/ Equipment Maintenance, Testing and Inspection Requirements 12.5 Fixed Laboratory Inspection and Acceptance Requirements for Supplies	20 21	- Fixed Laboratory Analytical Methods/SOPs - Fixed Laboratory Analytical Method/SOP Reference Table - Fixed Laboratory Instrument Maintenance and Calibration Table
B5	13.0 Quality Control Requirements 13.1 Sampling Quality Control 13.2 Analytical Quality Control 13.2.1 Field Analytical QC 13.2.2 Fixed Laboratory QC	**22a** **22b** **23a** **23b** 24a 24b	**Sampling** - **Field Sampling QC Table** - **Field Sampling QC Table cont.** **Analytical** - **Field Analytical QC Sample Table** - **Field Analytical QC Sample Table cont.** - **Field Screening/Confirmatory Analysis Decision Tree** - Fixed Laboratory Analytical QC Sample Table - Fixed Laboratory Analytical QC Sample Table cont.
B9	14.0 Data Acquisition Requirements	**25**	- **Non-Direct Measurements Criteria and Limitations Table**
A9, B10	15.0 Documentation, Records and Data Management 15.1 Project Documentation and Records 15.2 Field Analysis Data Package Deliverables 15.3 Fixed Laboratory Data Package Deliverables 15.4 Data Reporting Formats 15.5 Data Handling and Management 15.6 Data Tracking and Control	26	- Project Documentation and Records Table - Data Management SOPs
Assessment/Oversight			
C1	16.0 Assessments and Response Actions 16.1 Planned Assessments 16.2 Assessment Findings and Corrective Action Responses 16.3 Additional QAPP Non-Conformances	27a 27b 27c	- Assessment and Response Actions - Project Assessment Table - Project Assessment Plan - Audit Checklists
C2	17.0 QA Management Reports	28	- QA Management Reports Table

EPA-NE QAPP Worksheet # 2a (continued)

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA-NE QAPP Worksheet #	REQUIRED INFORMATION
Data Validation and Usability			
D1	18.0 Verification and Validation Requirements		- Validation Criteria Documents *
D2	19.0 Verification and Validation Procedures	29a 29b 29c	- Data Evaluation Process - Data Validation Summary Table - Data Validation Modifications
D3	20.0 Data Usability/Reconciliation with Project Quality Objectives	30	- Data Usability Assessment

* Include Data Validation Criteria Document as an attachment to the QAPP if Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses will not be used for validating project data.

Note: Required project-specific information should be provided in tabular format, as much as practicable. However, sufficient written discussion in text format should accompany these tables. Certain sections, by their nature, will require more written discussion than others. In particular, Section 8.0 should provide an in-depth explanation of the sampling design rationale and Sections 18-20 should describe the procedures and criteria that will be used to verify, validate and assess data usability.

EPA-NE QAPP Worksheet #3 - Rev. 10/99

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Document Control Number
Laureen Borochaner	Project Manager	U.S. Army Corps of Engineers, New England District	978-318-8802	NA
Kenneth E. Hitch	Chief, Engineering/Planning Division	U.S. Army Corps of Engineers, New England District	978-318-8500	NA
Marie Wojtas	Chemist	U.S. Army Corps of Engineers, New England District	978-318-8175	NA
Anna Krasko	Remedial Project Manager	EPA Region I	617-918-1232	NA
Cornell Rosiu	Work Assignment Manager	EPA Region I	617-918-1345	NA
Andy Beliveau	QA Officer	EPA Region I	781-860-4607	NA
Karen Foster	Program Manager	Battelle Duxbury	781-952-5370	NA
Don Gunster	Project Manager	Battelle Duxbury	781-952-5378	NA
Rosanna Buhl	QA Officer	Battelle Duxbury	781-952-5309	NA
Deirdre Dahlen	Tissue Chemistry Task Leader	Battelle Duxbury	781-952-5253	NA
Betsy Barrows	Freeze-Drying Task Leader	Battelle Marine Sciences Laboratory (MSL)	360-681-3643	NA
Deborah Coffey	QA Officer	Battelle MSL	360-681-3645	NA
Karen Tracy	Dioxin/Furan/HCX Task Leader	Battelle Columbus	614-424-4028	NA
Charles D. Lawrie	QA Officer	Battelle Columbus	614-424-3932	NA

EPA-NE QAPP Worksheet #4 - Rev. 10/99

Project Personnel Sign-Off Sheet

Organization: Battelle Duxbury

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Karen Foster	Program Manager	781-952-5370			
Don Gunster	Project Manager	781-952-5378			
Rosanna Buhl	QA Officer	781-952-5309			
Mark Guilmain	QA Coordinator	781-952-5316			
Deirdre Dahlen	Tissue Chemistry Task Leader	781-952-5253			
Robert Lizotte	Laboratory Manager	781-952-5235			
Micheal Meara	Laboratory Sample Custodian	781-952-5270			
Beth Kitson	Sample Preparation Chemist	781-952-5241			
Julie Fredriksson	PCB Task Leader/ GC/MS Analyst	781-952-5252			

Organization: Battelle Columbus

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Karen Tracy	Dioxin/Furan/HCX Task Leader	614-424-4028			
Charles D. Lawrie	QA Officer	614-424-3932			
Mary E. Schrock	Laboratory Manager	614-424-4976			
Mark F. Misita	Sample Preparation Chemist and Sample Custodian	614-424-7884			
Henry H. Pham	Sample Preparation Chemist	614-424-7849			
Wesley H. Baxter	Sample Preparation Chemist	614-424-7849			
Joseph E. Tabor	GC/HRMS Analyst	614-424-5130			

Organization: Battelle Marine Sciences Laboratory (MSL)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Betsy Barrows	Freeze-Drying Task Leader	360-681-3643			
Carolynn Suslick	Sample Custodian/Data Manager	360-681-3624			
Deborah Coffey	QA Officer	360-681-3645			

EPA-NE QAPP Worksheet #5a - Rev. 10/99

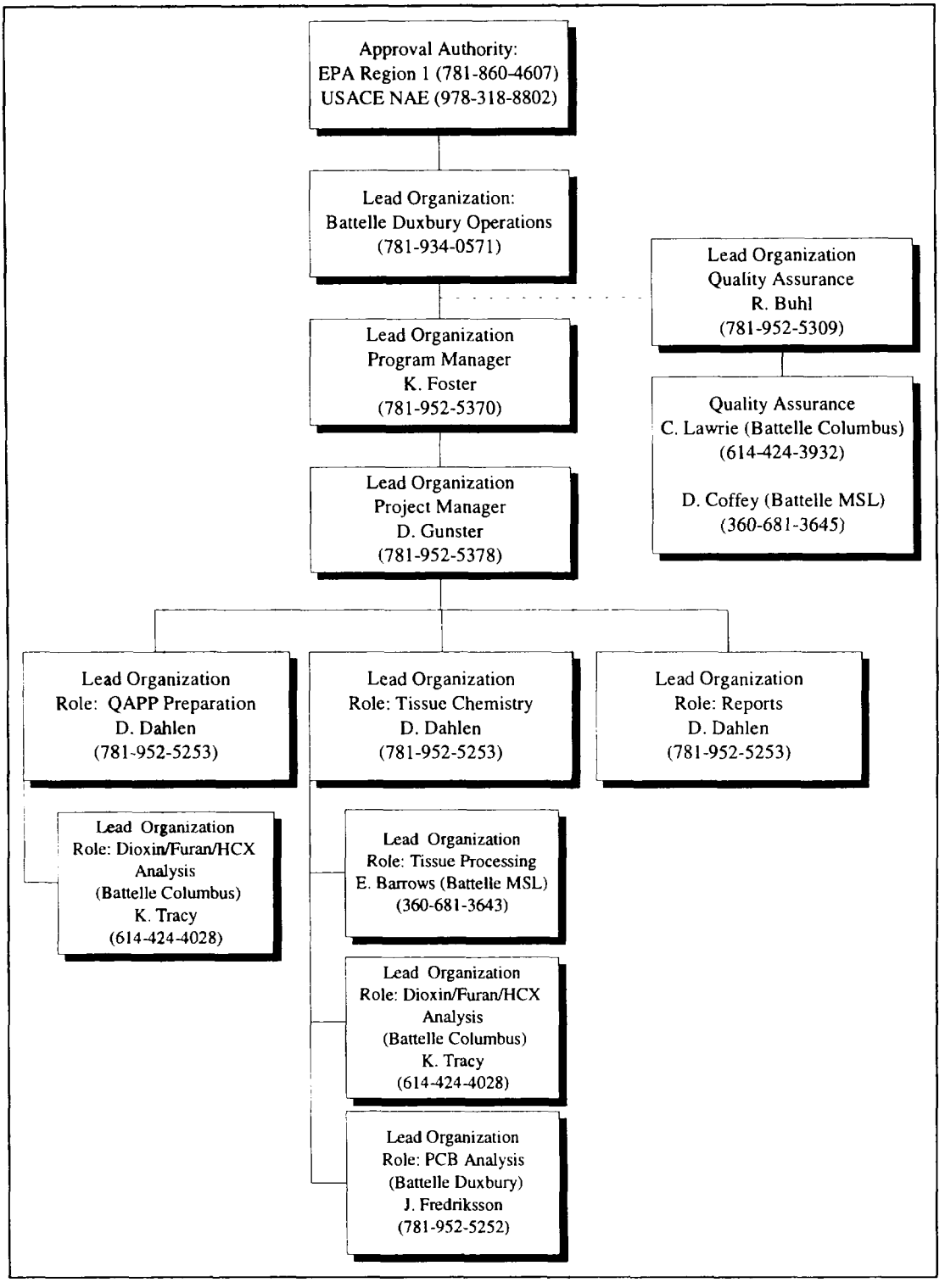


Figure 1. Project Organizational Chart.

EPA-NE QAPP Worksheet #5b - Rev. 10/99

Communication Pathways

Communication pathways will follow the project organization chart (Figure 1). Mr. Laureen Borochaner is the USACE NAE Project Manager. Mr. Don Gunster is Battelle's Project Manager and is responsible for the technical oversight, overall quality and conduct of the project. He will be the primary contact with the USACE NAE Project Manager. Mr. Gunster will ensure that the objectives of the project are met within budget and on schedule.

Ms. Rosanna Buhl will serve as Battelle's Quality Assurance (QA) Officer, and is responsible for identifying areas for corrective action, coordinating the QA activities such as systems and data audits, and preparing reports to management for this project. She will be assisted by the QA Officers at each of the participating laboratories.

As indicated in Figure 1, Task Leaders have been assigned for each of the major project tasks (*e.g.*, QAPP Preparation). The Task Leaders will serve as the point of contact and will direct task activities and monitor task performance to ensure adherence to technical standards, budget, and schedule. They also will be responsible for apprising Mr. Gunster of progress and notifying him of any significant problems or delays. For example, the point of contact for resolving issues with Dioxin/Furan/HCX analyses will be Ms. Karen Tracy.

The need for corrective action may be identified during analysis, during QA reviews, or during management reviews. EPA Worksheets #21 and #24a define the corrective action(s) options for quality control data and calibration exceedences. Corrective action implemented in response to QA audits is documented as part of the analyst's response to the audit. Battelle SOP 4-035 describes Battelle Duxbury's formal Corrective Action program. All internal corrective action is followed up by the QA Officer. Corrective action related to changes in scope, analytical techniques, or financial variances are formally communicated to Ms. Borochaner by Mr. Gunster.

All communications will be conducted using electronic mail, phone, telefaxes, and/or reports.

EPA-NE QAPP Worksheet #6 - Rev. 10/99

Personnel Responsibilities and Qualifications Table

Name	Organizational Affiliation	Responsibilities	Location of Personnel Resumes, if not included	Education and Experience Qualifications
Don Gunster	Battelle Duxbury	Responsible for the technical oversight, overall quality and conduct of the project for lead organization		See attached (Attachment B)
Rosanna Buhl	Battelle Duxbury	Oversee QA/QC activities performed for lead organization		See attached (Attachment B)
Mark Guilmain	Battelle Duxbury	Performs data audits		See attached (Attachment B)
Deirdre Dahlen	Battelle Duxbury	Responsible for preparing QAPP and overseeing technical conduct of tissue chemistry analyses.		See attached (Attachment B)
Robert Lizotte	Battelle Duxbury	Manager of Organics Laboratory		See attached (Attachment B)
Micheal Meara	Battelle Duxbury	Responsible for laboratory custody of samples	Not available[1]	Whitman Hanson Regional High School, 1995. Cape Cod Community College (one semester); 2-yrs experience as Sample Custodian, Battelle Duxubry.
Beth Kitson	Battelle Duxbury	Prepare tissue samples for PCB analyses		See attached (Attachment B)
Julie Fredriksson	Battelle Duxbury	Analyze tissue samples for 107 PCB congeners; prepare and validate final tables and submit data package(s) to QA for data audit		See attached (Attachment B)
Betsy Barrows	Battelle Marine Sciences Laboratory (MSL)	Oversee freeze-drying of nestling samples and determine moisture content.		See attached (Attachment B)
Deborah Coffey	Battelle MSL	Oversee project QA/QC activities associated with freeze-drying tissue samples; audit data		See attached (Attachment B)
Carolynn Suslick	Battelle MSL	Responsible for laboratory custody of tissue samples and reporting moisture content data		See attached (Attachment B)

[1] Resume not available. Education summarized in next column.

EPA-NE QAPP Worksheet #6 - Rev. 10/99 (continued)

Personnel Responsibilities and Qualifications Table

Name	Organizational Affiliation	Responsibilities	Location of Personnel Resumes, if not included	Education and Experience Qualifications
Karen Tracy	Battelle Columbus	Responsible for assisting with QAPP preparation; overseeing technical conduct of dioxin/furan/HCX tissue chemistry analyses: and prepare and validate final tables and submit data package(s) to QA for data audit		See attached (Attachment B)
Charles D. Lawrie	Battelle Columbus	Oversee QA/QC activities performed for Battelle Columbus; audit data		See attached (Attachment B)
Mary E. Schrock	Battelle Columbus	Dioxin Laboratory Manager		See attached (Attachment B)
Mark F. Misita	Battelle Columbus	Responsible for preparation laboratory and custody of samples		See attached (Attachment B)
Henry H. Pham	Battelle Columbus	Prepare tissue samples for dioxin/furan/HCX analyses		See attached (Attachment B)
Wesley H. Baxter	Battelle Columbus	Prepare tissue samples for dioxin/furan/HCX analyses	Not available[1]	Minerva High School (Middleburg Hgts., OH), 1995; Ohio State University – Spring 2001 expected graduation with B.A. Chemistry; 2 ½ months experience with sample preparation for Dioxin/Furan, PCBs and Pesticides by GC/HRMS, Battelle Columbus.
Joseph E. Tabor	Battelle Columbus	Analyze tissue samples for dioxin/furan/HCX by HRGC/HRMS		See attached (Attachment B)

[1] Resume not available. Education summarized in next column.

EPA-NE QAPP Worksheet #8a - Rev. 10/99

Project Planning Meetings

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Centredale Manor
Program (Brownfields, NPDES, etc.): Superfund	**Site Location**: North Providence, Rhode Island
Project Date(s) of Sampling: May and June, 2000	**CERCLA Site/Spill Identifier No.**: 016P
Project Manager: Don Gunster	**Operable Unit**:
	Other Site Number/Code: 016P
	Phase: ERA SA/SI Pre-RI RI (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:

Date of Meeting: July 17, 2000
Meeting Location: EPA Region 1 Offices in Boston, MA

Name	Project Role	Affiliation	Phone #	e-Mail Address
Anna Krasko	Remedial Project Manager	EPA Region 1	(617) 918-1232	Krasko.Anna@epa.gov
Cornell Rosiu	Work Assignment Manager	EPA Region 1	(617) 918-1345	Rosiu.Cornell@epa.gov
Richard Willey	Hydrologist	EPA Region 1	(617) 918-1266	Willey.Dick@epa.gov
Sarah Levinson	Human Health Risk Assessor	EPA Region 1	(617) 918-1390	Levinson.Sarah@epa.gov
Andy Beliveau	Quality Assurance Officer	EPA Region 1	(781) 860-4607	Beliveau.Andy@epa.gov
Laureen Borochaner	Project Manager	USACE NAE	(978) 318-8802	Laureen.A.Borochaner@usace.army.mil
Mike Penko	Environmental Scientist	USACE NAE	(978) 318-8139	John.M.Penko@usace.army.mil
Karen Foster	Program Manager	Battelle Duxbury	(781) 952-5370	foster@battelle.org
Don Gunster	Project Manager/ Risk Assessor	Battelle Duxbury	(781) 952-5378	gunster@battelle.org
Norm Richardson	Human Health Risk Assessor	Harding Lawson Associates	(781) 245-6606 x1033	nrichard@harding.com
Sarah Shah	Project Manager	Harding Lawson Associates	(781) 245-6606 x1037	sshah@harding.com

EPA-NE QAPP Worksheet #8a - Rev. 10/99 (continued)

Project Planning Meetings

Meeting Purpose. Key discussion points included

1) What are the project and schedule drivers?
2) What are the ultimate long-term goals for the study area (e.g. residential/commercial redevelopment)
3) May we have copies of all reports and data from previous phases of work at the Meeting on Monday July 17, 2000?
4) Which phases of work have already been performed on site? Which phases are currently ongoing? Which phases have been planed or anticipated for the future? How does the BRA work coordinate with other phases of work?
5) Will coordination with other contractors be required to share data from concurrent phases of work? If so, what level of interaction is anticipated between these teams? How would this interaction be arranged?
6) Should we standardize our data management systems with other contractors to provide consistency across the entire project?
7) What electronic deliverables will be required?
8) Should all plans be submitted together in one SMP document or separately as five individual plans?
9) Are deliverables associated with Tasks 5.1 - 5.4?
10) Will a mobile laboratory be required?
11) Will a laboratory audit be required for Battelle's laboratory or for a third party laboratory? If so, who will perform the audit?
12) Page 9, Section 2.2.1 - Will a site survey or the use of a mobile GPS unit be required to document sample locations?
13) Page 11, Section 2.2.8 - Are formal surveys required or are site observations sufficient?
14) Task 3 - How and where should screening tools/techniques be used?
15) Will waste storage and decontamination facilities be required?
16) Will HLA be responsible for disposal of all IDW and will the USACE agree to sign disposal manifests?
17) Should USACE reviews of all documents be performed prior to or concurrently with the EPA reviews?
18) Are formal responses to document review comments required? If so should those formal responses be included as part of the documents.
19) Are USAC and EPA monthly status report formats/contents consistent so only report will satisfy the needs of both agencies?
20) Schedule

Comments. The above discussion points, and the resolutions to these points, were summarized in meeting minutes that were submitted to NAE on 7/19/00. The meeting minutes were revised in response to NAE review comments and submitted as final to NAE and EPA on 7/28/00. Meeting minutes are not included in this QAPP – as the discussion points did not pertain to the tissue chemistry tasks described in this QAPP.

EPA-NE QAPP Worksheet #8b - Rev. 10/99

Problem Definition/Site History and Background

Site History and Background[1]

The Centredale Manor Site (Figure 2) is a multi-unit apartment complex that houses elderly and handicapped adults. It is located at 2074 Smith Street (Route 44) in Centredale, a village of North Providence, Rhode Island. The Centredale Manor apartment building and adjacent apartment building known as "Brook Village", are located on the site of the former Metro-Atlantic Chemical Corporation, which operated from the 1940s to the 1970s in a former mill complex on the site. The Woonasquatucket River follows the west boundary of the site. The remains of a raceway for the former mill complex are present on the eastern boundary of the site.

Historical records of Metro Atlantic Chemical researched by Weston (March 1999) indicate that the site manufactured hexachlorophene and that there were shipments of trichlorophenols to the site. The mill complex was destroyed by fire in the late 1970's and the apartment buildings were constructed in 1982. During construction of the apartment buildings 400 drums and 6,000 cubic yards of contaminated soil were removed from the site. Labels indicated that the drums contained caustics, halogenated solvents, PCBs, and inks.

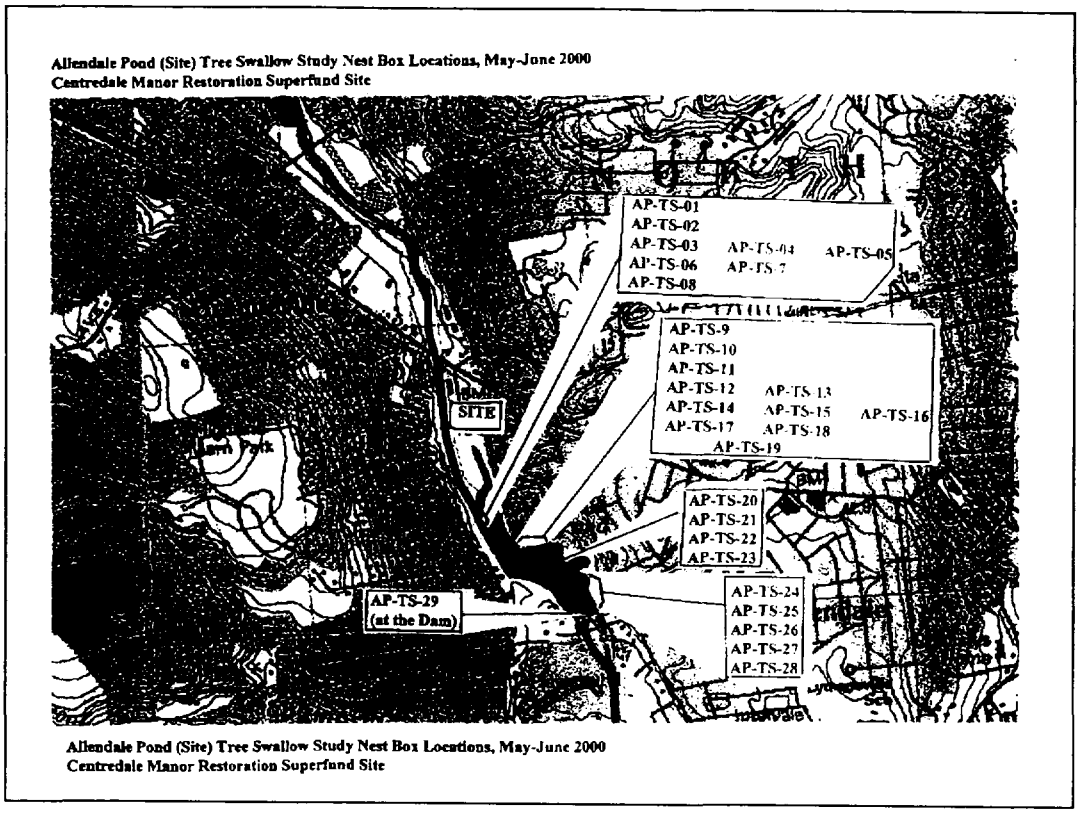
A study conducted in June 1996 by the EPA Narragansett Laboratories and the Providence Urban Initiative Program (EPA, 1996) determined that elevated levels of dioxin were present in fish collected from the River. A subsequent study of the Woonasquatucket River conducted by the USEPA OEME in June 1998 found elevated concentrations of dioxin and polychlorinated biphenyls (PCBs) in sediments in portions of the river and impoundments adjacent to the downstream of Centredale Manor (EPA, July 1998). Soil and sediment sampling conducted by EPA START personnel in September 1998 found dioxin at concentrations up to 10.1 ppb in sediments collected directly behind the Allendale dam that had a water depth of at least six feet (Weston, March 1999). Allendale Pond was an impoundment located immediately downstream of the Centredale Manor Site (Figure 2). The impoundments dam breached in 1991 exposing the sediments. Further sampling conducted in February 1999 on the Centredale Manor property also found elevated concentrations of dioxin in soils and sediment. Additional historical information on the Centredale Manor Site is available in the Expanded Site Inspection Report, prepared by Weston (March, 1999).

Contaminants of concern include dioxin, 1,2,4,5,7,8-hexachloroxanthene (HCX), and PCBs.

Objectives

The purpose of this study is to conduct chemical analyses on 33 biological tissue (20 egg, 10 nestling, and 3 diet) samples collected by EPA-NE in May and June, 2000. Samples were collected at three sampling locations (*i.e.*, Greystone Pond, Allendale Pond, and Woonasquatucket River) that may have been impacted by contaminants released from the former Metro-Atlantic Chemical property and transported and deposited by the waters of the Woonasquatucket River. Biological tissue samples will be analyzed for dioxins/furans, HCX, and PCBs and the data will be used to perform ecological risk assessments.

[1] Site history and background taken verbatim from the *Sampling and Analysis Plan Woonasquatucket River Sediment Investigation, Centredale Manor Site North Providence, Rhode Island* (Tetra Tech, 1999). Note – references cited in Tetra Tech SAP not available.



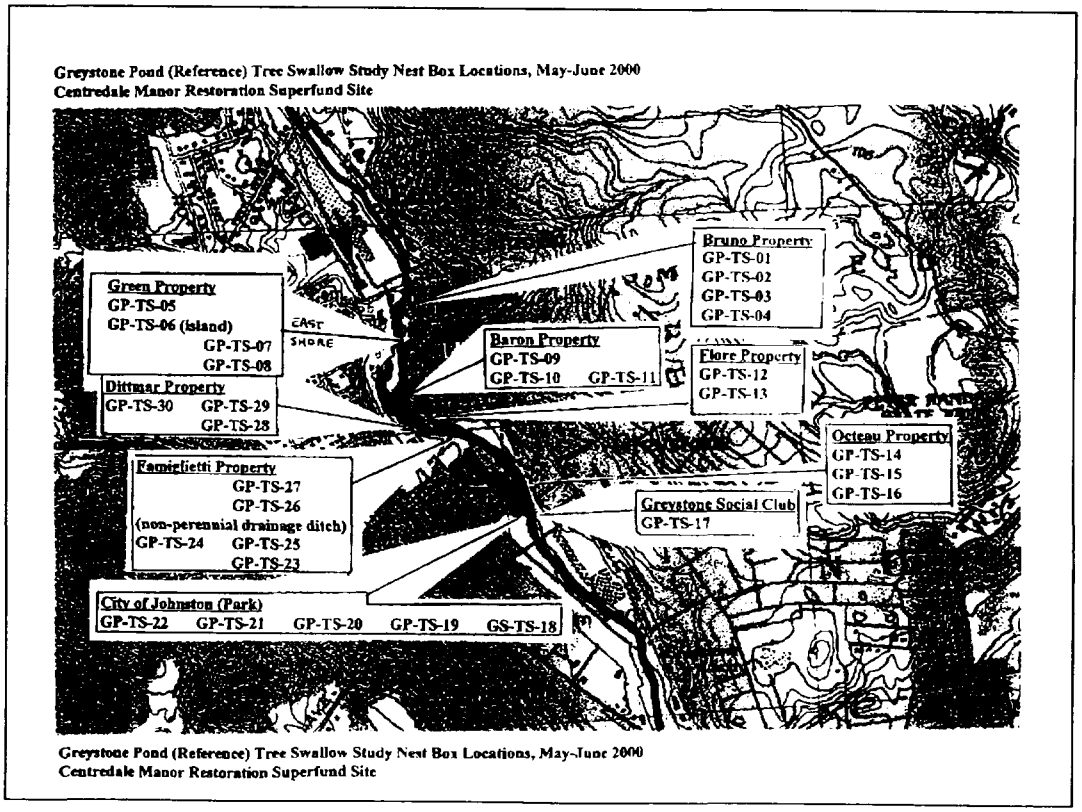


Figure 2. Site Maps.

EPA-NE QAPP Worksheet #9a - Rev. 10/99
Project Description and Schedule

Sampling Tasks:
Field sampling was conducted by EPA-NE in May and June, 2000 and followed methods outlined in Custer *et al.*, (2000).

Analysis Tasks:
Tissue samples will be analyzed for Dioxins/Furans, HCX, and PCB congeners. Definitive data will be produced for each analytical task. All analytical tasks will be performed in a fixed laboratory. General descriptions of analytical methods are described below.

Tissue Processing—Nestling samples will be freeze-dried at Battelle MSL and the freeze-dried samples shipped to Battelle Duxbury for homogenization by mortar and pestle. The homogenized sample will be split for chemical analysis and each sub-sample placed in pre-cleaned glass jars (with Teflon-lined hard cap). The sub-sample for Dioxin/Furan/HCX analysis will be shipped (frozen) by Federal Express (next business morning delivery) to Battelle Columbus. PCB congener analysis will be conducted at Battelle Duxbury.

Percent moisture will be determined for the nestling samples only, following Battelle MSL SOP MSL-C-003 (Attachment E). No other percent moisture determinations will be performed. Egg and diet samples will be consumed for analysis (*i.e.*, extracted whole) and as a result moisture content will not be determined. ***Note*** – chemistry results will only be reported on a wet weight basis so the percent moisture content is not a required parameter.

Dioxin/Furan/HCX—Tissue samples will be extracted and analyzed for the seventeen 2,3,7,8-substituted PCDD/PCDF and HCX following the general procedures in EPA Method 1613, Revision B, as described in Battelle Columbus Operations SOPs ASAT.II-001-01 and ASAT.II-002-01.

Approximately 1- 10 g (wet weight) of each tissue will be spiked with isotopically labeled analogs of fifteen of the seventeen 2,3,7,8-substituted PCDD/PCDF, and extracted with methylene chloride: hexane (1:1) in a Soxhlet apparatus for a minimum of sixteen hours. The extracts will be spiked with $^{37}Cl_4$-2,3,7,8-TCDD cleanup standard, partitioned against acid solutions, and processed through acid/base silica, alumina, and carbon Celite columns. Extracts will be spiked with $^{13}C_{12}$-1,2,3,4-TCDD/$^{13}C_{12}$-1,2,3,7,8,9-HxCDD recovery standard and concentrated to a final volume of 20 µL.

Sample extracts will be analyzed by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) in the selected ion monitoring mode (SIM) at a resolution of approximately 10,000. Initial analysis will be on a DB-5 or equivalent column. Because 2,3,,7,8-TCDF is not completely separated from all of the other TCDF isomers on the DB-5 column, second column confirmation of 2,3,7,8-TCDF levels above the lowest calibration level in the initial analysis will be carried out on a DB-Dioxin or DB-225 column. All analytes will be quantified by isotope dilution or by the method of internal standards using surrogate compounds.

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued)

Project Description and Schedule

Analysis Tasks (continued):

Dioxin/Furan/HCX cont.

HCX will be determined following guidance of the work by Jeffery Archer and Terry Crone [2]: The M+4 and M+6 ion masses used by Archer and Crone for HCX (389.8156 and 391.8127 are already included in the hexa descriptor for dioxin/furan analysis. According to the paper cited above, HCX elutes within one minute of $^{13}C_{12}$-1,2,3,7,8,9-HxCDF using conditions typical for dioxin/furan analysis. For that reason, the acquisition window for the hexa isomer groups will be extended to the beginning of the hepta isomer acquisition window. Due to the lack of commercially available HCX standard, HCX will be identified and quantitated in the following way:

- Peaks at ion mass 389.8156 and 391.8127 need to co-elute within two seconds.
- Ratio of peak areas for ion masses M+4/M+6 must be 2.31 ± 15%.
- The signal to noise ratio of peaks at the M+4 and M+6 ion masses must be > 2.5.
- HCX will be quantified against $^{13}C_{12}$-1,2,3,7,8,9-HxCDF using an assumed response factor of one.

PCB Congener Analysis—Tissues will be extracted and cleaned following procedures in Battelle SOP 5-190. Approximately 2-g of freeze-dried, tissue homogenate will be weighed into a Teflon extraction jar, spiked with the appropriate SIS, combined with 75 mL DCM and sodium sulfate, macerated with a Tissumizer and centrifuged. The extract will be decanted into a Erlenmeyer flask. This process is repeated once using 75 mL DCM. After each maceration (total of two solvent additions) the centrifuged solvent extracts will be combined in the Erlenmeyer flask. An additional extraction will be performed using 50 mL DCM and shaking techniques, the sample centrifuged a third time, and the extract combined with the other two. The combined extract will be dried over sodium sulfate, filtered (if necessary), and concentrated by Kuderna-Danish (KD) technique to approximately 25 mL. The extract will be transferred to a graduated cylinder, the volume adjusted to 25-mL exactly, and a 1-mL aliquot (measured exactly with a syringe) removed for "methylene chloride extractable" lipid weight determination. The remaining extract will be concentrated to 2 to 3 mLs and processed through an alumina cleanup column:

- Packing: 40 g F20 (2% deactivated) alumina, in DCM
- Elution: 150 mL DCM

After alumina column cleanup, all sample extracts will be concentrated by KD and nitrogen blow down techniques to approximately 900-µL for additional HPLC cleanup (Battelle SOP 5-191, PCB collection window). The post-HPLC extract will be solvent exchanged into hexane, concentrated under nitrogen to approximately 200-µL, and spiked with RIS. The extract will be transferred to the GC/MS Task Leader for PCB analysis.

Optional acid treatment may be performed if matrix interferences make the PCB analysis problematic.

Lipid weights will be performed following procedures described in Battelle SOP 5-190. Lipid content will be measured as extractable lipid.

[2] J. Archer and T.Crone, "Hexachloroxanthene Analysis with TCDD," Proceedings from the 20th International Symposium on Halogenated Environmental Organic Pollutants and POP, Monterey, CA; Volume 45; pp (29-32).

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued)

Project Description and Schedule

Quality Control Tasks:
A routine set of quality control samples will accompany every set of samples processed and analyzed for this project. The type and frequency of quality control samples are defined in EPA-NE QAPP Worksheet #24a, but generally include:

- one procedural/method blank (PB)
- one laboratory control sample (LCS)
- one matrix spike/matrix spike duplicate set (MS/MSD)
- one standard reference material (SRM)
- multiple surrogate internal standards (SIS) per sample
- one independent control sample (internal QA only)
- one sample duplicate (DUP)

Secondary Data:
Not applicable.

Data Management Tasks:
The dioxin data generated by CG/HRMS will be acquired on a Alpha station personal work station 600AU using VG OPUS and OPUSquan software. GC/MS data will be acquired and reduced on Hewlett-Packard PC based chemstation minicomputers with dedicated chromatography software (EnviroQuant). All GC/HRMS and GC/MS data files will be transferred electronically to a PC so that the data can be incorporated into an electronic database or spreadsheets for final quantification and tabular result presentation.

The appropriate analyst/data manager assigned to the project team will perform all data reduction. The final reduction of analytical chemistry data will account for the size of the processed sample and dilution factors. Data provided by participating laboratories will be requested in electronic format (Excel).

Chemistry reports will include:

- Sample Identification
- Moisture and lipid content (nestling samples only)
- Concentrations of the seventeen 2,3,7,8-PCDD/PCDF and HCX in wet-weight (pg/g); Concentrations of the 107 PCB congeners, total PCB by chlorination level, and total PCB (sum of the detected 107 PCB congeners) in wet-weight (ng/g). Sample-specific detection limits will also be reported. Results reported to two decimal places.
- PB results – reported on a concentration basis to two decimal places.
- Recovery of SISs added to each sample – reported as whole numbers.
- Amounts expected and recovered, and percent recoveries, for MS, MSD, and LCS samples. Concentration data reported to two decimal places and recovery data reported as whole numbers.
- The relative percent difference (RPD) between the MS and MSD results – reported to one decimal place.

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued)

Project Description and Schedule

Data Management Tasks (continued):

- The average and relative percent difference of the duplicate sample analysis – RPD reported to one decimal place.
- Results of SRM analyses, certified values, and the percent difference (PD) between the results and the certified values. All PD results will be reported to one decimal place. *Note* – for PCB analysis the PD is determined from the range of certified values (see EPA-NE QAPP Worksheet #24a, Attachment A)

Chemistry reports will also include a QA/QC narrative that define the QC criteria that were to be met along with results that were achieved. QA/AC narratives are further described in EPA-NE QAPP Worksheet #9a, Data Usability Assessment Tasks.

The following header information should be used, listed in this order on the final report tables:

Data Report Header Format

Header Information	Comments
Client Sample ID	
Battelle Sample ID	
Sample Type	Nestling, Egg, or Diet
Batch ID	
Moisture Content	Nestling samples only
Lipid Content (% wet)	Nestling samples only
Sample Wet Weight (g)	
Reporting Units	Dioxins/Furans/HCX: pg/g wet wt.; PCB: ng/g wet wt.

Documentation and Records:

Documentation associated with laboratory analyses will include sample receipt and log-in records, sample processing logs, sample preparation records, analytical instrument printouts, and equipment logs. Initially, all data will be recorded either (1) electronically onto a computer storage media from laboratory systems or (2) manually into laboratory notebooks or on established data forms. All notes will be written in ink. Corrections to hand-entered data will be initialed, dated and justified. Complete forms, laboratory notebooks, or other forms of hand-entered data will be signed and dated by the individual entering the data. It will be the responsibility of the laboratory manager and/or task leader to ensure that all data entries and hand calculations are verified. Laboratory records of sample preparation will be maintained in bound laboratory record books. In addition to these documentation procedures, sample logs associated with field and laboratory custody and tracking will be maintained in project files.

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued)

Project Description and Schedule

Data Packages:
Analytical Task Leaders will prepare a project-specific data package (project records), which will be submitted to the Quality Assurance Unit for an independent Quality Assurance review.

Data packages are considered a deliverable and will be maintained by the laboratory. Content and format of data packages prepared at Battelle Duxbury are defined below. ***Note*** – while the format of data packages prepared by participating laboratories may vary, the content will be consistent and will contain pertinent raw data elements (see Attachment D) necessary for potential third party validation. The format of data packages prepared at Battelle Duxbury is as follows.

Content of Data Packages Prepared at Battelle Duxbury

Section	Contents
Title Page	Project name; analysis parameter; batch ID; matrix; approvals
Signature Page	Name (printed, signature, and initials) of laboratory staff
Workplan	Hardcopy of laboratory QAPP and project-specific memorandums
Tables	Preliminary QC Checklist (summary of QC results evaluated against measurement performance criteria); Final hardcopy of Excel report tables
Misc. Docs.	Documentation of project-specific issues (corrective action, changes in scope, etc...) Data Validation Checklist – see Attachment D
Sample Prep	Hardcopy of sample preparation records
Calibrations	Initial and continuing calibration reports
Sample Data	Quantification reports for authentic and QC samples
Chromatograms	Sample chromatograms
Unused Data	File copy of data not used or reported

One copy of each data package, including raw data, will be submitted to USACE NAE for third party validation purposes. Raw data are defined in Attachment D.

Assessment/Audit Tasks:
Quality assurance encompasses all planned and systematic activities necessary to assure management that the products generated, and the services performed by Battelle meet the quality standards established in this QAPP. The primary mechanism for accomplishing this goal is audits. Audits refer to the formal assessment of conformance to the QA Program and its effectiveness. During an audit, the agreement with QA policy documents (*e.g.*, SOPs) is evaluated, deficiencies are identified, and corrective action is taken. Ideally, audits also serve to increase awareness and understanding of QA policies and procedures. Ms. Rosanna Buhl will serve as Battelle's QA Officer and is responsible for identifying areas for corrective action, coordinating the QA activities such as systems and data audits, and preparing reports to

EPA-NE QAPP Worksheet #9a – Rev. 10/99 (continued)
Project Description and Schedule

Assessment/Audit Tasks (continued):
management for this project. QA Officers at participating laboratories will be responsible for coordinating and performing QA activities at participating laboratories. The identity of auditors and their qualification are presented in EPA-NE QAPP Worksheet #6. The following QA audits are planned for this project.

- A technical system (initiation) audit is conducted as part of the review of this QAPP to (1) ensure that the work assignment scope and all required elements are addressed adequately, (2) verify that all required SOPs are approved and current, and (3) to verify that all participants have the required qualifications and documented training to perform their assigned tasks.
- Performance audits are independent checks of routinely obtained data. One Certified or Standard Reference Material (CRM or SRM, respectively) will be incorporated into each batch of tissue chemistry samples (as applicable) to assess the accuracy and precision with which target analytes of known concentration are recovered from a representative matrix. The acceptance criteria are discussed in EPA-NE QAPP Worksheet #11b.

Data audits will be conducted for all reported data. These audits will reconstruct representative data from each sample based on sample processing records, instrument calibration factors (*e.g.*, response factors) and output (*e.g.*, area counts), and sample manipulations and spiking. Samples will be tracked from receipt and processing through analysis and reporting to ensure that the reported data are accurate, complete, and traceable. Section 17 discusses the reporting of audit results to management and Section16.2 describes corrective action procedures resulting from audit findings. A QA Statement submitted to the Project Manager with each deliverable describes the audit and review activities conducted to assess the deliverable accuracy, and any outstanding issues that could impact data quality. Results of QA audits will be reported to the Task Leader and Project Manager. The audit reports will define any errors, deficiencies, or deviations from the QAPP. The responsible analyst documents the corrective action on the audit report and submits the audit report to the Project Manager for review and approval. Battelle audit reports are available for review at Battelle Duxbury.

The data reports submitted to USACE NAE will be reviewed by the appropriate Task Leaders, the Project Manager, and the Quality Assurance Unit.

Data Verification and Validation Tasks:
Data validation is the responsibility of those immediately responsible for overseeing and/or performing analyses, data entry, data reduction, and data reporting. The data validator will validate final report tables for accuracy and completeness (*i.e.*, calculation, manual entries). Battelle SOP 6-027 describes data validation procedures performed at Battelle Duxbury, whereas data review activities conducted at Battelle Columbus are described in SOPs ASAT.II-003 and ASAT.II-010.

A series of reviews by technical personnel will be implemented to ensure that the data generated for this work assignment meet the data quality objectives. These reviews will include the following activities.

- Data and related project records will be reviewed by laboratory personnel at the end of each working day to ensure that analytical activities are completely and adequately documented.

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued

Project Description and Schedule

Data Verification and Validation Tasks (continued):

- The Task Leaders will be responsible for reviewing analytical results and supporting documentation. The results of QC sample analyses will be compared to pre-established criteria as a measure of data acceptability.
- All hand-entered or transcribed data will be 100% validated.
- All calculations performed manually will be checked for accuracy. Calculations performed by software will be checked at a frequency sufficient to verify their accuracy.

All data will be validated to ensure that the measurement performance criteria (MPCs) described in EPA-NE QAPP Worksheet #11b have been met, instrument calibration and maintenance requirements also specified in EPA-NE QAPP Worksheet #21 have been met, and that the data are complete, accurate, and traceable.

All data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed at the Project Manager's discretion. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (below), and the rationale for accepting the analysis will be thoroughly documented.

PCB and dioxin/furan/HCX data will receive validation by a third party. Third party validation is not the responsibility of Battelle.

Data Reporting Qualifiers

Data Qualifier	Definition
J	Detected, but below the QL[1] (for PCBs) or EDL[1] (for Dioxins/Furans/HCX).
E	Estimate; significant matrix interference.
B	Analyte detected at > QL and concentration in associated samples <10× blank levels.
U	Not detected; sample-specific QL (for PCBs) or EDL (for Dioxins/Furans/HCX) reported.
&	QC value outside the accuracy or precision criteria goal (EPA-NE QAPP Worksheet #11b).
~	QC value outside the accuracy or precision criteria goal (EPA-NE QAPP Worksheet #11b) – but meets contingency criteria.
#	Result from second column confirmation analysis

QL = Quantitation Limit; EDL = Estimated Detection Limit
[1]Analyte signal sufficient to confidently identify and quantify analyte (signal-to-noise ratio of approximately 5:1), but concentration is below the reported QL or EDL. QLs or EDLs will be reported with the data.

EPA-NE QAPP Worksheet #9a - Rev. 10/99 (continued

Project Description and Schedule

Data Usability Assessment Tasks:
The review of quality control data is a critical step in the data validation process because quality control data that are within the QAPP acceptance criteria indicate that the sample processing and analysis systems are in control. EPA-NE QAPP Worksheet #24a describes the type of quality control samples that will be analyzed with each analytical batch and corrective action for out-of-control quality control data and instrumentation calibrations. All quality control data that do not meet the data quality objectives will be flagged (see above table of data qualifiers) and brought to the attention of the Task Leader and the Project Manager, who will determine the appropriate corrective action (*e.g.*, reanalysis or data reported with qualifiers).

QA/QC narratives will present quality control criteria and the quality control results. They will be prepared for each analytical batch and will describe any MPC exceedances and what, if any, impact they may have on the overall data.

EPA-NE QAPP Worksheet #9b - Rev. 10/99

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: Dioxin/Furan/HCX
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP[1]: L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (WET WT)	Project Quant Limit (Units) (WET WT)	Analytical Method		Achievable Laboratory Limits (WET WT)	
				MDLs[2]	Method QLs[2]	MDLs[3]	QLs[3]
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	Not provided in SOW	Not provided in SOW	Not provided	1	0.5	0.5
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	As above	As above	As above	1	0.4	0.5
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	As above	As above	As above	5	1.7	2.5
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	As above	As above	As above	5	1.2	2.5
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	As above	As above	As above	5	0.9	2.5
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	As above	As above	As above	5	3.4	2.5
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	As above	As above	As above	5	3.6	2.5
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	As above	As above	As above	5	2.2	2.5
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	As above	As above	As above	5	1.7	2.5
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	As above	As above	As above	5	1.7	2.5
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	As above	As above	As above	5	1.1	2.5
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	As above	As above	As above	5	1.4	2.5
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	As above	As above	As above	5	14.4	2.5
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	As above	As above	As above	5	1.6	2.5
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-89-7	As above	As above	As above	5	2.9	2.5
Octachlorodibenzo-p-dioxin	3268-87-9	As above	As above	As above	10	4.2	5
Octachlorodibenzofuran	39001-02-0	As above	As above	As above	10	13.1	5
1,2,4,5,7,8-Hexachloro-9-Xanthene		As above	As above	As above	NA	NA	NA

[1] Specify appropriate reference number/letter from the Field and Fixed Laboratory Analytical Method/SOP Reference Tables (EPA-NE QAPP Worksheets #17 and #20).
[2] Analytical method MDLs and QLs documented in validated methods. These limits are based on a sample size of 10 g. QLs are usually 3-10 times higher than the MDLs.
[3] Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. These limits are based on a sample size of 10g. The MDL values are from a seven replicate MDL study and the QL values are based on the lowest calibration standard, sample size (10g), and extract volume 20µL).

EPA-NE QAPP Worksheet #9b - Rev. 10/99

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte[a]	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB1	2051-60-7	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB3	2051-62-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB4/10	13029-08-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB6	25569-80-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB7/9	33284-50-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB8/5[b]	34883-43-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB12/13	2974-92-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB16/32	38444-78-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB17	37680-66-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB18	37680-65-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB19	38444-73-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB21	55702-46-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB22	38444-85-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB24/27	55702-45-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB25	55712-37-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB26	38444-81-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB28	7012-37-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB29	158-07-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB31	16606-02-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB33/20	38444-86-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB40	38444-93-8	As above	As above	As above	As above	As above	0.4 ng/g

EPA-NE QAPP Worksheet #9b - Rev. 10/99 (continued)

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB41/64/71	52663-59-9	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB42	36559-22-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB43	70362-46-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB44	41464-39-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB45	70362-45-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB46	41464-47-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB47/75	2437-79-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB48	70362-47-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB49	41464-40-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB51	68194-04-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB52	35693-99-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB53	41464-41-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB56/60	41464-43-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB59	74472-33-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB63	74472-34-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB66	32598-10-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB70/76	32598-11-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB74	32690-93-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB82	52663-62-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB83	60145-20-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB84	52663-60-2	As above	As above	As above	As above	As above	0.4 ng/g

EPA-NE QAPP Worksheet #9b - Rev. 10/99 (continued)

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB85	65510-45-4	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB87/115	38380-02-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB89	73575-57-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB91	68194-05-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB92	52663-61-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB95	38379-99-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB97	41464-51-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB99	38380-01-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB100	39485-83-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB101/90	37680-73-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB105	32598-14-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB107	70424-68-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB110	38380-03-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB114	74472-37-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB118	31508-00-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB119	56558-17-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB124	70424-70-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB128	38380-07-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB129	55215-18-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB130	52663-66-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB131	61798-70-7	As above	As above	As above	As above	As above	0.4 ng/g

EPA-NE QAPP Worksheet #9b - Rev. 10/99 (continued)

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB132	38380-05-1	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB134	52704-70-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB135/144	52744-13-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB136	38411-22-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB137	35694-06-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB138/160/163	35065-28-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB141	52712-04-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB146	51908-16-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB149	38380-04-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB151	52663-63-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB153	35065-27-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB156	38380-08-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB158	74472-42-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB167	52663-72-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB169	32774-16-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB170/190	35065-30-6	As above	As above	As above	As above	As above	0.4 ng/g
PCB171	52663-71-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB172	52663-74-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB173	68194-16-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB174	38411-25-5	As above	As above	As above	As above	As above	0.4 ng/g
PCB175	40186-70-7	As above	As above	As above	As above	As above	0.4 ng/g

EPA-NE QAPP Worksheet #9b - Rev. 10/99 (continued)

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB176	52663-65-7	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB177	52663-70-4	As above	As above	As above	As above	As above	0.4 ng/g
PCB178	52663-67-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB180	35065-29-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB183	52663-69-1	As above	As above	As above	As above	As above	0.4 ng/g
PCB184	74472-48-3	As above	As above	As above	As above	As above	0.4 ng/g
PCB185	52712-05-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB187/182	52663-68-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB189	39635-31-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB191	74472-50-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB193	69782-91-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB194	35694-08-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB195	52663-78-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB197	33091-17-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB198	68194-17-2	As above	As above	As above	As above	As above	0.4 ng/g
PCB199	52663-75-9	As above	As above	As above	As above	As above	0.4 ng/g
PCB200	52663-73-7	As above	As above	As above	As above	As above	0.4 ng/g
PCB201	40186-71-8	As above	As above	As above	As above	As above	0.4 ng/g
PCB203/196	52663-76-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB205	74472-53-0	As above	As above	As above	As above	As above	0.4 ng/g
PCB206	40186-72-9	As above	As above	As above	As above	As above	0.4 ng/g

EPA-NE QAPP Worksheet #9b - Rev. 10/99 (continued)

Medium/Matrix: Tissue
Region I Matrix Code (from EPA-NE DQO Summary Form): BD
Analytical Parameter: PCBs
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

Analyte	CAS Number	Project Action Limit (Units) (wet weight)	Project Quantitation Limit (Units) (wet weight)	Analytical Method[1]		Achievable Laboratory Limits (WET WT)	
				MDLs	Method QLs	MDLs[2]	QLs[3]
PCB207	52663-79-3	Not provided in SOW	Not provided in SOW	Not applicable	Not applicable	Not available	0.4 ng/g
PCB209	2051-24-3	As above	As above	As above	As above	As above	0.4 ng/g
Total PCB by Chlorination Level [c]	NA	As above	As above	As above	As above	As above	—
Total PCB [d]	1336-36-3	As above	As above	As above	As above	As above	—

[1] There is no applicable EPA methods for analysis of PCBs in tissue. In addition, there are no applicable EPA methods for analysis of PCBs by low resolution mass spectrometry (MS).
[2] MDLs for 107 PCB congeners by MS are not available. PCB data will be evaluated against QLs.
[3] QLs determined from the low calibration standard and adjusted for sample processing volumes and factors.

QL = [(low calibration std., 0.012 ng/µL) * (pre-injection volume, 200 µL) * (dilution factor, 1.667)] / (Sample wet wt., 10-g). Actual QLs will be reported with the data.

[a] Coeluting congeners are listed in order of abundance in Aroclors 1242/1248/1254 (most abundant listed first). The most abundant single congener will be used to calibrate the instrument for the coeluting congener sets. CAS listing is applicable to the more abundant single isomer (*e.g.*, PCB4/10 – the CAS listing is for PCB4).
[b] The 18 bolded congeners are the NOAA NS&T and EPA EMAP PCB analytes.
[c] Total PCB by chlorination level = sum of the detected PCB congeners for chlorination level 1, 2, 3, etc.
[d] Total PCB = sum of the detected 107 PCB congeners.

EPA-NE QAPP Worksheet #9c- Rev. 10/99

Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Dup Pairs	Organic		Inorganic		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks	No. of PE Samples[3]	Total No. of Samples to Lab[4]
						No. of MS	No. of MSD	No. of Dups	No. of MS					
Tissue	Moisture Content	Low	L-2	10 (nestling)	0	0	0	0	0	0	0	0	0	10
Tissue	Dioxin/ Furan/HCX	Low	8290M/L-3	33	0	0	0	0	0	0	0	0	0	33
Tissue	PCB	Low	NS&T/L-20	10	0	0	0	0	0	0	0	0	0	10

[1] Complete the Field Analytical Method/SOP Reference Table (EPA-NE QAPP Worksheet #17), and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20) and specify the appropriate letter/number reference in the above table.

[2] If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location/station.

[3] PE samples include standard reference material (SRM) samples. Note – a tissue SRM will be prepared in the fixed laboratory with each batch of 20 or fewer authentic samples (see EPA-NE QAPP Worksheet #11b).

[4] Other laboratory control samples not included in Total Number of Samples to Lab include: laboratory sample replicates, laboratory method/procedural blanks, instrument control checks and laboratory control samples. Laboratory QC samples are identified in EPA-NE QAPP Worksheet #24 and include method blanks, IC, LCS, MS/MSD and sample duplicates.

EPA-NE QAPP Worksheet #9d- Rev. 10/99
Analytical Services Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Analytical Method/SOP[1]	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/ Organization (Name and Address: Contact Person and Telephone Number)
Tissue	Percent Moisture	Low	L-2	14 days	Battelle MSL 1529 Sequim Bay Road Sequim, WA 98382 (360) 681-3643	Backup balances available at Battelle Columbus and Duxbury
Tissue	Dioxin/ Furan/ HCX	Low	8290M/L-3	56 days	Battelle Columbus 505 King Avenue Columbus, OH 43201 (614) 424-4028	Backup GC/HRMS system available at Battelle Columbus
Tissue	PCB	Low	NS&T/L-20	45 days	Battelle Duxbury 397 Washington Street Duxbury, MA 02332 (781-934-0571)	Backup GC/MS systems available at Battelle Duxbury and Battelle Columbus

[1] Specify appropriate reference number/letter from the Field Analytical Method/SOP Reference Table (EPA-NE QAPP Worksheet #17) and from the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #10- Rev. 10/99

Project Schedule Timeline Table

Activities	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
	Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Task 15A QAPP Preparation				
Draft	10/18/00	10/24/00	QAPP Document	10/25/00 (draft)
NAE/EPA Review	10/25/00	11/03/00		
Final	11/03/00	11/07/00		11/08/00 (final)
Task 15 Tissue Chemical Analyses[1]				
Tissue Processing	10/18/00	10/31/00	Not Applicable	Not Applicable
Tissue Analyses (Dioxin/Furan/HCX, PCB)	11/08/00	12/15/00		
QA Data Audit	12/18/00	12/20/00		
Report				
Letter Data Report	12/20/00	12/21/00	Report	12/22/00
Copy of data packages/Raw Data	—	—	Data Package	1/10/01

[1] Participating laboratories will be responsible for sample custody and data quality control through validation process.

EPA-NE QAPP Worksheet #11a- Rev. 10/99

Project Quality Objectives/Decision Statements

Project Quality Objectives

The project quality objective for Task 15 Tissue Chemistry is to generate data of a quality to be used for ecological risk assessment (ERA). The data needed for ERA must be definitive data of very high quality (tight measurement performance criteria). Sensitivity requirements have not been defined.

Measurement Performance Criteria

Method performance criteria (MPC) chosen to ensure that the definitive data will be of high quality are defined by analysis parameter in EPA-NE QAPP Worksheet #11b.

Data quality may be defined in terms of accuracy, precision, representativeness, completeness, comparability, and sensitivity.

- *Accuracy* is the agreement between an observed value and an accepted value. Analytical accuracy is monitored for analytical chemistry measurements as specified in EPA Worksheet #11b (Attachment A).

  Applicable samples: LCS, MS, MSD, SRM, SIS (each sample).
  Applicable analyses: Dioxins/Furans and PCBs.

- *Precision* is defined as the degree of reproducibility among individual measurements of the same property, obtained under similar conditions. Measures of analytical precision will be determined in all phases of the program.

  Applicable samples: MS/MSD, DUP.
  Applicable analyses: Dioxins/Furans and PCBs. HCX (DUP only)

- *Representativeness* is the degree to which data accurately and precisely represent a characteristic of a population. Representativeness is addressed primarily in the sample design, through the selection of sampling sites and procedures that reflect the project goals and environment being sampled. It is ensured by the proper handling, homogenizing, compositing, and storage of samples and analysis within the specified holding times so that the material analyzed reflects the material collected as accurately as possible.

  Applicable analyses: Dioxins/Furans/HCX and PCBs.

- *Completeness* is defined as the amount of data collected as compared to the amount that is needed to make valid decisions. 100% completeness is targeted for all analyses. Study objectives will not be compromised if ≥95% of the analytes are reported.

  Applicable analyses: Dioxins/Furans/HCX and PCBs.

- *Comparability* is the measure of the confidence with which one data set can be compared to another. Data comparability for this project is addressed through the use of established laboratory methods that are comparable to other low-level methods.

  Applicable samples: SRM.
  Applicable analyses: Dioxins/Furans and PCBs.

EPA-NE QAPP Worksheet #11a- Rev. 10/99 (continued)

Project Quality Objectives/Decision Statements

- *Sensitivity* is the capability of methodology or instrumentation to discriminate among measurement responses for quantitative differences or a parameter of interest. Sensitivity expressed as the detection limits is presented in (EPA Worksheet #9b).

  Applicable analyses: Dioxins/Furans/HCX and PCBs.

- *Quantitation Limits*. Project-required limits of quantitation were not specified in the Statement of Work and are not available.

All deviations from protocols described in this QAPP will be documented and approved by the Project Manager and discussed in the final report. All data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results.

Affected samples may be reanalyzed at the Project Manager's discretion. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a, Data Verification and Validation Tasks), and the rationale for accepting the analysis will be thoroughly documented.

The calculation of quality control statistics is described in Battelle SOP 7-029 and are summarized in EPA-NE QAPP Worksheet #30 (Attachment A).

EPA-NE QAPP Worksheet #11b - Rev. 10/99

Measurement Performance Criteria Table

Medium/ Matrix	Tissue
Analytical Parameter	Dioxin/Furan/ HCX[a]
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
NA	L-3	Accuracy	<5× MDL, or associated samples >10× blank values	Blank	A
			PD≤30% from a range of certified values	Standard Reference Material	A
			50-120% R (Analyte concentration in MS/MSD must be >5× background concentration to be used for data quality assessment)	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			25-150% R	Internal Standards	A
		Precision	RPD≤30% (for analytes detected at level >10x MDL)	Sample Duplicates	
			RPD≤30% (Analyte concentration in MS/MSD must be >5× background concentration to be used for data quality assessment)	MS/MSD	A
		Comparability	See SRM above	Intercomparison exercises (*e.g.*, SRM analyses), follow defined SOPs	

MDL = Method Detection Limit; QL = Quantitation Limit; PD = Percent Difference; R = Recovery; RPD = Relative Percent Difference

[a] MPC criteria (*i.e.*, SRM, LCS, MS/MSD, SIS) is not applicable to HCX analysis. There is no available standard to fortify LCS, MS, or MSD samples with.

[1] Sampling was the responsibility of EPA-NE and is not discussed in this QAPP.

[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20 .

[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability)

EPA-NE QAPP Worksheet #11b - Rev. 10/99

Measurement Performance Criteria Table

Medium/ Matrix	Tissue
Analytical Parameter	PCBs
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
NA	L-20	Accuracy	< QL, or associated samples >10× blank values	Blank	A
			PD≤30% from a range of certified values (using surrogate corrected data; certified concentration in SRM must be >3× QL)	Standard Reference Material	A
			90% of congeners meet the following: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls (Concentration of spiked analytes in MS/MSD must be >5× background concentrations to be used for data quality assessment)	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			40-125% R	Surrogates	A
		Precision	RPD≤30% for at least 90% of analytes (for analytes detected at level >3× QL)	Sample Duplicates	A
			RPD≤30% for at least 90% of analytes (Using R data; Concentration of spiked analytes in MS/MSD must be >5× background concentrations to be used for data quality assessment)	MS/MSD	
		Comparability	See SRM above	Intercomparison exercises (*e.g.*, SRM analyses), follow defined SOPs	

QL = Quantitation Limit; PD = Percent Difference; R = Recovery; RPD = Relative Percent Difference

[1] Sampling was the responsibility of EPA-NE and is not discussed in this QAPP.

[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.

[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability)

EPA-NE QAPP Worksheet #16 Rev. 10/99

Sample Handling, Tracking and Custody Requirements

SAMPLE COLLECTION, PACKAGING AND SHIPMENT
Sample Collection: CM & TW Custer, C. Rosiu, and K. Munney, EPA-NE
Sample Packing: Andy Beliveau, EPA-NE
Coordination of Shipment: Andy Beliveau, EPA-NE
Type of Shipment (Courier): Federal Express
SAMPLE RECEIPT AND ANALYSIS
Responsible Organization: Battelle MSL (receive samples for freeze-drying) Battelle Duxbury (PCB analysis) Battelle Columbus (Dioxin/Furan/HCX analysis)
Sample Receipt: Carolynn Suslick, Sample Custodian Battelle MSL Micheal Meara, Sample Custodian Battelle Duxbury Mark Misita Sample Custodian Battelle Columbus
Sample Custody and Storage: Carolynn Suslick, Sample Custodian Battelle MSL Micheal Meara, Sample Custodian Battelle Duxbury Mark Misita Sample Custodian Battelle Columbus
Sample Preparation: Betsy Barrows, Freeze-drying at Battelle MSL Beth Kitson, PCBs at Battelle Duxbury Mark Misita and Henry Pham (assisted by Wesley Baxter), Dioxin/Furan/HCX at Battelle Columbus
Sample Determinative Analysis: Julie Fredriksson, PCB Analysis at Battelle Duxbury Joe Tabor, Dioxin/Furan/HCX at Battelle Columbus
SAMPLE ARCHIVAL
Field Sample Storage (No. of days from sample collection): 1 year if held frozen
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 40-d
SAMPLE DISPOSAL
Responsible Organization: Each participating laboratory is responsible for disposal of samples received for required analyses
Responsible Personnel: Sample Custodian

EPA-NE QAPP Worksheet #20 Rev. 10/99

Fixed Laboratory Analytical Method/SOP Reference Table

Ref Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code*	Analytical Parameter	Instrmt	Mod for Project Work Y or N
L-1	Battelle Marine Sciences Laboratory (MSL)	Sample Chain-of-Custody (SOP MSL-A-002)	NA	NA	NA	NA	N
L-2	Battelle MSL	Percent Dry Weight and Homogenizing Dry Sediment, Soil, and Tissue (SOP MSL-C-003)	Definitive	NA	Moisture Content	Analytical balance	N
L-3 [1]	Battelle Columbus	The Analysis of Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Using Modified Method 8290 (ASAT.II-001-01)	Definitive	NA	Dioxin/ Furan/ HCX	GC/ HRMS	N
L-4 [1]	Battelle Columbus	Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Sample Preparation Using Modified Methods 8290 (ASAT-II-002-01)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-5	Battelle Columbus	Internal QA Inspection and Corrective Action Procedures for Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) and Related Compounds Analytical Programs (ASAT.II-003-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-6	Battelle Columbus	Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Desiccating Agent And Adsorbent Preparation and Storage (ASAT.II-005-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N

[1] SOP was recently revised and is now included in Appendix E.

EPA-NE QAPP Worksheet #20 Rev. 10/99 (continued)

Fixed Laboratory Analytical Method/SOP Reference Table

Ref Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code*	Analytical Parameter	Instrmt	Mod for Project Work Y or N
L-7	Battelle Columbus	Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Standards and Reagents Preparation and Storage for Modified Method 8290 Analysis (ASAT.II-006-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-8 [1]	Battelle Columbus	Chain of Custody for Dioxin/Furan Analysis (ASAT.II-007-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-9 [2]	Battelle Columbus	Standard Operating Procedure for Dioxin/Furan Technical Data Review (ASAT.II-010-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-10 [2]	Battelle Columbus	Standard Operating Procedure for using Electronic and Mechanical Balances (ASAT.II-011-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-11 [2]	Battelle Columbus	Standard Operating Procedure (SOP) for the Use of Refrigerators and Freezers used for Dioxin-Related Projects (ASAT.II-012-00)	NA	NA	Dioxin/ Furan/HCX	GC/ HRMS	N
L-12 [2]	Battelle Columbus	Standard Operating Procedure (SOP) for the Use and Calibration of Digital and Glass Thermometers (ASAT.II-013-00)					
L-13	Battelle Duxbury	Use of the Cahn Model 25 and Cahn Model 28 Electrobalances(SOP 3-004)	NA	NA	PCB	GC/MS	N
L-14	Battelle Duxbury	Operation and Maintenance of Hewlett-Packard 5970B, 5972A and 5973A Gas Chromatograph/Mass Selective Detector (GC/MSD) using Hewlett-Packard Software (SOP 3-092)	Definitive	NA	PCB	GC/MS	N
L-15	Battelle Duxbury	Operation and Maintenance of Gas Chromatographs (SOP 3-116)	NA	NA	PCB	GC/MS	N
L-16	Battelle Duxbury	Use of Electronic Balances (SOP 3-160)	NA	NA	PCB	GC/MS	N

[1] SOP was recently revised and is now included in Appendix E.
[2] New SOP now provided in Appendix E.

EPA-NE QAPP Worksheet #20 (continued)

Fixed Laboratory Analytical Method/SOP Reference Table

Ref Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code*	Analytical Parameter	Instrmt	Mod for Project Work Y or N
L-17	Battelle Duxbury	Quality Assurance Facilities Inspections (SOP 4-009)	NA	NA	PCB	GC/MS	N
L-18	Battelle Duxbury	Quality Assurance Audits of Reported Data (SOP 4-015)	NA	NA	PCB	GC/MS	N
L-19	Battelle Duxbury	Non-Conformance and Corrective Action (SOP 4-035)	NA	NA	PCB	GC/MS	N
L-20	Battelle Duxbury	Identification and Quantification of Polynuclear Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry (SOP 5-157)	Definitive	NS&T Methods	PCB	GC/MS	Y[3]
L-21	Battelle Duxbury	Tissue Extraction for Trace Level Semivolatile Organic Contaminants (Draft SOP 5-190)	NA	NA	PCB	GC/MS	N
L-22	Battelle Duxbury	HPLC Cleanup of Samples for Semivolatile Organic Pollutants (SOP 5-191)	NA	NA	PCB	GC/MS	N
L-23	Battelle Duxbury	Cleaning of Organic Chemistry Labware (SOP 5-216)	NA	NA	PCB	GC/MS	N
L-24	Battelle Duxbury	Chemistry Laboratory Sample Identification (SOP 6-007)	NA	NA	PCB	GC/MS	N
L-25	Battelle Duxbury	Sample Receipt, Custody and Handling (SOP 6-010)	NA	NA	PCB	GC/MS	N
L-26	Battelle Duxbury	Documentation Procedures in the Gas Chromatography/Mass Spectrometry (GC/MS) Facility (SOP 6-011)	NA	NA	PCB	GC/MS	N
L-27	Battelle Duxbury	Data Recording (SOP 6-017)	NA	NA	PCB	GC/MS	N
L-28	Battelle Duxbury	Laboratory Verification and Validation of Analytical Data (SOP 6-027)	NA	NA	PCB	GC/MS	N
L-29	Battelle Duxbury	Preparation of Analytical Control Charts (SOP 7-028)	NA	NA	PCB	GC/MS	N
L-30	Battelle Duxbury	Preparation, Analysis, and Reporting Quality Control Data in the Chemistry Laboratory (SOP 7-029)	NA	NA	PCB	GC/MS	N

[3] Modified to include mass ions for the 107 PCB congeners. Quantification and confirmation mass ions provided in Appendix E with Battelle Duxbury SOP 5-157.

EPA-NE QAPP Worksheet #21 Rev. 10/99

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[1]	Method/ SOP Ref*
Freeze Dryer	Freeze Drying	Performance check (seals, vacuum pump operation, temperature) with each use	NA	NA	Maintenance or repair as needed	Deborah Coffey QA Officer	L-2
Analytical balance	Moisture Content	Daily performance check (or with each use).	1×/daily using two Class "S" weights bracketing expected weight range	within 0.001 g of value	Calibration and service performed as needed or at regular intervals by professional metrology technician.	Deborah Coffey QA Officer	L-2
GC/HRMS	Dioxin/ Furan/ HCX Analysis	Column if needed; clip retention gap if needed; tune HRMS; check for leaks; analyze calibration point 3, window set, and column performance check	IC: prior to analytical run; CC: every 12 hours	IC: RSD≤25% for native compounds and RSD≤35% for labeled compounds CC: Within limits of Method 1613B, Table 6 "VER" requirements	Remedial maintenance, new initial calibration, reanalyze samples or use of CC response factors in calculations. Document and justify	Joe Tabor (GC/HRMS Analyst) or Task Leader	L-3
GC/MS	PCB Analysis	Change injection port liner; tune MSD; check for leaks	IC: prior to analytical run; CC: every 10 samples	IC: RSD≤25% mean RSD≤15% CC: PD from initial <25%; mean PD≤15%	Remedial maintenance, new initial calibration, or reanalyze samples. Document and justify	Julie Fredriksson (GC/MS Analyst)	L-20

IC = Initial Calibration; CC = Continuing Calibration; RSD = Relative Standard Deviation; PD = Percent Difference.

[1] If specified project personnel not available; alternate and equally trained staff will perform task/corrective action

* Specify appropriate reference letter/number from Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24a - Rev. 10/99

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Tissue
Sampling SOP	NA
Analytical Parameter	Dioxin/ Furan/HCX
Concentration Level	Low
Analytical Method/ SOP Reference*	L-3
Laboratory Name	Battelle Columbus
No. of Sample Locations	3

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	1/sample set	NA	Reextract, reanalyze, or justify	GC/HRMS Analyst	Accuracy	<5× MDL, or associated samples >10× blank values
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	1/ sample set	NA	Review with Laboratory Manager; re-analyze or justify in project records	Task Leader	Precision	RPD≤30% (for analytes detected at level >10x MDL)
Laboratory Matrix Spike/Matrix Spike Duplicate	1/sample set	NA	Review with Laboratory Manager; re-analyze or justify in project records	Task Leader	Accuracy/ Precision	50-120% R RPD≤30% (Analyte concentration in MS/MSD must be >5× background concentration to be used for data quality assessment)
LCS	1 /sample set	Method 1613B, Table 6, "OPR" requirements	Review with Laboratory Manager; re-analyze or justify in project records NA	Task Leader	Accuracy	Method 1613B, Table 6, "OPR" requirements
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	15 per sample	25 – 150% R	Review with Laboratory Manager; re-analyze or justify in project records	GC/HRMS Analyst/ Preparation Analyst	Accuracy	25 – 150% R
Other: SRM	1/sample set	NA	Reextract, reanalyze or justification documented	GC/HRMS Analyst	Precision, Comparability	PD≤30% from certified values

MDL = Method Detection Limit; R = Recovery; RPD = Relative Percent Difference; OPR = Ongoing Precision and Recovery; PD = Percent Difference.

EPA-NE QAPP Worksheet #24a - Rev. 10/99

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Tissue
Sampling SOP	NA
Analytical Parameter	PCB
Concentration Level	Low
Analytical Method/ SOP Reference*	L-20
Laboratory Name	Battelle Duxbury
No. of Sample Locations	3

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	1/sample set	NA	Reextract and/or reanalyze; document corrective actions	GC/MS Analyst	Accuracy	< QL, or associated samples >10× blank values
Reagent Blank	1/lot purchase	NA	Review findings with laboratory manager; check different lot	GC/MS Analyst	Accuracy	Review findings with laboratory manager; check different lot
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA (as required)	NA	NA	NA	NA	NA
Laboratory Duplicate	1/ sample set	NA	Review with Project Manager; re-analyze or justify in project records	GC/MS Analyst	Precision	RPD≤30% for at least 90% of analytes (for analytes detected at level >3× QL)
Laboratory Matrix Spike/Matrix Spike Duplicate	1/sample set	NA	Reextract, reanalyze or justification documented	GC/MS Analyst	Accuracy/ Precision	90% of congeners meet the following: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls RPD≤30% for at least 90% of analytes (Concentration of spiked analytes in MS/MSD must be >5× background concentrations to be used for data quality assessment)

QL = Quantitation Limit; RPD = Relative Percent Difference; R = Recovery

EPA-NE QAPP Worksheet #24a (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Tissue
Sampling SOP	NA
Analytical Parameter	PCB
Concentration Level	Low
Analytical Method/ SOP Reference*	L-20
Laboratory Name	Battelle Duxbury
No. of Sample Locations	3

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	GC/MS Analyst	Accuracy	90% of congeners meet the following: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls
LFB	NA	NA	NA	NA	NA	NA
Surrogates	3 per sample	NA	Reextract, reanalyze or justification documented	GC/MS Analyst	Accuracy	40-125% R
Internal Standards (ISs)	3 per sample	NA	NA	NA	NA	NA
Other: SRM	1/sample set	NA	Reextract, reanalyze or justification documented	GC/MS Analyst	Precision, Comparability	PD≤30% from a range of certified values [a] (using surrogate corrected data; certified concentration in SRM must be >3× QL)
ICS	1/ sample set	NA	Internal QC check only	GC/MS Analyst	Precision	PD≤15% from true values

QL = Quantitation Limit; PD = Percent Difference; R = Recovery; ICS = Independent Control Sample

[a] If detected value for PCB falls within the *range of certified values*, then the PD is reported as 0.0. However, if the detected value for the PCB falls outside the range of certified values, then the PD is determined from either the upper or lower limit of the range. See Battelle SOP 7-029.

*Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99

Sampling SOP**: NA
Analytical Method/SOP: L-3 (Tissue); Dioxin/Furan/HCX

Fixed Laboratory Analytical QC Sample Table cont.

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (pg/g Wet Weight)	Analytical Precision	Analytical Accuracy/Bias
2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.5	RPD ≤ 30% between replicates PD ≤ 30% for SRM (for values >10x MDL)	50 – 120% R for MS and MSD 25 – 150% R for Internal Standards (where concentration in MS/MSD > 5x background levels)
2,3,7,8-Tetrachlorodibenzofuran	0.5	As above	As above
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	2.5	As above	As above
1,2,3,7,8-Pentachlorodibenzofuran	2.5	As above	As above
2,3,4,7,8-Pentachlorodibenzofuran	2.5	As above	As above
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	2.5	As above	As above
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	2.5	As above	As above
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.5	As above	As above
1,2,3,4,7,8-Hexachlorodibenzofuran	2.5	As above	As above
1,2,3,6,7,8-Hexachlorodibenzofuran	2.5	As above	As above
1,2,3,7,8,9-Hexachlorodibenzofuran	2.5	As above	As above
2,3,4,6,7,8-Hexachlorodibenzofuran	2.5	As above	As above
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	2.5	As above	As above
1,2,3,4,6,7,8-Heptachlorodibenzofuran	2.5	As above	As above
1,2,3,4,7,8,9-Heptachlorodibenzofuran	2.5	As above	As above
Octachlorodibenzo-p-dioxin	5	As above	As above
Octachlorodibenzofuran	5	As above	As above
1,2,4,5,7,8-Hexachloro-9-Xanthene	NA	As above	NA

RPD = Relative Percent Difference; PD = Percent Difference; R = % Recovery; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; MDL = Method Detection Limit.

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99

Sampling SOP**: NA
Analytical Method/SOP: L-20 (Tissue); PCBs

Fixed Laboratory Analytical QC Sample Table cont.

Analyte[a]	Achievable Laboratory Sensitivity/ Quantitation Limits (wet weight)	Analytical Precision	Analytical Accuracy/Bias
PCB1	0.4 ng/g	RPD≤30% for at least 90% of analytes between replicates PD ≤30% for SRM (for values >3× QL)	90% of congeners in LCS/MS/MSD meet: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls 40 – 125% R for SISs (Concentration of spiked analytes in MS/MSD must be >5x background concentrations to be used for data quality assessment)
PCB3	0.4 ng/g	As above	As above
PCB4/10	0.4 ng/g	As above	As above
PCB6	0.4 ng/g	As above	As above
PCB7/9	0.4 ng/g	As above	As above
PCB8/5[b]	0.4 ng/g	As above	As above
PCB12/13	0.4 ng/g	As above	As above
PCB16/32	0.4 ng/g	As above	As above
PCB17	0.4 ng/g	As above	As above
PCB18	0.4 ng/g	As above	As above
PCB19	0.4 ng/g	As above	As above
PCB21	0.4 ng/g	As above	As above
PCB22	0.4 ng/g	As above	As above
PCB24/27	0.4 ng/g	As above	As above
PCB25	0.4 ng/g	As above	As above
PCB26	0.4 ng/g	As above	As above
PCB28	0.4 ng/g	As above	As above
PCB29	0.4 ng/g	As above	As above
PCB31	0.4 ng/g	As above	As above
PCB33/20	0.4 ng/g	As above	As above
PCB40	0.4 ng/g	As above	As above
PCB41/64/71	0.4 ng/g	As above	As above
PCB42	0.4 ng/g	As above	As above
PCB43	0.4 ng/g	As above	As above
PCB44	0.4 ng/g	As above	As above

RPD = Relative Percent Difference; PD = Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; SIS = Surrogate Internal Standards; R = Recovery; QL = Quantitation Limit

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99 (continued)

Sampling SOP**: NA
Analytical Method/SOP: L-20 (Tissue); PCBs

Fixed Laboratory Analytical QC Sample Table cont.

Analyte[a]	Achievable Laboratory Sensitivity/ Quantitation Limits (wet weight)	Analytical Precision	Analytical Accuracy/Bias
PCB45	0.4 ng/g	RPD≤30% for at least 90% of analytes between replicates PD ≤30% for SRM (for values >3× QL)	90% of congeners in LCS/MS/MSD meet: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls 40 – 125% R for SISs (Concentration of spiked analytes in MS/MSD must be >5x background concentrations to be used for data quality assessment)
PCB46	0.4 ng/g	As above	As above
PCB47/75	0.4 ng/g	As above	As above
PCB48	0.4 ng/g	As above	As above
PCB49	0.4 ng/g	As above	As above
PCB51	0.4 ng/g	As above	As above
PCB52	0.4 ng/g	As above	As above
PCB53	0.4 ng/g	As above	As above
PCB56/60	0.4 ng/g	As above	As above
PCB59	0.4 ng/g	As above	As above
PCB63	0.4 ng/g	As above	As above
PCB66	0.4 ng/g	As above	As above
PCB70/76	0.4 ng/g	As above	As above
PCB74	0.4 ng/g	As above	As above
PCB82	0.4 ng/g	As above	As above
PCB83	0.4 ng/g	As above	As above
PCB84	0.4 ng/g	As above	As above
PCB85	0.4 ng/g	As above	As above
PCB87/115	0.4 ng/g	As above	As above
PCB89	0.4 ng/g	As above	As above
PCB91	0.4 ng/g	As above	As above
PCB92	0.4 ng/g	As above	As above
PCB95	0.4 ng/g	As above	As above
PCB97	0.4 ng/g	As above	As above
PCB99	0.4 ng/g	As above	As above

RPD = Relative Percent Difference; PD = Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; SIS = Surrogate Internal Standards; R = Recovery; QL = Quantitation Limit

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99 (continued)

Sampling SOP**: NA
Analytical Method/SOP: L-20 (Tissue); PCBs

Fixed Laboratory Analytical QC Sample Table cont.

Analyte[a]	Achievable Laboratory Sensitivity/ Quantitation Limits (wet weight)	Analytical Precision	Analytical Accuracy/Bias
PCB100	0.4 ng/g	RPD≤30% for at least 90% of analytes between replicates PD ≤30% for SRM (for values >3× QL)	90% of congeners in LCS/MS/MSD meet: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls 40 – 125% R for SISs (Concentration of spiked analytes in MS/MSD must be >5x background concentrations to be used for data quality assessment)
PCB101/90	0.4 ng/g	As above	As above
PCB105	0.4 ng/g	As above	As above
PCB107	0.4 ng/g	As above	As above
PCB110	0.4 ng/g	As above	As above
PCB114	0.4 ng/g	As above	As above
PCB118	0.4 ng/g	As above	As above
PCB119	0.4 ng/g	As above	As above
PCB124	0.4 ng/g	As above	As above
PCB128	0.4 ng/g	As above	As above
PCB129	0.4 ng/g	As above	As above
PCB130	0.4 ng/g	As above	As above
PCB131	0.4 ng/g	As above	As above
PCB132	0.4 ng/g	As above	As above
PCB134	0.4 ng/g	As above	As above
PCB135/144	0.4 ng/g	As above	As above
PCB136	0.4 ng/g	As above	As above
PCB137	0.4 ng/g	As above	As above
PCB138/160/163	0.4 ng/g	As above	As above
PCB141	0.4 ng/g	As above	As above
PCB146	0.4 ng/g	As above	As above
PCB149	0.4 ng/g	As above	As above
PCB151	0.4 ng/g	As above	As above
PCB153	0.4 ng/g	As above	As above
PCB156	0.4 ng/g	As above	As above

RPD = Relative Percent Difference; PD = Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; SIS = Surrogate Internal Standards; R = Recovery; QL = Quantitation Limit

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99 (continued)

Sampling SOP**: NA
Analytical Method/SOP: L-20 (Tissue); PCBs

Fixed Laboratory Analytical QC Sample Table cont.

Analyte[a]	Achievable Laboratory Sensitivity/ Quantitation Limits (wet weight)	Analytical Precision	Analytical Accuracy/Bias
PCB158	0.4 ng/g	RPD≤30% for at least 90% of analytes between replicates PD <30% for SRM (for values >3× QL)	90% of congeners in LCS/MS/MSD meet: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls 40 – 125% R for SISs (Concentration of spiked analytes in MS/MSD must be >5x background concentrations to be used for data quality assessment)
PCB167	0.4 ng/g	As above	As above
PCB169	0.4 ng/g	As above	As above
PCB170/190	0.4 ng/g	As above	As above
PCB171	0.4 ng/g	As above	As above
PCB172	0.4 ng/g	As above	As above
PCB173	0.4 ng/g	As above	As above
PCB174	0.4 ng/g	As above	As above
PCB175	0.4 ng/g	As above	As above
PCB176	0.4 ng/g	As above	As above
PCB177	0.4 ng/g	As above	As above
PCB178	0.4 ng/g	As above	As above
PCB180	0.4 ng/g	As above	As above
PCB183	0.4 ng/g	As above	As above
PCB184	0.4 ng/g	As above	As above
PCB185	0.4 ng/g	As above	As above
PCB187/182	0.4 ng/g	As above	As above
PCB189	0.4 ng/g	As above	As above
PCB191	0.4 ng/g	As above	As above
PCB193	0.4 ng/g	As above	As above
PCB194	0.4 ng/g	As above	As above
PCB195	0.4 ng/g	As above	As above
PCB197	0.4 ng/g	As above	As above
PCB198	0.4 ng/g	As above	As above
PCB199	0.4 ng/g	As above	As above

RPD = Relative Percent Difference; PD = Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; SIS = Surrogate Internal Standards; R = Recovery; QL = Quantitation Limit

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #24b - Rev. 10/99 (continued)

Sampling SOP**: NA
Analytical Method/SOP: L-20 (Tissue); PCBs

Fixed Laboratory Analytical QC Sample Table cont.

Analyte[a]	Achievable Laboratory Sensitivity/ Quantitation Limits (wet weight)	Analytical Precision	Analytical Accuracy/Bias
PCB200	0.4 ng/g	RPD≤30% for at least 90% of analytes between replicates PD ≤30% for SRM (for values >3× QL)	90% of congeners in LCS/MS/MSD meet: 50 to 125% R for tri- through decachlorobiphenyls 30 to 125% R for mono- and dichlorobiphenyls 40 – 125% R for SISs (Concentration of spiked analytes in MS/MSD must be >5x background concentrations to be used for data quality assessment)
PCB201	0.4 ng/g	As above	As above
PCB203/196	0.4 ng/g	As above	As above
PCB205	0.4 ng/g	As above	As above
PCB206	0.4 ng/g	As above	As above
PCB207	0.4 ng/g	As above	As above
PCB209	0.4 ng/g	As above	As above
Total PCB by Chlorination Level [c]	—	—	—
Total PCB [d]	—	—	—

RPD = Relative Percent Difference; PD = Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; SIS = Surrogate Internal Standards; R = Recovery; QL = Quantitation Limit

[a] Coeluting congeners are listed in order of abundance in Aroclors 1242/1248/1254 (most abundant listed first). The most abundant single congener will be used to calibrate the instrument for the coeluting congener sets. CAS listing is applicable to the more abundant single isomer (*e.g.*, PCB4/10 – the CAS listing is for PCB4).
[b] The 18 bolded congeners are the NOAA NS&T and EPA EMAP PCB analytes.
[c] Total PCB by chlorination level = sum of the detected PCB congeners for chlorination level 1, 2, 3, etc.
[d] Total PCB = sum of the detected 107 PCB congeners.

**Specify appropriate reference number/letter from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13) and the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #26- Rev. 10/99

Project Documentation and Records Table

Sample Collection Records[1]	Field Analysis Records	Fixed Laboratory Records	Data Assessment Records	Other
Field Logs	N/A	Sample Receipt, Custody and Tracking Forms	Performance Audit Reports (sample preparation, GC/MS, GC/HRMS)	
Custody Forms		Standard Preparation Logs (certificates of analysis, QC checks)	Lab-wide Systems Audit Reports	
Sample Labels		Equipment Calibration Logs	PE Sample Results	
Custody Seals		Sample Preparation Records	Corrective Action Forms	
Telephone Logs		GC/MS and GC/HRMS Logbooks (acquisition, maintenance, tuning)	Control Charts	
		Calibration Reports	Telephone Logs and/or electronic mail	
		Sample Quantification Reports		
		Sample Chromatograms		
		Final Report Tables (authentic samples and QC results)		
		Data Validation Checklist[2]		
		Preliminary QC Checklist		
		Corrective Action Logs (miscellaneous documentation)		
		Study Records (*e.g.*, Data Package)		
		Sample Disposal Records		
		Telephone Logs and/or electronic mail		

[1] EPA-NE collected samples and maintenance of Sample Collection Records is the responsibility of EPA-NE.
[2] Example of Validation Checklist used at Battelle Duxbury and Columbus provided in Attachment D.

EPA-NE QAPP Worksheet #27a- Rev. 10/99

Assessment and Response Actions

Quality assurance encompasses all planned and systematic activities necessary to assure management that the products generated, and the services performed by Battelle meet the quality standards established in this QAPP. The primary mechanism for accomplishing this goal is audits. Audits refer to the formal assessment of conformance to the QA Program and its effectiveness. During an audit, the agreement with QA policy documents (*e.g.*, SOPs) is evaluated, deficiencies are identified, and corrective action is taken. Ideally, audits also serve to increase awareness and understanding of QA policies and procedures. Ms. Rosanna Buhl will serve as Battelle Duxbury's QA Officer and is responsible for identifying areas for corrective action, coordinating the QA activities such as systems and data audits, and preparing reports to management for this project. QA Officers at participating laboratories will be responsible for coordinating and performing QA activities at participating laboratories. Identity and qualifications of auditors are presented in EPA-NE QAPP Worksheet #6. The following QA audits are planned for this project.

- A technical system (initiation) audit is conducted as part of the review of this QAPP to (1) ensure that the work assignment scope and all required elements are addressed adequately, (2) verify that all required SOPs are approved and current, and (3) to verify that all participants have the required qualifications and documented training to perform their assigned tasks.

- Performance audits are independent checks of routinely obtained data. One Certified or Standard Reference Material (CRM or SRM, respectively) will be incorporated into each batch of tissue chemistry samples (as applicable) to assess the accuracy and precision with which target analytes of known concentration are recovered from a representative matrix. The acceptance criteria are discussed in EPA-NE QAPP Worksheet #11b.

- Systems audits at Battelle Duxbury are conducted at least quarterly by the Program QA Officer or her designee. The purpose of these audits is to evaluate facilities, equipment, and processes for conformance to Battelle standards. Battelle SOP 4-009 describes the procedures for conducting the audits. Specific laboratory activities (*e.g.*, sample custody, preparation of standard solutions, training records) may be targeted for review based on need and level of laboratory activity in addition to the general criteria specified in the inspection SOP. Data audits will conducted for all reported data. These audits will reconstruct representative data from each sample based on sample processing records, instrument calibration factors (*e.g.*, response factors) and output (*e.g.*, area counts), and sample manipulations and spiking. Samples will be tracked from receipt and processing through analysis and reporting to ensure that the reported data are accurate, complete, and traceable. Section 17 discusses the reporting of audit results to management and Section 16.2 describes corrective action procedures resulting from audit findings. A QA Statement submitted to the Project Manager with each deliverable describes the audit and review activities conducted to assess the deliverable accuracy, and any outstanding issues that could impact data quality.

Data audits are also conducted as described in EPA-NA QAPP Worksheet #9a (Attachment A).

EPA-NE QAPP Worksheet #27b- Rev. 10/99

Project Assessment Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) responsible for performing assessment, title and organizational affiliation	Person(s) responsible for responding to assessment findings, title and organizational affiliation	Person (s) responsible for identifying and implementing corrective actions (CA), title and organizational affiliation	Person (s) responsible for monitoring effectiveness of CA, title and organizational affiliation
Fixed Laboratory Data Package Audit	each data package	Internal	Battelle MSL	Deborah Coffey QA Officer Battelle MSL	Freeze-Drying Task Leader Battelle MSL	Betsy Barrows Project Manager Battelle MSL	Deborah Coffey QA Officer Battelle MSL
Fixed Laboratory Data Package Audit	each data package	Internal	Battelle Columbus	Charles D. Lawrie QA Officer Battelle Columbus	Analysts (GC/HRMS, Sample Preparation) Battelle Columbus	Mary Schrock Laboratory Manager and/or Karen Tracy Task Leader Battelle Columbus	Charles D. Lawrie QA Officer Battelle Columbus
Fixed Laboratory Data Package Audit	each data package	Internal	Battelle Duxbury	Rosanna Buhl QA Officer Battelle Duxbury	GC/MS Analyst Battelle Duxbury	Robert Lizotte Laboratory Manager Battelle Duxbury	Rosanna Buhl QA Officer Battelle Duxbury

EPA-NE QAPP Worksheet #27c - Rev. 10/99

Project Assessment Plan

QAPP Title:	Centredale Manor QAPP
Assessed Organization:	Battelle Columbus
Location of Assessment:	Battelle Columbus, Columbus, OH
Dates of Assessment:	Completion of analytical task
Assessment Team Members:	Charles D. Lawrie
Type of Assessment:	Data audit
Assessment Scope:	Audit data package for completeness and accuracy
Documents to be Reviewed:	Dioxin/Furan/HCX data packages (custody, sample processing data, GC/HRMS data and calibrations, and final report tables)
Notification Date(s):	At completion of audit (mid to late December, 2000)
Proposed Schedule:	Estimated mid to late December, 2000
Assessment No.:	
Contract No.:	

EPA-NE QAPP Worksheet #27c - Rev. 10/99

Project Assessment Plan

QAPP Title:	Centredale QAPP
Assessed Organization:	Battelle Duxbury
Location of Assessment:	Battelle Duxbury, Duxbury, MA
Dates of Assessment:	Completion of analytical task
Assessment Team Members:	Rosanna Buhl
Type of Assessment:	Data audit
Assessment Scope:	Audit data package for completeness and accuracy in accordance with Battelle SOP 4-015
Documents to be Reviewed:	PCB data packages (custody, sample processing data, GC/MS data and calibrations, and final report tables)
Notification Date(s):	At completion of audit (mid to late December, 2000)
Proposed Schedule:	Estimated mid to late December, 2000
Assessment No.:	
Contract No.:	

EPA-NE QAPP Worksheet #28 - Rev. 10/99

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation, Title and Organizational Affiliation	Report Recipients, Title and Organizational Affiliation
Data Audit	1/data package (percent moisture - nestling)	At end of analytical task	Deborah Coffey QA Officer Battelle MSL	Freeze-Drying Task Leader Battelle MSL
Data Audit	1/data package (Dioxin/Furan/HCX)	At end of analytical task	Charles D. Lawrie QA Officer Battelle Columbus	Sample Preparation, HRGC/HRMS Analyst, Laboratory Manager, Task Leader Battelle Columbus
Data Audit	1/data package (PCB)	At end of analytical task	Rossana Buhl QA Officer Battelle Duxbury	GC/MS Analyst, Laboratory Manager, and Project Manager Battelle Duxbury

EPA-NE QAPP Worksheet #29a - Rev. 10/99

Data Verification Process

Verification Task	Description	I/E	Responsible for Verification (Name, Organization)
QA Audit (Percent Moisture)	See Section 19.0 of this QAPP and EPA-NE QAPP Worksheet #9a		Betsy Barrows (Freeze-Drying Task Leader) Deborah Coffey (QA Officer) Battelle MSL
QA Audit (Dioxin/Furan/HCX)	As above		Mark Misita (Sample Preparation) Joe Tabor (HRMS Analyst) Karen Tracy (Task Leader) Mary Schrock (Laboratory Manager) Charles D. Lawrie (QA Officer) Battelle Columbus
QA Audit (PCB)	As above		Beth Kitson (Sample Preparation) Julie Fredriksson (GC/MS Analyst) Deirdre Dahlen (Task Leader) Rosanna Buhl (QA Officer) Battelle Duxbury

EPA-NE QAPP Worksheet #29b - Rev. 10/99

Data Validation Summary Table

Medium/ Matrix	Analytical Parameter	Conc Level	Validation Criteria[1]	Validation Criteria Modified[2]	Data Validation Tier Level	Modified Tier Level Used[3]	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
Tissue	Percent Moisture (Nestling)	Low	see below Section 18 of this QAPP and EPA-NE QAPP Worksheet #9a	NA	NA	NA	Freeze-Drying ask Leader Battelle MSL	Freeze-Drying Task Leader Battelle MSL
Tissue	Dioxin/ Furan/HCX	Low	As above	NA	NA	NA	GC/HRMS Analyst Battelle Columbus	GC/HRMS Analyst Battelle Columbus
Tissue	PCB	Low	As above	NA	NA	NA	GC/MS Analyst Battelle Duxbury	GC/MS Analyst Battelle Duxbury

[1] If the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses will not be used to validate project data, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the alternate validation criteria and/or procedures that will be used.

[2] If the Region I validation criteria will be modified to meet project objectives, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the modified validation criteria that will be used.

[3] If a modified validation Tier will be used to validate project data, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the Tier modifications that will be used.

EPA-NE QAPP Worksheet #29c - Rev. 10/99

Data Validation Modifications

Participating laboratories (*i.e.*, Battelle MSL, Battelle Columbus.) will be responsible for performing data validation and verification of the data package (*i.e.*, Percent Moisture, Dioxin/Furan/HCX). Data validation and verification procedures will follow internal laboratory SOPs and are further described in EPA-NE QAPP Worksheet #9a. The appropriate Task Leader will document and justify modifications to data validation/verification procedures, followed up by notification to the Project Manager for approval.

EPA-NE QAPP Worksheet #30 - Rev. 10/99

Data Usability Assessment

Data Usability Assessment
Data will be evaluated for usability as described in Figure 3 (EPA-NE QAPP Worksheet #30).

Calculation of Quality Control Data
The calculation of quality control statistics is described in Battelle SOP 7-029 and routine methods used to assess precision and accuracy are described below.

Accuracy —Accuracy is the closeness of agreement between an observed value and an accepted value. Accuracy of analyses is assessed through analysis of laboratory control samples (LCS), standard reference materials (SRM), matrix spikes (MS/MSD), surrogate internal standards (SIS), and method or procedural blanks.

Accuracy is quantified through the use of the following equations:

- Percent Recovery (R) in authentic samples (*e.g.*, matrix spikes)

$$R = [(C_s - C_u) \div S] \times 100$$

Where,
C_s = concentration of spiked sample
C_u = concentration in unspiked sample and
S = expected concentration of spike in sample

- Percent Recovery based on known concentrations (*e.g.*, surrogates)

$$R = (C_s \div C_c) \times 100$$

Where,
C_c = certified or true concentration and
C_s = concentration of sample

- Percent Difference (PD) based on known concentrations (*e.g.*, SRM)

$$PD = (|C_c - C_s| \div C_c) \times 100$$

Where,
C_c = certified or true concentration and
C_s = concentration of sample

EPA-NE QAPP Worksheet #30 - Rev. 10/99 (continued)

Data Usability Assessment

Precision—Precision is defined as the degree of reproducibility among individual measurements of the same property, obtained under similar conditions. Measure of analytical precision may be determined by the analysis of laboratory replicate and matrix spike duplicates. Laboratory replicates are prepared by homogenizing and splitting samples in the laboratory, and carrying the subsamples through the entire analytical process.

Precision is quantified through the use of the following mathematical formulae:

- For two samples, Relative Percent Difference (RPD)

$$RPD = [\,|A - B|\, \div (A + B)] \times 200$$

Where,
A and B are the concentrations (or percent recoveries) detected in the two samples.

Limits of Detection—All PCB results will be reported relative to the quantitation limit (QL) for that compound. The QL is equivalent to a final extract concentration that is the same as the low calibration standard concentration.

- QLs are calculated as

QL = (Concentration in Low Std. × final extract volume × dilution factors)/ Sample size

Method detection limits for Dioxins/Furans/HCX are presented in EPA-NE QAPP Worksheet #9b. However, all Dioxin/Furan/HCX results will be reported relative to the sample specific estimated detection limit (EDL) for that compound. The sample specific EDL is defined as 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.

- EDLs are calculated as

$$EDL = (2.5 \times H_x \times Q_{is}) / (H_{is} \times W \times RF_n)$$

Where,

H_x = Sum of the height of the noise level for each quantitation ion for the unlabeled PCDDs/PCDFs.
H_{is} = Sum of the height of the noise level for each quantitation ion for the labeled internal standard.
W = Sample size, g wet weight
RF = Calculated mean relative response factor for each analyte
Q_{is} = Quantity, in pg, of the internal standard added to sample before extraction.

Results reported below the sample-specific QLs and EDLs are flagged as such.

EPA-NE QAPP Worksheet #30 - Rev. 10/99 (continued)

Data Usability Assessment

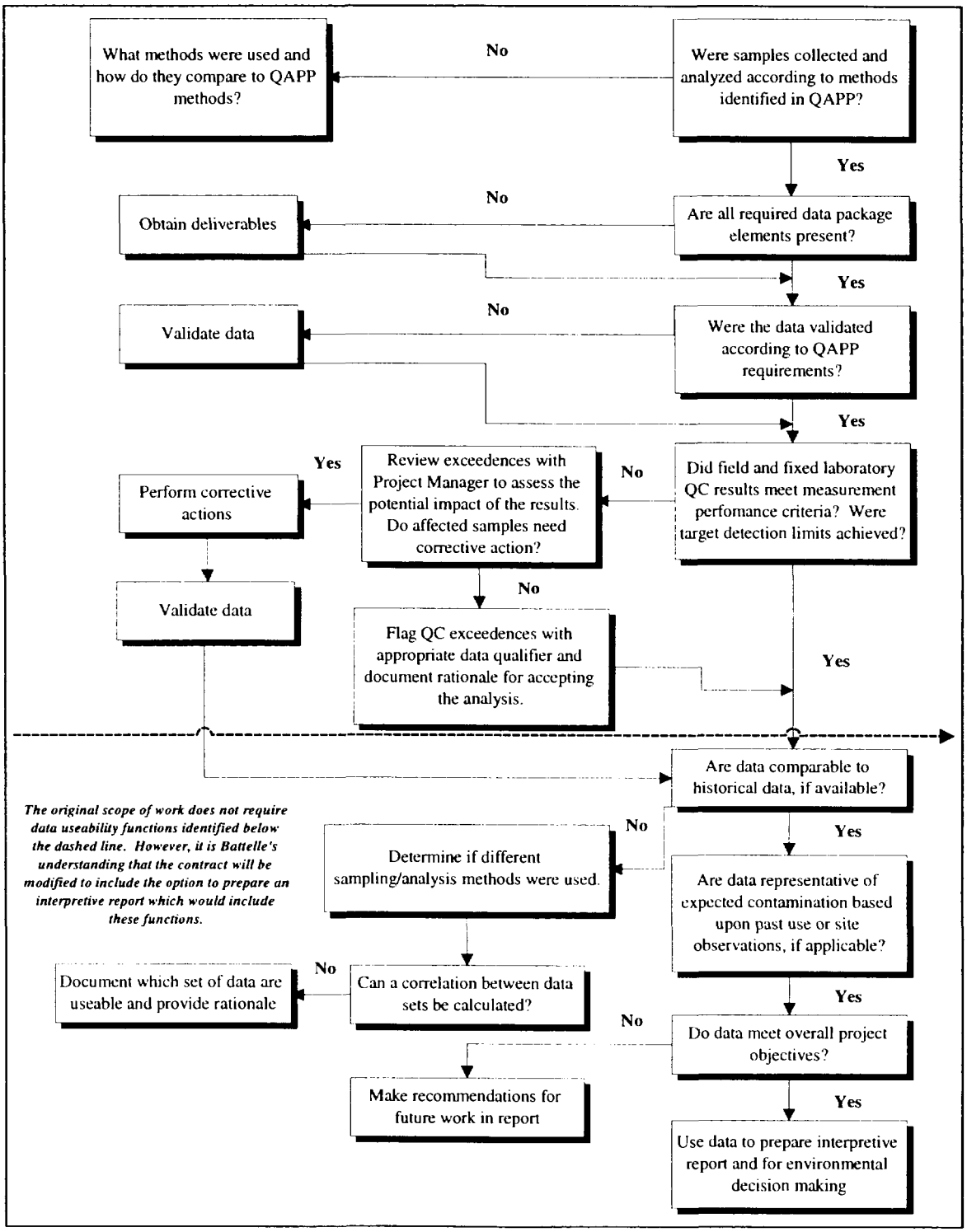


Figure 3. Preliminary Data Review Decision Tree.

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ATTACHMENT B

Resumes

PROJECT MANAGER

Donald G. Gunster **Battelle Duxbury**

Principal Research Scientist

Education:

M.E.M. (Master of Environmental Management), Ecotoxicology, Duke University School of Forestry and Environmental Studies, Durham, North Carolina, 1990
B.S. Biology, Dickinson College, Carlisle, Pennsylvania, 1986

Qualifications:

Mr. Gunster has ten years' experience in managing all aspects of large and small environmental assessments, focusing on ecological and human health risk assessments, and designing and implementing multidisciplinary ecological investigations. His qualifications encompass broad-based undergraduate and graduate degrees in biology and environmental science. As marine researcher, he has examined the effects of herbicides and insecticides in agricultural runoff on blue crabs, mud crabs, and other marine invertebrates and assisted in the development of natural and synthetic compounds which inhibit invertebrates from attaching to submerged surfaces.

As a member of the Massachusetts Ecological Risk Assessment Work Group, a peer review committee established by Massachusetts Department of Environmental Protection, Mr. Gunster assisted in the development of regulatory risk assessment guidance for the Commonwealth of Massachusetts. Through his project work, Mr. Gunster has participated in numerous negotiations with state and federal agencies on behalf of private industrial clients and federal entities.

With experience conducting ecological risk assessments in estuarine, freshwater, wetland, and terrestrial systems, he has designed, implemented, and managed large and small multidisciplinary projects combining biological, soil, sediment, and surface water quality assessments to support risk evaluations and risk-based remedial decisions. He managed a two-year ecological field investigation for a large RCRA corrective action project in a major estuarine system in Louisiana and has successfully managed project with budgets ranging from $10,000 to $8,100,000. His work as Technical Lead and Co-Project Manager for a large CERCLA ecological investigation at the Otis Air National Guard Base at the Massachusetts Military Reservation on Cape Cod, Massachusetts encompassed a wide range of responsibilities including the development of an overall strategy, program plan, site-specific work plans, and field sampling plans. Each of these plans received approval from state and federal environmental regulatory agencies. In addition, this project involved assessing ecological impacts and risks, developing monitoring plans to evaluate potential long-term ecological impacts, data interpretation, and participating in regulatory negotiations.

Relevant Experience:

Lead Risk Assessor/Co-Project Manager: Assessed the ecological risk from contaminated groundwater and the ecological impact from the construction and operation of groundwater treatment systems. Evaluated ponds, rivers, vernal pools, wetlands, and estuarine ecosystems which were hydraulically down-gradient of contaminated groundwater plumes and groundwater treatment systems. Responsible for developing the overall program plan and site-specific field sampling plans. Interpreted the results and assessed data gaps for seven groundwater plumes. Responsible for the supervision of staff and subcontractors ranging from two to eight people.

Risk Assessor: Provided human health and ecological risk assessment support to the engineering design team in evaluating remedial treatment options. Evaluated the potential human health and ecological risks associated with the various construction and operation scenarios for the proposed groundwater treatment systems and evaluated the risks associated with exposure to the unmitigated portion of groundwater plumes. In addition, provided human health risk assessment support on behalf of local communities to assist in evaluating the need for and priority of identifying alternative potable water sources.

Risk Assessor/Assistant Project Manager: In support of a RCRA facility investigation of a Fortune 500 chemical manufacturing plant in Louisiana, evaluated ecological and human health risks associated with historical disposal practices, accidental releases, and releases from other local industrial facilities to an adjacent river and estuary system. Developed sampling work plans for sediment, water, and biota; participated in community education and out-reach programs; participated in negotiations and presentations to several state and federal regulatory agencies, and addressing issues related to dredging activities, dredged material disposal, and natural resource damages. Responsible for the supervision of staff, field crews, and subcontractors ranging from two to 10 people.

Risk Assessor: Member of a peer review work group established by the Massachusetts Department of Environmental Protection to develop ecological risk assessment guidance under the Massachusetts Contingency Plan (MCP). This guidance addressed terrestrial, aquatic, and wetland ecosystems.

Risk Assessor/Task Manager: Manager of a biological and habitat evaluation of a six-square-mile study area as the initial phase (i.e., problem formulation) of an ecological risk assessment. The investigation included characterizing terrestrial and aquatic vegetation including wetlands, development of a habitat map, and the compilation of a species list through a 12-month survey of small mammal, bird, fish, reptile, amphibian, and benthic communities. The results of this investigation were used to develop a food web model and to focus subsequent phases of the ecological risk assessment.

Risk Assessor: Assisted in conducting an ecological risk assessment of a river system receiving runoff from an adjacent Superfund site. This project involved evaluating the impact of contaminated sediment, surface water, and groundwater on aquatic biota.

Risk Assessor: Conducted a human health evaluation of the potential risks associated with formaldehyde and epichlorohydrin in packaging material in accordance with the California Proposition 65.

Technical Lead: Instrumental in reducing the yearly ecological project costs from 12 million to 8.1 million dollars, through the development of a focused comprehensive approach that targeted specific ecosystems and ecological receptor groups that were most likely to be impacted by groundwater treatment systems. Instrumental in developing a set of ecological thresholds to identify physicochemical and biological attributes of concern and defining acceptable levels of change in downgradient ecosystems. This threshold provided a means to balance the benefits of treating contaminated groundwater while minimizing unacceptable impacts to sensitive ecosystems.

Project Manager: Developed, implemented, and managed, a monitoring program of physical and physio-chemical parameters in an estuarine system. Measurements included profile sampling of dissolved oxygen, salinity, temperature, redox potential, biological oxygen demand in surface water and total organic carbon and ammonia in sediments. The results were combined with biological surveys to evaluate non-chemical impacts to aquatic organisms.

Project Manager: Prepared and submitted a comprehensive dredge permit application to the US Army Corps of Engineers, New Orleans District, for maintenance dredging of a ship terminal facility. This permit application required collecting, compiling, analyzing, and interpreting physical and chemical data for sediment and surface water within the guidelines and structure outlined by the US Army Corp of Engineers and US EPA.

Project Manager: Evaluate potential ecological impacts from historical disposal of heavy metals into a tidal cove ecosystem including associated forested and marsh wetlands. This project examined potential impacts to the flora and fauna as well as the functional and structural components of the wetland system resulting from various remedial designs.

Assistant Project Manager: Assisted in the development of an alternative wetland restoration design as part of the remediation of a Superfund site. This project addressed restoration of bathymetry, vegetative species, planting design, pre- and post-monitoring design, and associated materials and costs for implementation.

Field Manager: Conducted an ecological evaluation of potential impacts to fish, small mammal, terrestrial and aquatic vegetation, and benthic macroinvertebrate species resulting from historical disposal practices at the Portsmouth Naval Shipyard facility in Maine. This evaluation included designing and implementing field-sampling procedures in compliance with RCRA guidelines.

Scientist: Conducted a quantitative vegetation surveys as part of two New England site investigations. Both surveys involved the identification of indigenous plant species, canopy, and percent cover analysis. Results were compared to reference sites and literature studies to evaluate potential impacts. Phytotoxic analysis, based on chemical concentration in the soil, was conducted at one of these sites to evaluate adverse effects.

Scientist: Conducted an ecological evaluation of potential risks associated with PCB contamination in a northeastern river and its floodplain. Designed and performed population-level and reproductive studies on insectivorous breeding birds and small mammals. Compared density, population size, diversity, age structure, and reproductive success of 15 species from impacted areas, control locations, and similar habitats evaluated in literature studies.

Scientist: Contributed to the development of testimonies for expert witnesses in support of proposed ambient water quality standards for several states.

Scientist: Compared a risk-based approach to establishing fish consumption health advisories to the New Jersey Department of Public Health approach for the Newark Bay region. This project evaluated the potential risks associated with the consumption of fish and shellfish exposed to a mixture of chemical contaminants.

Graduate School Research: Examined the metabolic fate of chlorinated phenolic compounds found in bleached kraft pulp and paper effluent. Examine the metabolites found in the gall bladder bile and blood of catfish exposed to a mixture of chlorinated phenolic compounds.

Graduate School Research: Developed a gas chromatography method to analyze chlorinated phenolic compounds and their metabolites from the gall bladder bile and blood plasma samples collected from channel catfish exposed to a mixture of chlorinated phenolic compounds.

Graduate School Research: Investigated the ecological impact of feral horses grazing on barrier island vegetative communities. Evaluated the subsequent effects on the physical processes of barrier islands and the effects grazing has on maritime forest, dune, and marsh structure and stability. Prepared an ecological management plan for the Shackleford Island horses that was submitted to the North Carolina Division of Coastal Management.

Marine Researcher: Designed and conducted toxicological experiments to evaluated the sublethal effects of herbicides and insecticides on estuarine crab species. Developed a method to quantify sublethal effects of contaminants on crab species. Results submitted to the United States Department of Energy.

Marine Researcher: Investigated the attachment of marine invertebrate larvae on laboratory and field surfaces using toxicological and behavioral studies. Contributed to the development of natural and synthetic compounds which inhibit invertebrate attachment to submerged surfaces and resulted in three patents for paint additives which inhibit invertebrate attachment.

Professional Affiliations:

Society of Environmental Toxicology and Chemistry (SETAC)
American Society of Testing and Materials (ASTM)

Additional Training and Licenses:

OSHA 29 CFR 1910.120 Hazardous Waste Operations and Emergency Response
Hazardous Waste Operations and Emergency Response Supervisor Training
Southwest Louisiana Health and Safety Training
First Aid, CPR, and Infectious Disease/Blood Borne Pathogens

Peer-Reviewed Publications:

Curry, C.L., P.S. Price, N.L. Bonnevie, T.B. Abel, and D.G. Gunster. 1995. Inter-lake variation in PCB bioaccumulation for the Great Lakes. *Proceedings of 1995 Water and Environment Federation: Toxic Substances in Water Environments.*

Algeo, E.R., J. Ducey, M.H. Henning, D.G. Gunster, and C. Schmidt. 1994. Workable ecological risk assessment guidance: selecting indicators species. Fifteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). Denver, Colorado. October 30-November 3.

Gunster, D.G. 1994. What is risk assessment and how is it used? Presentation to PPG, Inc., Lake Charles Facility Accounting Department. Lake Charles, Louisiana. February 18.

Gunster, D.G., N.L. Bonnevie, C.A. Gillis and R.J. Wenning. 1993. Assessment of chemical loadings to Newark Bay, New Jersey, from petroleum and hazardous chemical accidents from 1986-1991. *Ecotoxicology and Environmental Safety* 25: 202-213.

Gunster, D.G., C.A. Gillis, N.L. Bonnevie, T.B. Abel, R.J. Wenning. 1993. Petroleum and hazardous chemical spills in the Newark Bay, New Jersey, from 1982 to 1991. *Environmental Pollution* 82:245-253.

Gunster, D.G., M.N. Gray, and P.E. Goodrum. 1993. A Monte Carlo approach for examining chemical distribution in food webs. Fourteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). Houston, Texas. November 14-18.

Gunster, D.G., M.N. Gray, P.S. Price, and E.S. Ebert. 1993. A Monte Carlo approach for examining chemical distribution in food webs. Third Symposium on Environmental Toxicology and Risk Assessment: Aquatic, Plant, and Terrestrial. ASTM. Atlanta, Georgia. April 25-28.

Henning, M.H., D.G. Gunster, E.S. Ebert, J. Ducey, S.G. Martin, and J. W. Duncan. 1993. Population approach to assessing ecological impacts of contaminated sediments in a floodplain ecosystem. Annual Meeting for the Society for Risk Analysis, Savannah, Georgia. December 5-8.

Knight, J.W., D.G. Gunster, and P.S. Price. 1993. The role of predator (mink) feeding ranges in establishing soil/sediment cleanup standards. Fourteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). Houston, Texas. November 14-18.

Price, P.S., J.W. Knight, D.G. Gunster, and M.N. Gray. 1993. The role of predator (mink) feeding ranges in establishing soil/sediment cleanup standards. Third Symposium on Environmental Toxicology and Risk Assessment: Aquatic, Plant, and Terrestrial. ASTM. Atlanta, Georgia. April 25-28.

Sherman, W.R., R.E. Keenan, and D.G. Gunster. 1993. Reevaluation of TCDD bioconcentration and bioaccumulation factors. *Proceedings of 1993 TAPPI Environmental Conference*, pp. 325-333.

Gunster, D.G., W.R. Sherman, and R.E. Keenan. 1992. Reevaluating TCDD bioconcentration and bioaccumulation factors. Society of Environmental Toxicology & Chemistry (SETAC). Cincinnati, Ohio. November 9-13.

Bonnevie, N.L., D.G. Gunster, and R.J. Wenning. 1992. Distribution of lead in surficial sediments from

the Newark Bay estuary. *Environment International* 18:497-508.

Sherman, W.R., R.E. Keenan, D.G. Gunster. 1992. A reevaluation of dioxin bioconcentration and bioaccumulation factors for regulatory purposes. *Journal of Toxicology and Environmental Health* 37:211-229.

Wenning, R.J., Gunster, D.G., and N.L. Bonnevie. 1992. Nonpoint source loading of toxic chemicals to Newark Bay. Society of Environmental Toxicology & Chemistry (SETAC). Cincinnati, Ohio. November 9-13.

Wenning, R.J., M. Ungs, and D.G. Gunster. 1992. Statistical optimization of aquatic sampling programs. EPA Regional Risk Assessment Workshops, Workshop 5: Northeast States. Boston, Massachusetts. April 20-23.

Gunster, D. G. 1991. Limitations of using the toxic equivalency approach for establishing regulatory standards. Platform Presentation. Society of Environmental Toxicology and Chemistry (SETAC). Seattle, Washington. November.

Keenan, R.E., E.S. Ebert, D. Gunster, J. Knight, E.R. Algeo, M. Gray, N.W. Harrington, 1991. Critical risk assessment factors for establishing a water quality standard for 2,3,7,8-tetrachlorodibenzo-p-dioxin. *Proceedings Dioxin <91 -- The Eleventh International Symposium on Chlorinated Dioxins and Related Compounds.* Research Triangle Park, North Carolina, p. 97.

Ritchoff, D., D. Gerhart, and D. Gunster. 1989. Molecular meditation of settlement of selected invertebrate larvae. International Conference on Bioactive Compounds from Marine Organisms. Goa, India.

Pertinent Presentations:

Gunster, D.G., R. Blackburn, L. Scally, and W.J. Schwalbaum. 1997. Application of seepage meters to evalute expsoures from groundwater contaminants entering ponds. Eighteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). San Francisco, California. November 16-20.

Gunster, D.G. and N.L. Bonnevie. 1997. Petroleum and hazardous chemical spills in Newark Bay, New Jersey, USA from 1991 to 1995. Eighteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). San Francisco, California. November 16-20.

Truchon, S.P., T.J. Iannuzzi, L.W. Barnthouse, N.M. Shear, and D.G. Gunster. 1996. An ecological risk evaluation of sediment metal toxicity and bioavailability in a gulf coast estuary. Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC), North Atlantic Chapter. Newport, Rhode Island. April 26-27.

Dombrowski, F., D.G. Gunster, J. Tull, and P. Sheehan. 1995. A phased approach to structuring an ecological risk assessment at a RCRA Site. Society of Environmental Toxicology and Chemistry (SETAC), Second World Congress. Vancouver, British Columbia, Canada. November 5-9.

Gunster, D.G., M.H. Henning, E.S. Ebert, and R.E. Keenan. 1994. A population approach to assessing ecological impacts of contaminated floodplain soils. Fifteenth Annual Meeting of the Society of Environmental Toxicology and Chemistry (SETAC). Denver, Colorado. October 30-November 3.

TASK LEADER – FREEZE DRYING

Betsy Barrows **Battelle MSL**

Principal Research Scientist

Education:

B.A. Biology, Wheaton College, Norton, MA, 1978

Qualifications:

Ms. Barrows is a senior research scientist in Battelle's Marine Sciences Laboratory (MSL) Marine Chemistry group. Her experience in environmental sciences includes project management, analytical organic and inorganic chemistry, marine and freshwater toxicity testing, and field work. She currently serves as project/program manager on evaluations of sediment for dredge and disposal operations for the U.S. Army Corps of Engineers (USACE) and US EPA, which include conduct of water-column toxicity tests, benthic acute toxicity tests, bioaccumulation studies, and chemical analyses of sediment, water, and tissue samples. As project/program manager, Ms. Barrows is responsible for maintaining the primary point-of-contact with clients; developing and implementing data quality objectives and planning documents (SAPs and QAPPs); technical and cost proposal preparation; establishing work breakdown structures and monitoring project/program budgets; coordinating field, laboratory, and data management activities conducted by multiple organizations within Battelle as well as subcontractors.

Ms. Barrows is also manages analytical chemistry tasks associated with sediment contamination and water quality programs, particularly analysis of trace and heavy metals. Her responsibilities include all aspects of client service, proposal/cost quote preparation, monitoring sample progress through the laboratory, assessing data quality objectives, and project deliverables.

Research Experience:

Dredged Material Evaluation Programs

Program Manager: USACE-New York District's Kill van Kull Deepening Project, 1999 phase. In preparation for dredging Kill van Kull to a depth of 50 ft, this $1.2 million dredged material evaluation project involved collection of over 150 sediment core samples and biological testing and chemical analysis of sediment composites from 14 reaches of Kill van Kull, according to New York regional guidance and Green Book requirements.

Project Manager: metals analysis of dredged material from the Oakland 50-ft Deepening Project. Responsible for analysis of over 250 sediment samples for trace and heavy metals. Monitored sample throughput and delivery of sample data sets under 1-, 2-, and 4-week turnaround times and strict data quality requirements.

Program Manager: series of evaluations of dredged materials from federal waterways in New York and New Jersey, proposed for ocean disposal, for the USACE-New York District.

Project Manager: Evaluation the suitability of four candidate sites as potential reference sites for the USACE-New York District dredged material disposal program. Project involved sample collection and evaluation of sediments through bioassays and chemical analyses.

Project Manager: Revisions to the regional implementation manual for testing and disposal of dredged material for the USACE-New York District. The project also included two accompanying products -- a set of biological and chemical testing procedures, and a PC-based statistical software system designed to statistically evaluate dredged material toxicity and bioaccumulation testing data generated by applicants for ocean disposal permits.

Contributing Author: .Preparation of a standard methods manual for environmental sampling and analysis in San Francisco Bay, in support of the USACE-San Francisco District's Long Term Management Strategy Program for San Francisco Bay. Compiled, edited, and adapted analytical chemistry methods for the manual.

Work Assignment Leader: Evaluation of the suitability of four candidate sites as potential dredged material disposal sites for EPA Region II. Project involved conduct of a shipboard rapid bioassessment of benthic resources at candidate sites, collection of sediment and tissue samples for chemical analyses, and preparation of a series of literature searches and data reports. Participated in survey of the New York Bight in August 1992 and was contributing author to reports and documents.

Contributing Author: Series of technical reports and data summaries for a program to conduct ecological evaluations of proposed discharge of dredged material from Oakland Harbor into ocean waters for several district offices of the USACE-San Francisco District.

Author: Technical report on chemical and biological evaluations of sediment in the Pier 27 area for the Port of Seattle, Seattle, Washington. Results of evaluations were compared to Puget Sound Dredged Disposal Analysis (PSDDA) guidelines as well as to sediment quality standards drafted by the State of Washington Department of Ecology.

EPA Ocean Dumping Program

Participated in numerous projects under EPA's Ocean Dumping Program. Assisted EPA in the preparation of the following reports and documents:

- Report to Congress: *Ocean Disposal Monitoring Programs in Response to the Ocean Dumping Ban Act*
- Report to Congress: *Surveillance and Enforcement of Sewage-Sludge Dumping*
- Report to Congress: *Sludge Recycling Alternatives*
- Report to Congress: *Ocean Dumping*
- Report to Congress: *Progress in Stopping Ocean Dumping 1990*
- Newsletter/compliance report: *The ODBA Advocate* (April 1990, October 1990, April 1991, and October 1991 issues).

Participated in the 1987 baseline survey to track sewage sludge plumes and monitor dumping of sewage sludge at the 106-Mile Dumpsite. Performed shipboard extractions of 1000-L seawater samples for analysis of organic compounds.

Participated in development of EPA's research strategy and proposed regulations for ocean incineration. Work included responding to public comments on EPA's proposed regulation for ocean incineration; developing SOPs for sampling and analytical methods for the Ocean Incineration Research Burn Program (RBSA Plan); preparing work/quality assurance plans for analysis of environmental samples, and preparing final reports on analytical results of samples collected during baseline surveys to potential ocean incineration sites. Participated in 1987 baseline survey to potential ocean incineration sites along the Southeast Coast of the United States. Conducted pesticide, PCB, and petroleum hydrocarbon analyses on environmental samples collected during surveys to potential ocean incineration sites.

Workshops and Public Outreach Activities

Work Assignment Leader: Implementation of EPA Region III's Middle Atlantic Bight Initiative, an EPA-sponsored effort to join federal, state, and local agencies in discussion and prioritization of coastal issues. Project involved conducting a series of regional workshops in Maryland, Delaware and Virginia in January-April 1990; project also required management of subcontractors responsible for other project products, including preparation of a monograph compiling data on benthic ecology of the Dam Neck Disposal Site off the coast of Virginia, and development of a database for Region III's coastal aerial surveillance surveys.

Work Assignment Leader: Roundtable on alternative sludge disposal methods, November 1989, in East Rutherford, New Jersey. Conference was designed to assist New York and New Jersey ocean dumping sewerage authorities in the phase-out of ocean disposal of sludge and implementing land-based sludge disposal alternatives. Participated in conference organization and planning; prepared proceedings report.

Work Assignment Leader: Assisting EPA OMEP in management and development of a series of public outreach activities designed to enhance the public's awareness of the oceanographic and estuarine research conducted on the EPA's Ocean Research Vessel, the *Peter W. Anderson*. Public education materials resulting from this project included a report describing the goals and outcomes of EPA's scientific surveys conducted using the ship; a video geared toward a school-age audience portraying the ship's contributions to studying marine pollution; and a pictorial display with posters and brochures for distribution to ship visitors. Products of this assignment also included organizing a National Chief Scientists' Meeting, September 1990, in Narragansett, Rhode Island. Workshop was designed for EPA staff scientists to discuss technology transfer of marine sampling techniques, shipboard safety issues, and future direction of EPA's vessel, the *Peter W. Anderson*.

Participated in planning and organization of the Ocean Dumping Workshop 106-Mile Site, March 1989, in Ocean City, New Jersey; served as rapporteur during sessions.

Laboratory Experience

Conducted sample preparation and gas chromatographic analysis of marine environmental samples and aquatic toxicology. Prepares written standard operating procedures for analytical methods. As an assistant laboratory manager in the Organic Chemistry Laboratory at Battelle Ocean Sciences in Duxbury, Massachusetts, supervised technicians in sample preparation for analysis of pesticides, PCBs, and saturated and aromatic hydrocarbons in water, tissue, air and sediment samples and coordinated sample scheduling.

Conducted toxicity tests on pesticides and priority pollutants under federal guidelines for Good Laboratory Practices (GLP); conducted on-site bioassays of industrial wastewaters at paper mill and chemical plants.

Publications:

Gruendell, B.D., E.S. Barrows, and A.B. Borde. 1997. *Evaluation of Dredged Material Proposed for Ocean Disposal from Arthur Kill Project Area, New York*. PNNL-11478. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Gruendell, B.D., E.S. Barrows, and A.B. Borde. 1997. *Evaluation of Dredged Material Proposed for Ocean Disposal from Hackensack River Project Area, New York*. PNNL-11479. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Rosman, L.B., and E.S. Barrows. 1997. "Sediment Concentrations of pesticides and PCBs and Associated Laboratory-Measured Bioaccumulation in New York/New Jersey Waterways." PNNL-SA-29332. Presented at the 18th Annual meeting of Society of Environmental Toxicology and Chemistry (SETAC): "Bridging the Global Environment: Technology, Communication, and Education," November 16-20, 1997, San Francisco, California.

Antrim, L.D., W.W. Gardiner, E.S. Barrows, A.B. Borde. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Shark River Project Area.* PNNL-11351. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Antrim, L.D., M.R. Pinza, E.S. Barrows, W.W. Gardiner, J.J.S. Tokos, B.D. Gruendell, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Eastchester Project Area, New York.* PNNL-11232. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Antrim, L.D., M.R. Pinza, A.B. Borde, S.L. Nieukirk, E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Claremont Project Area, New York.* Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington. (Draft)

Barrows, E.S., L.D. Antrim, M.R. Pinza, W.W. Gardiner, N.P. Kohn, B.D. Gruendell, H.L. Mayhew, J.Q. Word, L.B. Rosman. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Federal Projects in New York and New Jersey and the Military Ocean Terminal (MOTBY).* PNNL-11280. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Barrows, E.S., B.D. Gruendell. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Gravesend Bay Anchorage, New York.* PNNL-11316. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington;

Pacific Northwest National Laboratory, Richland, Washington.

Barrows, E.S., H.L. Mayhew, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from MOTBY.* PNNL-11343. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Barrows, E.S., H.L. Mayhew, J.Q. Word, J.J.S. Tokos. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Port Chester, New York.* PNNL-11286. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Gardiner, W.W., E.S. Barrows, L.D. Antrim, B.D. Gruendell, J.Q. Word, J.J.S. Tokos. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Buttermilk Channel, New York.* PNNL-11287. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Gardiner, W.W., E.S. Barrows, L.D. Antrim, B.D. Gruendell, J.Q. Word, J.J.S. Tokos. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Hudson River, New York.* PNNL-11342. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Gardiner, W.W., E.S. Barrows, L.D. Antrim, B.D. Gruendell, J.Q. Word, J.J.S. Tokos. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from South Brother Island Channel, New York.* PNNL-11317. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest Laboratory, Richland, Washington.

Gardiner, W.W., E.S. Barrows, J.Q. Word. 1996. *Ecological Evaluation of Proposed Reference Sites in the New York Bight, Great South Bay, and Ambrose Light, New York.* PNNL-11361. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest Laboratory, Richland, Washington.

Gardiner, W.W., A.B. Borde, S.L. Nieukirk, E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Shoal Harbor / Compton Creek Project Area, New Jersey.* PNNL-11356. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Gruendell, B.D., W.W. Gardiner, A.B. Borde, S.L. Nieukirk, E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Bronx River Project Area, New York.* PNNL-11443. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Gruendell, B.D., M.R. Pinza, A.B. Borde, S.L. Nieukirk, E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Port Jersey Project Area, New York.* Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington. (Draft)

Pinza, M.R., E.S. Barrows, A.B. Borde. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Red Hook / Bay Ridge Project Areas, New York.* PNNL-11350. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Pinza, M.R., E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Liberty Island Anchorage, New York.* PNNL-11394. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest National Laboratory, Richland, Washington.

Pinza, M.R., W.W. Gardiner, A.B. Borde, S.L. Nieukirk, E.S. Barrows, J.Q. Word. 1996. *Evaluation of Dredged Material Proposed for Ocean Disposal from Westchester Creek Project Area, New York.* PNNL-11404. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest National Laboratory, Richland, Washington.

Rosman, L.B., and E.S. Barrows. 1996. "Spatial Distribution of PAHs and Associated Laboratory-Measured Bioaccumulation in New York/New Jersey Waterways." PNNL-SA-27593. Presented at the 17th Annual Society of Environmental Toxicology and Chemistry (SETAC) conference November 17 - 21, 1996, Washington, DC. Prepared by the U.S. Army Corps of Engineers, New York District, and Battelle Marine Sciences Laboratory, Sequim, Washington/Pacific Northwest National Laboratories, Richland, Washington.

Barrows, E.S., W.W. Gardiner, L.D. Antrim, B.D. Gruendell, J.Q. Word, J.J.S. Tokos. 1995. *Evaluation of Dredged Material Proposed for Ocean Disposal from South Brother Island Channel, New York.* Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

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Ward, J.A., J.Q. Word, A.D. Uhler, E.S. Barrows, V.I. Cullinan, R. Cuello, D.K. Niyogi. 1995. *Suggested Methods for Environmental Sampling and Analysis in San Francisco Bay.* Volumes 1-3. PNL-10565. Prepared for the U.S. Army Corps of Engineers, San Francisco District, in support of the Long-Term Management Strategy Program for San Francisco Bay, by Battelle/Marine Sciences Laboratory, Sequim, Washington; Pacific Northwest Laboratory, Richland, Washington.

Barrows, E.S., M.R. Pinza, W.W. Gardiner, N.P. Kohn, B.D. Gruendell, H.L. Mayhew, J.Q. Word, L.B. Rossman. 1994. *Evaluation of Dredged Material Proposed for Ocean Disposal from Federal Projects in New York and New Jersey and the Military Ocean Terminal (MOTBY).* Draft. Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

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Gardiner, W.W., E.S. Barrows, J.Q. Word. 1994 (in press). *Ecological Evaluation of the 0-2 Mile Reach and the 2-4 Mile Reach of the Raritan River, New York.* Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Gardiner, W.W., E.S. Barrows, J.Q. Word. 1994 (in press). *Ecological Evaluation of Proposed Reference Sites in the New York Bight and Great South Bay, New York.* Prepared for the U.S. Army Corps of Engineers, New York District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Kohn, N.P., E.S. Barrows, J.Q. Word, B.D. Gruendell, and L.B. Rosman. 1993. "Ammonia Levels in Sediment Toxicity Tests with the Marine Amphipods *Ampelisca abdita* and *Rhepoxynius abronius*." Presented at the 14th Annual Society for Environmental Toxicology and Chemistry (SETAC) meeting in Houston, Texas, November 15-18, 1993. Prepared by Battelle/Marine Sciences Laboratory, Sequim, Washington, and the U.S. Army Corps of Engineers, New York District, New York, New York.

Kohn, N.P., J.A. Ward, H.L. Mayhew, J.Q. Word, E.S. Barrows, S.M. Goodwin, L.F. Lefkovitz. 1992. *Ecological Evaluation of Proposed Discharge of Dredged Material from Oakland Harbor into Ocean Waters (Phase III B of -42-Foot Project).* PNL-8174. Prepared for the U.S. Army Corps of Engineers, San Francisco District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Pearson, W.H., E.S. Barrows, W.W. Gardiner, J.Q. Word. 1992. *Rapid Bioassessment of Benthic Resources of Potential Disposal Areas in the New York Bight, Summer Survey, August 1992.* Prepared for the U.S. Environmental Protection Agency, Oceans and Coastal Protection Division, Washington, D.C., EPA Contract No. 68-C8-0105, Work Assignment 4-301, by Battelle/Marine Sciences Laboratory, Sequim, Washington, under contract to Battelle Ocean Sciences, Duxbury, Massachusetts.

Pearson, W.H., E.S. Barrows, J.Q. Word. 1992. *Qualitative Assessment of Benthic Infauna Resources in Four Areas of the New York Bight.* Prepared for the U.S. Environmental Protection Agency, Oceans and Coastal Protection Division, Washington, D.C., EPA Contract No. 68-C8-0105, Work Assignment 3-301, by Battelle/Marine Sciences Laboratory, Sequim, Washington, under contract to Battelle Ocean Sciences, Duxbury, Massachusetts.

Pinza, M.R., J.Q. Word, E.S. Barrows, H.L. Mayhew, R.W. Bienert. 1992. *Evaluation of the Southwest Pass of the Mississippi River Ocean Dredged Material Disposal Site and Channel.* Prepared for the U.S. Environmental Protection Agency, Oceans and Coastal Protection Division, Washington, D.C., and Region VI, Dallas, Texas, under EPA Contract No. 68-C8-0105, Work Assignment No. 3-305, by Pacific Northwest Laboratory, Richland, Washington.

Pinza, M.R., J.Q. Word, E.S. Barrows, H.L. Mayhew, R.M. Ecker, D.R. Clark. 1992. *Snake and Columbia River Sediment Sampling Project.* PNL-8479. Prepared for the U.S. Army Corps of Engineers, Walla Walla District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Ward, J.A., J.Q. Word, M.R. Pinza, H.L. Mayhew, E.S. Barrows, N.P. Kohn, L.K. Lefkovitz. 1992. *Ecological Evaluation of Proposed Discharge of Dredged Material from Oakland Harbor into Ocean Waters (Phase III of 38-Foot Project).* PNL-7890. Prepared for the U.S. Army Corps of Engineers, San Francisco District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Ward, J.A., J.Q. Word, M.R. Pinza, H.L. Mayhew, E.S. Barrows, L.F. Lefkovitz. 1992. *Ecological Evaluation of Proposed Discharge of Dredged Material from Oakland Harbor into Ocean Waters (Phase III A of -42-Foot Project).* PNL-8302. Prepared for the U.S. Army Corps of Engineers, San Francisco District, by Battelle/Marine Sciences Laboratory, Sequim, Washington. Published by Pacific Northwest Laboratory, Richland, Washington.

Smolski, E.M., L.F. Lefkovitz, R.M. Thom, J.Q. Word. 1991. *Port of Seattle Pier 27 Report on Field*

Sampling, Sediment Chemistry, and Biological Analyses. Prepared for the Port of Seattle, Seattle, Washington, by Battelle/Marine Sciences Laboratory, Sequim, Washington.

Battelle. 1989. *Development, Testing, and Validation of the Marine Incineration Biological Assessment Sampler.* Final report to U.S. EPA Office of Marine and Estuarine Protection, Washington, DC. (Contributing author).

Sauer, T., J. Brown, G. Durell, E. Smolski, L. Antrim, J. Buck, D. Lewis, H. Trulli, J. Droppo, J. Hardy, P. Boehm, and C. Werme. 1988. *Analytical Results of Samples and Data Collected During the Baseline Survey to the Existing and Alternative Gulf of Mexico Incineration Sites.* Final report to U.S. EPA Office of Marine and Estuarine Protection, Washington, DC.

Sauer, T.C., W.M. Cooke, E.M. Smolski, and P.D. Boehm. 1987. *Analytical Results of Samples Collected During the 1985 U.S. Southeast Coast survey.* Final report to U.S. EPA Office of Marine and Estuarine Protection, Washington, DC.

Sauer, T.C., W.M. Cooke, E.M. Smolski, D.P. Redford, and P.D. Boehm. 1987. *Analytical Results of Samples Collected During the 1985 North Atlantic Incineration Site (NAIS) Survey.* Final report to U.S. EPA Office of Marine and Estuarine Protection, Washington, DC.

QUALITY ASSURANCE OFFICER

Rosanna L. Buhl **Battelle Duxbury**

Quality Systems Manager

Education:

ASQ Certified Quality Auditor (1996)
Occupational and Environmental Radiation Protection, Harvard School of Public Health (1993)
Quality Assurance for the Analytical Chemistry Laboratory (1991)
Good Laboratory Practice Standards, Center for Professional Advancement (1989)
Quality Assurance Basic Training course, Society of Quality Assurance (1989)
Bridgewater State College (also Philadelphia College of Bible and Bob Jones University)

Qualifications:

Ms. Buhl joined Battelle in 1973 and brings a broad background of technical experience to her present position as Quality Systems Manager. With 10 years' experience in the biology/biochemistry laboratories she was appointed study director for aquatic toxicology tests conducted according to Good Laboratory Practice (GLP) standards and served in that capacity for five years. In 1989, Ms. Buhl joined the Quality Assurance Unit and was the site Quality Assurance Coordinator for six years until her appointment to the position of Quality Systems Manager in 1998. She has received formal QA instruction through the Center for Professional Advancement, the Society of Quality Assurance, and American Society for Quality (ASQ).

As Quality Systems Manager for Battelle, Ms. Buhl is responsible for overseeing all site quality assurance activities, verifying that technical activities are conducted in compliance with the site Quality Management Plan (QMP), and managing the technical activities of the Battelle Duxbury Operations' Quality Assurance Office.

Ms. Buhl develops and implements the quality management system at Battelle, and is responsible for preparation of the site QMP, for preparing and reviewing standard operating procedures, identifying and implementing staff training, conducting laboratory inspections of both Battelle and subcontractor activities, and verifying reported data through data audits. Ms. Buhl coordinates, supervises, and participates in all QA activities associated with the collection of environmental data. She has extensive knowledge of state and Federal QA programs, and has written Quality Management Plans for client and regulatory approval.

Professional Positions:

Quality Systems Manager, Quality Assurance Unit, Battellle. 1998 - present. Oversees the implementation of the Quality Assurance Program at Battelle and in particular oversees the activities of the Quality Assurance Unit, report production, and information services.
Quality Assurance Coordinator, Quality Assurance Unit, Battellle. 1992 - 1998. Responsible for management of the Quality Assurance Unit activities.

Quality Assurance Specialist, Quality Assurance Unit, Battelle. 1989 - 1992. Responsible for performing data audits, laboratory inspections, and administration of standard operating procedures.

Researcher, Environmental Toxicology, Battelle. 1984 - 1989. Study director for toxicology testing under GLPs and related analyses, including analytical HPLC.

*Technican/Research Technician,*Biology, Battelle. 1973 - 1983. Various assignments: benthic ecology technician and polychaete taxonomist; biochemistry lab technician for analytical HPLC and specialized instrumentation.

Relevant Experience:

As *Quality Systems Manager* Ms. Buhl prepares the site Quality Management Plan, the Quality Assurance Manual that describes the analytical laboratory's procedures and quality control requirements, and she is responsible for maintaining the laboratory's State certifications.

As *Program QA Officer* for the EPA Oceans and Coastal Protection Division contract Ms Buhl prepared the original Program Quality Management Plan and is responsible for the annual review and revision of this document. She has also prepared Quality Assurance Program Plans for a large multidisciplinary field and laboratory study conducted for the Massachusetts Water Resources Authority (MWRA).

Program QA Officer: Ms. Buhl is responsible for scheduling and conducting quarterly laboratory inspections at Battelle, and of field and subcontractor laboratories activities as necessary to assess compliance with QA Program Plans, SOPs, and regulatory requirements, and for preparing written reports to management. Ms. Buhl conducts routine inspections of analytical activities conducted in the Chemistry laboratory for GLP studies.

Program QA Officer: Ms. Buhl has a wide variety of experience in support of extrinsic inspections conducted at Battelle by EPA, State agencies, clients, third-party auditors, and the Nuclear Regulatory Agency. She coordinates preparation, accompanies inspectors during the inspection, assists laboratory personnel in corrective action to audit findings, and prepares written reports of findings to management and written responses to inspection reports. Recent inspections have included two client inspections related to Battelle's analysis of samples for Superfund site. Battelle was audited twice by Exxon Corporation during the analysis of samples collected after the *Exxon Valdez* oil spill. Ms. Buhl coordinated all laboratory activities associated with the inspections.

Program QA Officer: In addition to the planning and inspection phases of each project, a major role of the QA Office at Battelle is the verification that all reported data are accurate, traceable, and collected according to the project QA plan and associated SOPs. Ms. Buhl coordinates and participates in this essential activity. Her expertise ranges from the audit of analytical chemistry and biology data, to verification of data contained in database systems. She functions as the QA Officer for all projects and is the lead auditor for many. She participated in CLP-level data validation of environmental monitoring data collected at the Pantex Plant in Amarillo, TX. Because Battelle offers the capability to conduct large, multidisciplinary studies, like the EPA Office of Water investigations at the 106-mile dump site, Ms Buhl is skilled in verifying large and complex data sets, including sediment trap, settling rate, microlayer, productivity, and *in situ* electronically-captured oceanographic data. In support of GIS development for the Resource Conservation Recover Act (RCRA) Facility Investigation (RFI) for three divisions of United Technologies, Ms. Buhl conducts audits of database contents prior to inclusion in the client database.

Program QA Officer for Battelle's task order technical services contract with the EPA's Oceans and Coastal Protection Division (OCPD). In this role Ms. Buhl reviews all work assignment QAPjPs for adequacy, evaluates the QA programs of subcontractors, supervises audits of all reported data, and reviews technical deliverables.

Program QA Officer for Battelle Columbus' EPA NRMRL contract. She is responsible for the QA review of QAPPs produced in compliance with the requirements of EPA's Risk Reduction Engineering Laboratory.

Task Leader responsible for preparing the Quality Management Plan for the Gulf of Mexico Program Office. This effort involved interviewing numerous State and Federal agencies to identify common ground for establishing a program-wide quality assurance program.

Program QA Officer for the Harbor and Outfall Monitoring program conducted by Battelle for the Massachusetts Water Resources Authority. Seven separate Quality Assurance Program Plans for the 7 monitoring tasks are being prepared for this program, which included extensive field activities, *in situ* water quality measurements, sample collection for laboratory analyses, extensive analytical chemistry and nutrient analyses, and the development of a client-compatible database. This assignment includes the oversight of several offsite subcontractors to ensure that their QA/QC procedures meet the program requirements.

Program QA Officer for Newtown Creek Facilities Upgrade program conducted by Battelle for the City of New York. Five separate Quality Assurance Program Plans for the 5 monitoring tasks were prepared for this program which was conducted in support of NPDES permit revision. Certification by the New York Department of Health Environmental Laboratory Approval Program was required, The program consisted of a rigorous field program of 100 stations visited 10 times during a one-year period. Nutrient, water quality measurements, productivity, and discharge monitoring data were entered into a modeling database.

Project QA Officer for the Ecological and Human Health Assessment for the Trecate 24 Oil Spill in Northern Italy study conducted for a major European oil company. Chemical analysis of sediments, soils, oils, plant, and animal tissues are being conducted and entered into a client-compatible database. The damage assessments are being performed according to U.S. Natural Resource Damage Assessment Guidelines.

Project QA Officer for the Broadkill Beach Interim Feasibility Study conducted for the U.S. Army Corps of Engineers, Philadelphia District. Battelle evaluated the physical characteristics and benthic biological resources at potential fill borrow sources, analyzing benthic infauna samples and associated data. These data were used to identify the abundance and distribution of rare and commercial species that may be impacted by sediment removal activities.

Program QA Officer for the program Monitoring the Fate and Effects of the *Exxon Valdez* Oil Spill in Prince William Sound, Alaska conducted for Exxon Corporation. This project involved long-term natural resource damage assessment (NRDA) studies and assistance with shoreline treatment operations. Battelle provided program design and management, field support, analytical support, data interpretation, and database management for more than 20 studies associated with the *Exxon Valdez* oil spill.

Program QA Officer for Battelle's contract with NOAA for the Period 2 Status and Trends Mussel Watch Program, a program involving the collection of bivalve molluscs and surficial sediment at estuarine/coastal sites on the each and west coasts of the United Sates, and the analyses of these samples for selected organic and inorganic analytes.

QA Auditor for Analytical Chemistry Services in Support of Remedial Investigation Projects at U.S. Navy Sites in Hawaii and Guam conducted for the U.S. Navy Pacific Division/Ogden Environmental Inc. Sediment and tissue samples collected in support of the Navy CLEAN program were processed and analyzed for PAH, PCB, chlorinated pesticides, petroleum hydrocarbons, trace elements, and butyltins. Performance on this contract required NEESA certification The certification process included review and approval of a laboratory quality assurance plan and all SOPs used on the project, successful analysis of several laboratory performance evaluation samples, and successful completion of a site audit by Navy inspectors.

Researcher/Research Technician. Ms. Buhl has extensive experience in conducting aquatic toxicity tests according to the stringent requirements of both TSCA (40 CFR Part 720) and FIFRA (40 CFR Part 160). She has been the GLP study director for over 15 studies with overall responsibility for the technical conduct of each study, as well as data interpretation, analysis, documentation, and reporting. Due to her success in conducting several tests for a Fortune 500 Chemical Company, and no findings during related EPA inspections, she was requested specifically to oversee all testing for this client. Ms. Buhl served as polychaete taxonomist and as the laboratory technician for a nationally-recognized research team, assisting in the development of innovative applications of biochemical and physiological techniques as indicators of sublethal stress in marine organisms.

Professional Affiliations:

National Environmental Laboratory Accreditation Conference; On-Site Assessment Committee
ASQ Training
Society for Quality Assurance
New England Regional Chapter Society for Quality Assurance

Additional Training and Licenses:

ASQ Training
Society for Professional Advancement Training
Society for Quality Assurance Training

Peer-Reviewed Publications:

Breteler, R.J., J.W. Williams, and R.L. Buhl. 1982. Measurements of chronic toxicity using the opossum shrimp Mysidopsis bahia. Hydrobiolgia 93:189-194.

Carr, R.S., J.W. Williams, F.I. Saksa, R.L. Buhl, and J.M. Neff. 1985. Bioenergetic alterations correlated with growth, fecundity, and body burden of cadmium for mysids (Mysidopsis bahia). Envir. Toxicol. Chem. 4:181-188.

Neff, J.M., R.E. Hillman, R.S. Carr, R.L. Buhl, and J.I. Lahey. 1987. Histopathologic and biochemical responses in arctic marine bivalve molluscs exposed to experimentally spilled oil. Arctic 40 (Suppl. 1): 220-229.

Breteler, R.J., Buhl, R.L., and Maki, A.W., The effects of dissolved H_2S and CO_2 on short-term photosynthesis of Skeletonema costatum, a marine diatom. Plants for Toxicity Assessment: Second Volume, ASTM STP 1115, J.W. Gorsuch, W.R. Lower, W. Wang, and M.A.Lewis, Eds., American Society for Testing and Materials, Philadelphia, 1991. pp 118-125.

QUALITY ASSURANCE OFFICER

Deborah Coffey **Battelle MSL**

Senior Quality Engineer

Education:

M.S. Resource Ecology, School of Natural Resources, University of Michigan , 1983
B.S. Biology, University of Michigan-Flint, 1977

Qualifications:

Ms. Coffey brings to Battelle over 16 years of quality assurance (QA) experience. Her current responsibilities include serving as the primary contact for client QA staff and providing Quality Engineering (QE) assistance for implementing QA Program requirements at the Marine Sciences Laboratory in Sequim, Washington. She assures implementation of QA Program requirements by assisting project personnel in developing QA Project and QA Program Plans, reviewing procurement documents and technical procedures, assisting in the resolution of QA system problems, promoting continuous improvement practices, managing the controlled documents guiding analyses and overall QA Program implementation, planning and performing QA training, performing quality verification activities such as internal and supplier assessments and audits, and reviewing final data and reports. Ms. Coffey has five years experience as an NQA-1 certified lead auditor. She has participated in well over 175 audits.

Prior to Battelle, Ms. Coffey served four years as Deputy QA Manager for the Waste Isolation Pilot Plant (WIPP) project managed by Sandia National Laboratories, Albuquerque, New Mexico. She also provided nearly ten years of QA support for a wide variety of research projects conducted by the EPA's Environmental Research Laboratory, Corvallis, Oregon. These projects included assessing the regional health of streams, lakes, soils and forest; assessing the effect of pollutants on plants, animals, forests and the global climate; and assessing the affects of the introduction of genetically engineered organisms. As an Associate Chemist at Tetra Tech, Bellevue, Washington, Ms. Coffey contributed to data collection and analysis for EPA Superfund projects and NPDES permitting. She has also served as Director of an analytical laboratory managed jointly by the USDA Forest Service and Oregon State University in Corvallis, Oregon, which focused on the analysis of low-level surface water samples. In this position, Ms. Coffey conducted field sampling at Spirit Lake, Washington, following the eruption of Mount Saint Helens and analyzed samples from Crater Lake in support of U.S. Army Corps of Engineers research and the Long Term Ecological Monitoring (LTER) project site in the H.J. Andrews Experimental Forest, near Blue River, Oregon.

During her career, Ms. Coffey has designed and implemented QA programs for projects meeting CERCLA, RCRA, NQA-1, EPA and GLP requirements. Working with scientists, Deborah Coffey has implemented QA requirements in a variety of projects including disposal of ocean dredged material, site characterization and feasibility studies, biological studies associated with oil spills, and environmental characterizations, including field sampling, biological toxicity testing and analytical chemistry. In many of these projects, her participation began with project design and extended to the end of the project and into the publication phase of the work.

Relevant Experience:

Lead Role in obtaining and maintaining State accreditations for the following states: Washington, New Jersey, South Carolina, Florida, and Wisconsin. Part of this role includes assisting in the Laboratory's participation in Performance Evaluation Sample programs.

Implementation and Periodic Review And Revision of the Marine Sciences Laboratory (MSL) Quality Assurance Management Plan. Responsible for the annual review and revision of the over 100 MSL operating procedures and maintenance of the controlled document system.

QA Representative on Superfund, site characterization, and environmental risk assessment projects.

Developed a Good Laboratory Practice Program for studies evaluating effects of pollutants on wildlife. Served as the Quality Assurance Unit and was responsible for monitoring each study to assure management that the facilities, equipment, personnel, methods, practices, records, and controls were in conformance with regulations.

Worked on Numerous Programs/Projects dealing with the following nationally recognized QA programs:

- Department of Energy Order 5700.6C, Quality Assurance
- QAMS-005/80, Guidelines and Specifications for Preparing EPA Quality Assurance Project Plans
- EPA (40 CFR Part 160, 40 CFR Part 792) and FDA (21 CFR Part 58) Good Laboratory Practices regulations
- ASME NQA-1, Quality Assurance Program Requirements in Nuclear Facilities
- 10CFR, Appendix B, Quality Assurance Criteria for Nuclear Power Plants and Fuel Processing Plants
- IEEE 730-1984, Software Quality Assurance.

Publications:

Erickson, H.E., and D.S. Coffey. 1991. "The Watershed Manipulation Project: Case Study of an Interlaboratory Program." *Accountability in Research*, 1:195-206.

Lee, J., R. Church, D. Lammers, L. Liegel, M. Johnson, D. Coffey, R. Holdren, D. Stevens, R. Turner, L. Blume. 1989. "Watershed Surveys to Support an Assessment of the Regional Effects of Acidic Deposition on Surface Water Chemistry." *Environmental Management*, 13(1):95-108.

Coffey, D.S., J.C. Sprenger, D.T. Tingey, G.E. Neely, and J.C. McCarty. 1988. "National Crop Loss Assessment Network: Quality Assurance Program." *Environmental Pollution*, 53:89-98.

TASK LEADER: TISSUE CHEMISTRY

Deirdre Dahlen **Battelle Duxbury**

Research Scientist

Education:

B.S. Bates College, 1987

Qualifications:

Ms. Dahlen has over 13 years of professional experience in the field of environmental chemistry and specializes in the analysis of environmental samples for trace organic contaminants. Since joining Battelle, Ms. Dahlen has managed a variety of monitoring studies involving both terrestrial and aquatic environments. She also has extensive experience with development and implementation of quality control programs for laboratory studies and oversight of methods development studies. Currently Ms. Dahlen is managing chemistry tasks related to Battelle's contract with the U.S. Army Corps of Engineers/New England District. She has also acts as a project manager/analytcal task leader for a variety of government (EPA, Navy) and industrial monitoring programs. In this role, Ms. Dahlen coordinates field efforts, oversees conduct of chemical analyses, monitors data quality, interprets sample results, oversees subcontractor activities and prepares final reports.

Professional Positions:

Chemistry Laboratory Manager, 1995. While at Battelle, Ms. Dahlen completed a one-year assignment as manager of the chemistry laboratory. She scheduled all analytical work, directly oversaw 17 technical staff, and was ultimatlely responsible for the quality of data generated in the chemistry facility.

Chemistry Laboratory Supervisor, 1992-1995. Prior to managing the chemistry laboratory, Ms. Dahlen served as the supervisor of the chemistry sample preparation laboratory. She was responsible for scheduling all analytical work in the sample preparation laboratory and directly oversaw 8 technical staff. Ms Dahlen was also responsible for development and implementation of quality control and technical training programs.

GC/MS Analyst, 1988-1992. Ms. Dahlen was responsible the the analysis of marine tissues, sediments, soils, dredged material, ocean water, wastewater and sludges for a variety of environmental contaminants including polynuclear aromatic hydrocarbons, linear alkyl benzenes and petroleum biomarkers.

Chemistry Laboratory Technician, 1987-1988. Ms. Dahlen was responsible for the preparation (extraction, cleanup) of environmental samples for trace organic analysis.

Relevant Experience:

Project Manager, for U.S. Army Corps of Engineers/New England District Delivery Order #41, Cochato River Sampling and Analysis. Sediment and fish samples were collected at five locations along the Cochato River in Holbrook, MA and near the former superfund site of Baird & McGuire. The objectives of this study were to 1) collect and analyze sediment and fish tissue samples for chemical analyses, 2) evaluate the spatial distribution of contaminants in the study area, 3) evaluate the correspondence within bulk sediment properties and against contaminants, 4) evaluate the temporal response of contaminants in sediment and fish, and 5) evaluate the temporal response of physiological conditions in fish. Prior to sampling activities, a project quality assurance project plan (QAPP) was prepared following Region 1, EPA-New England Compendium of Quality Assurance Project Plan Guidelines. Sediment samples were analyzed for grain size, TOC, metals, VOAs, chlorinated pesticides and PAH. Fish samples (whole body, fillet, offal) were analyzed for pesticides and PAH. Reconstructed whole body concentrations of organic contaminants were also determined from fillet and offal results. Data and interpretive reports were prepared to address the study objectives listed above.

Project Manager, for WA 2-51. Shellfish samples collected from Raritan Bay, NJ were analyzed for organic and metals contaminants in support of a human health assessment study. Shellfish samples were prepared in the laboratory and analyzed for 107 PCB congeners, PAH and metals. PCB data were evaluated to determine the distribution of PCB congeners by chlorination level. PCB distribution patterns were also examined to determine resemblance to known Aroclor patterns. The distribution of PAHs were examined to identify dominant PAHs detected in the shellfish samples. Analytical data were used by EPA and NJ DEP to determine whether a human health threat still exists from consumption of shellfish in the Rartan and Sandy Hook Bay areas.

Task Leader, Analytical Chemistry, for WA 133. Sediment and resident polychaete species were collected within the New York Bight Dredged Material Disposal Site, or Mud Dump Site (MDS) and historic disposal areas adjacent to the MDS. Sediment and tissue samples were prepared in the laboratory and analyzed for chemical, physical, and biological parameters. Chemical parameters included PAH, Pesticides/PCBs, organotins, major and trace elements, AVS/SEM, and dioxin/furan analyses. Physical parameters included TOC and sediment grain-size analyses. Biological parameters included performance of acute toxicity tests on sediments using the amphipod *Ampelisca abdita* and benthic infauna abundance and composition.

The objectives of this study were to 1) characterize the sediments within the MDS and in the 16-square-mile area surrounding the site, 2) determine the areal extent of each sediment characteristic, 3) determine which sediments were degraded, nondegraded, or marginal, 4) review published literature and other historic studies of the MDS and environs and compare chemical and physical parameters where applicable, and 5) define background contaminant concentrations (body burden) in polychaete tissues and identify the relationship between contaminant concentrations in bulk sediment and concentrations in polyuchaete tissues at the Study Area.

Project Manager for OOC. The bioaccumulaton potential in marine organisms (fish, crustaceans, and bivalves) of selected inorganic and organic compounds associated with discharges of produced water in the marine enviornment were addressed in a long-term monitoring study within the Gulf of Mexico. Tissue and water samples were collected in three sampling events over a two-year period. After each sampling event, samples were shipped to the laboratory for tissue processing and sample analyses.

Tissue processing activities included sectioning, compositing, and aliquoting all tissue samples for analysis of selected volatile organic compounds (VOC), semivolatile organic compounds (SVOC), metals, and radiochemical compounds by Battelle and other laboratories. Sample analysis activities included the preparation of water and tissue samples for target VOCs and SVOCs.

Standard EPA methodologies were not available for the analysis of VOCs in tissue by purge and trap GC/MS. A literature search was performed and method validation studies carried out to develop and optimize the purge and trap GC/MS method. Standard EPA methodologies were not available for the extraction, cleanup, and analysis of target SVOCs (phenol and bis(2-ethylhexylphthalate) in tissue. As was the case with the VOC method, Battelle performed literature searches and devised method validation studies to optimize sample extraction and cleanup methods for this study.

Task Leader, Analytical Chemistry, for environmental assessment for the *Exxon Valdez* oil spill. Working for Exxon USA, was a team member of the first group to immediately study the spill. Responsible for laboratory analysis of sediment, water, and tarball samples for petroleum product identification. Used sensitive analytical techniques, including triterpane and sterane biomarker measurement, to determine the distinct signature in samples and representative petroleum products including North Slope Crude Oil.

Task Manager, Analytical Chemistry, for the U.S. Army Corps of Engineers dredge material disposal study in San Francisco Bay; New York Harbor; Wilmington Bay, NC; Charleston Harbor, SC; and Panama City, FL. Working with Battelle team to implement USEPA/USACE "Green Book" dredge material testing, has been responsible for analysis of over 1000 water, sediment and marine tissue samples for polynuclear aromatic hydrocarbons, pesticides/PCBs and TBT compounds. Directed chemical analyses, quality control, and electronic reporting.

Additional Training and Licenses:

Hewlett-Packard Training: 1) Fundamentals of Gas Chromatography, 2) RTE-A GC/MS Operation, 3) Advanced RTE-A Operation, 4) RTE-A Procedural Files (macro writing)

Environmental Applications of GC/MS, Indiana University

Peer-Reviewed Publications:

Douglas, G.S., K.J. McCarthy, D.T. Dahlen, J.A. Seavey, W.G. Steinhauer, R.C. Prince, and D.L. Elmendorf. 1992. The Use of Hydrocarbon Analysis for Environmental Assessment and Remediation. *J. Soil Contamination.* 1(2): 197-216. Also in *Contaminated Soils.* (Calabrese, E.J. and Kostecki, P.T., Eds.) Chelsea, MI, Lewis Publishers, Chapter 1. pp. 1-21.

Douglas, G.S., K.J. McCarthy, D.T. Dahlen, J.A. Seavey, W.G. Steinhauer, R.C. Prince, and D.L. Elmendorf. 1991. "Hydrocarbon Fingerprinting Analysis – Fact or Fiction." In: Proceedings of the Conference on Hydrocarbon Contaminated Soils: Analysis, Fate, Environmental and Public Health Effects, University of Massachusetts, Amherst. September 1991.

Douglas, G.S., K.J. McCarthy, D.T. Dahlen, J.A. Seavey, and E.L. Butler. 1991. "Fingerprinting Petroleum Hydrocarbons in Water." Invited paper presented at the 14th Annual EPA Conference on Analysis of Pollutants in the Environment, Norfolk, VA. May 1991.

Peven, C.S., W.G. Steinhauer, L.M. Altshul, D.T. Dahlen, J.A. Seavey. 1990. Polycyclic Aromatic Hydrocarbons in Shellfish, Overview of the 1989 Mussel Watch Field Season (Abstract). *J. Shellfish.* 8:487.

Wenzel, T.J., L.M. Collette, D.T. Dahlen, S.M. Hendrickson, and L.W. Yarmaloff. 1988. "Liquid Chromatographic and Flow Injection Analysis of Tetracycline Using Sensitized Europium (III) Luminescence Detection." *J. Chromat.* 433:149-158.

Pertinent Presentations:

Society of Environmental Toxicology and Chemistry (SETAC). 1995. *Petroleum-Specific Analytical and Interpretive Techniques for Product Identification and Source Allocation*

SETAC. 1994. *Ultratrace Analysis of Organic Contaminants in Sediments, Water, and Tissues from Marine Superfund Site*

Dahlen, D., Lefkovitz, L, Hunt, C. 2000. *Sediment Contaminant Monitoring in Massachusetts and Cape Cod Bays.* In Trulli, H.R., Coniaris, C., Carroll, S.R. (Eds.). Outfall Monitoring Science Advisory Panel Technical Workshop 1999 [compact disc]. Boston: Massachusetts Water Resources Authority. Report ENQUAD ms-57.

PROGRAM MANAGER

Karen Foster **Battelle Duxbury**

Program Manager

Education:

M.S. Oceanography, University of Rhode Island, 1982
B.S. Biology, University of Connecticut, 1979

Qualifications:

Ms. Foster is a senior project manager specializing in fishery resource and environmental impact projects. She has more than 10 years' experience in fishery resource assessment and monitoring. Ms. Foster has used this expertise in preparing Environmental Impact Statements, conducting field studies to evaluate the feasibility of using artificial reefs as mitigation for habitat loss due to dredged material disposal, and preparing National Environmental Policy Act Section 7 biological assessments to identify impacts to marine mammals. She also is the program manager for Battelle's five-year task order contract for the U.S. Army Corps of Engineers, New England District.

Professional Positions:

Fishery Biologist, Research, Conservation and Utilitization Division, Department of Commerce, National Oceanic and Atmospheric Administration, National Marine Fisheries Service. 1987 to 1989. Responsible for assessing the status of specific groundfish and shellfish species stocks, including statistical analysis, developing sampling strategy, preparing annual stock assessment reports.

Fishery Biologist, Conservation and Utilitization Division, Department of Commerce, National Oceanic and Atmospheric Administration, National Marine Fisheries Service 1982 to 1987. Assist senior scientist in conducting assessments of fishery resources in the Northwest Atlantic using statistical methods and modeling.

Relevant Experience:

Program Manager for the USACE, New England District, 5-year task-order fixed-price contract. Under this contract, Ms. Foster, oversees the conduct of all tasks issued under this contract. She selects Battelle stafff and subcontractors with the appropriate technical and management skills to manage the task. She directly manages subcontractors conducting task orders. All task budgets are prepared and negotiations conducted under her direction. Ms. Foster is responsible for ensuring that all task are completed within the allocated budget, and working with the project managers to identify deviaitons from the scope of work which require additional budget. The client has given Battelle excellent scores for management of this contract under Ms. Foster.

Project Manager for preparation of programmatic baseline assessments for the U.S. Coast Guard operations in the Gulf of Mexico (District X), Alaska (District X), along the Pacific Coast (Districts X,X). Ms. Foster managed this project which required conducting a programmatic review of the U.S. Coast Guard Operations along the coastal regions of the United States to identify potential conflicts with the Endangered Species Act, National Environmental Policy Act, and Marine Mammal Protection Act. This review included investigating all vessel, aircraft, and facilities operations that operate along the coast or in the coastal waters. In addition, Ms. Foster was involved in the identification of all biological resources habitating the coastal and marine waters that could be at risk due to USCG operations. These baseline assessments will serve as the basis for developing environmental assessments or environmental impact statements, if necessary.

Project Manager and author of several project and federal program-related Biological Assessments. Ms. Foster has prepared two biological assessments on proposed projects in Cape Cod Bay, a designated right whale critical habitat. One of the biological assessment involved the construction and operation of a sea scallop aquaculture facility in Cape Cod Bay. The critical issue in this Biological Assessment was the potential impact for entanglement by marine turtles that may forage on sea scallops. The other Biological Assessment involved the use of a dredged material disposal site that was designated prior to Cape Cod Bay being designated a right whale critical habitat. The purpose of this Biological Assessment was to evaluate if the continued use of the site would result in adverse impacts to endangered and threatened species that pass through the bay. In addition, Ms. Foster prepared a Biological Assessment related to expansion of a dredged material disposal site in the inner New York Bight. Potential impacts to six whales and five turtles were evaluated for three alternatives. The quality of these Biological Assessments resulted in all of the proposed projects being approved by the NMFS (as steward of marine resources) as unlikely to adversely impact endangered and threatened species.

Project Manager. Ms. Foster, in accordance with NEPA regulations, prepared an Environmental Impact Statement for the U.S. Coast Guard, Headquarters on their Atlantic Protected Living Marine Resources Initiative. This was the first programmatic EIS prepared for the USCG for their operations on the Atlantic Coast. Ms. Foster evaluated the impacts of current USCG operations and operations proposed under the Initiative. She evaluated impacts for both vessel and aircraft on the physical, biological environment, and socioeconomic environment. Ms. Foster investigated the cumulative impacts of each of the alternatives to determine if the impacts of USCG activities, in addition to impacts from other vessel and aircrafts, resulted in an adverse impact to the biological environment, physical, and socioeconomic environment. This 1000 page EIS was prepared and delivered in three months in response to a court order imposed by a federal judge.

Project Manager. Ms. Foster authored the fish and shellfish affected environment sections of a Supplemental Environmental Impact Statement for Designation of a Historic Area Remediation Site in New York Bight. Ms. Foster provided a summary of the abundance and distribution, feeding habits, and spawning habits of more than 15 fish and shellfish species that occupy the New York Bight throughout the year. An assessment of the potential impact of disposal activities on each of these species was assessed. In addition to fish and shellfish, Ms. Foster also authored the affected environment sections for marine mammals, turtles, and birds.

Project Manager for an EPA project to evaluate the cumulative impacts of several flood control alternatives described in the American River Watershed Investigation Feasibility Report and Final EIS and the Folsom Reservoir Reoperation Feasibility Report and Draft EIS. Best Professional Judgement was used to compare potential cumulative impacts on wetlands, fisheries, water quality, the San Francisco Bay/Delta, air quality, and fish and wildlife habitat to a combination of interim and permanent flood protection alternatives, including the no action alternative.

Project Manager for the 5-year, multidisciplinary Delaware Artificial Reef Study (DARS) for the United States Army Corps of Engineers (USACE) and the Environmental Protection Agency (EPA) to investigate the feasibility of using artificial reefs as compensation for shallow- and deep-water habitat loss. Chief Scientist for conduct of DARS activities on board the OSV *Peter W. Anderson* and R/V *Skimmer.* Project manager for development of artificial reef in Boston Harbor as mitigation for subtidal habitat loss associated with disposal of dredged material from the third harbor tunnel project on Spectacle Island. Ms. Foster is providing expertise on siting, reef design, and monitoring of artificial reef to ensure compensation of lost resources. In both mitigation projects, Ms. Foster is responsible for developing and tracking budget and on-time submittal of deliverables.

Project Manager. Prepared a risk assessment on consumption of fish resident near oil and gas platforms in the Gulf of Thailand for a large monitoring program. For EPA, summarized the document *Assessing Human Health Risks from Chemically Contaminated Fish and Shellfish: A Guidance Manual.* This document provided a brief summary of the manual. Work Assignment Leader to prepare a fact sheet on *Fish and Shellfish Tissue Contaminant and Risk Assessment Studies* to provide information to EPA regional staff on recent studies, procedures, and Federal agency activities. Prepared a report for EPA, *Summary of Fish and Shellfish Tissue Contaminant Studies,* which summarized fish and shellfish contaminant studies conducted by EPA Regions. This was a technology transfer document informing EPA Regions of EPA activities being conducted around the country.

Work Assignment Leader to produce a report for EPA *(Determination of Sludge Settling Rates)* that described the results of settling experiments conducted on samples of sewage sludge disposed from four Publicly Owned Treatment Works (POTW) that are disposed at the 106-Mile Deepwater Municipal Sludge Dump Site (106-Mile Site). Work Assignment Leader responsible for preparing a *Dredged Material Disposal Reference Document* for EPA, which provides an annotated bibliography of all Federal and published documents relating to dredged-material disposal in ocean waters. Project leader to prepare the *Final Environmental Impact Report for the Identification of Dredged-Material Disposal Sites(s) in Cape Cod Bay, Massachusetts.* This document recommends one of four disposal sites and includes a discussion on the impact that disposal could have on the physical (sediment grain size), chemical (trace-metal concentrations), and biological (endangered mammal distribution) aspects of the recommended site. In addition, Ms. Foster prepared the Sampling and Monitoring chapters for the EPA document *Managing Ocean Disposal of Dredged Material.* These chapters provided guidance choosing appropriate sampling equipment, selecting a statistical design, and analyzing data, and using a step-wise approach to develop a monitoring plan. She also assisted in the development of EPA/USACE guidance for selecting dredged material disposal alternatives.

Fisheries

Research Scientist: Prepared summary of histopathology and disease studies of fish and shellfish collected in Boston Harbor. Fishery Biologist (Research) at the NEFC responsible for monitoring stock levels of fish (scup, winter flounder) and shellfish (long-finned squid, ocean quahog) resources in the northwest Atlantic Ocean. Annual monitoring required designing analyses of historical commercial, recreational, and research survey data and interpreting results using statistical techniques (e.g., time-series analysis) to present in reports and at meetings. Also responsible for evaluating resource sampling and advising management on commercial and research survey data-collection requirements. Served as Watch Chief for NEFC groundfish research vessel surveys, supervising the collection of finfish and shellfish data to monitor populations in the northwest Atlantic Ocean. Responsible for determining level of and setting priorities for sampling. Experienced in the analysis of age and growth data. Performed multivariate statistical analyses on winter flounder growth curves. Generated growth curves for surf clams. Designed and conducted research on feeding interactions of predators and prey of Georges Bank, the Gulf of Maine, and Southern New England areas. Served as a member of NEFC Research Council to advise management on present and future research. Special Assistant to the NEFC Center Directorate responsible for coordinating domestic and international travel, advising center directorate on staff issues. Participated in international meetings: International Workshop on Stock Assessment for Tropical Small Scale Fisheries (1979); International Council for Exploration of the Sea (ICES); 69th Statutory Meeting (1981, rapporteur); U.S. Canada Joint Assessment Meetings/Workshops (1984, 1986); Multivariate Analysis of Shellfish Data Meeting (1989). Collaborated with United Kingdom scientist to complete Master's thesis research. Participated in NEFC submersible research survey to observe squid populations on Stellwagon Bank (1976).

Field Experience:

Research Scientist: Ms. Foster has served as chief scientist or a member of the scientific staff on over 20 research surveys on U.S. and foreign vessels. She has been involved in a submersible survey.

Professional Affiliations:

Massachusetts Coastal Zone Management (appointed by Governor), Coastal Resources Advisory Board
1992 - present

Additional Training and Licenses:

Conducting Ecological Risk Assessments – December 1994, Tufts Environmental Management Institute
Managing for Results – January 1998, Simmons College, Graduate School of Management

Peer-Reviewed Publications:

Neff, J., Foster, K, and Nieukirk, S. 1996. NEPA Final Environmental Impact Statement for the U.S. Coast Guard Atlantic Protected Living Marine Resources (APLMR) Initiative. Final report prepared by Battelle Ocean Sciences for U.S. Coast Guard, Headquarters. Washington, D.C., 178 pp + appendices.

Foster, K.L., Kropp,R.K., Steimle, F.W., Muir, W.C., Conlin, B.E. 1995. Fish Community and Feeding Habits at a pre-fabricated concrete artificial reef in Delaware Bay, U.S.A. Proceedings International Conference on Ecological System Enhancement Technology for Aquatic Environments. Volume II. 266-271.

Steimle, F., Muir, W., Foster, K., Conlin, B. 1995. Benthic community enhancement by a prefabricated concrete artificial reef in lower Delaware Bay, U.S.A. Proceedings International Conference on Ecological System Enhancement Technology for Aquatic Environments. Volume II. 581-586.

Neff, J., Nieukirk, S., and Foster, K. 1995. Endangered Species Biological Assessment for the U.S. Coast Guard Activities along the Atlantic Coast. Final report prepared by Battelle Ocean Sciences for U.S. Coast Guard, Headquarters. Washington, D.C., 174 pp + appendices.

Neff, J., Nieukirk, S., and Foster, K. 1995. Enviromental Assessment for the U.S. Coast Guard Activities along the Atlantic Coast. Draft report prepared by Battelle Ocean Sciences for U.S. Coast Guard, Headquarters. Washington, D.C., 82 pp + appendices.

Smith, L.B., Neff, J., Foster, K., Dragos, P., Paquette, R.C., Lychwala, M.E., and Buchholz, K. 1996. Cape Cod Disposal Site Biological Assessment. Final report prepared by Coler and Colantonio and Battelle Ocean Sciences for U.S. Army Corps of Engineers, New England Division. Waltham, MA. 141 pp + appendices.

Smith, L.S., Neff, J., Foster, K., Dunk, D. 1995. Biological Assessment of the Dutra Sea Scallop Aquaculture Proposal. Final report prepared by Coler and Colantonio and Battelle Ocean Sciences for U.S. Army Corps of Engineers, New England Division. Waltham, MA. 80 pp.

Foster, K., and Neff, J. 1996. Biological Assessment for Expansion of the Mud Dump Site. Draft report prepared by Battelle Ocean Sciences for U.S. Environmental Protection Agency, Region II. New York, New York. 98 pp.

Foster, K.L., F.W. Steimle, W.C. Muir, R.K. Kropp, and B.E. Conlin. 1994. Mitigation Potential of Habitat Replacement: Concrete Artificial Reef in Delaware Bay - Preliminary Results. *Bulletin of Marine Science* 55(2-3): 783-795.

Foster, K.L. and W. Steinhauer. 1994. Final Report, Mercury in Gulf of Thailand Finfish: An Ecological and Health Assessment. Submitted to Industrial client. 32 pp.

Foster, K.L., R.K. Kropp, A.M. Spellacy, F. Querzoli, and R. Lordo. 1994. Delaware Bay Artificial Reef Study, Summary: 1990-1993. Final report prepared by Battelle Ocean Sciences for United States Army Corps of Engineers and [U.S.] Environmental Protection Agency, Region III. Philadelphia, PA. Work assignment 18. 89 pp. + appendices

Foster, K.L., R.K. Kropp, A.M. Spellacy, F. Querzoli, R. Lordo, and J. Ryther, Jr.. 1992. Delaware Bay

Artificial Reef Study • Year 3. Draft Final report prepared by Battelle Ocean Sciences for United States Army Corps of Engineers and [U.S.] Environmental Protection Agency, Region III. Philadelphia, PA. Work assignment 3-135. 126 pp. + appendices

Foster, K.L., R.K. Kropp, J.H. Ryther, and A.M. Spellacy. 1992. Delaware Bay Artificial Reef Study - Year 2. Final report prepared by Battelle Ocean Sciences for United States Army Corps of Engineers and [U.S.] Environmental Protection Agency, Region III. Philadelphia, PA. Work assignment 2-135. 92 pp. + appendices.

Foster, K.L., R.K. Kropp, and T.L. Burch. 1991. Delaware Bay Artificial Reef Study - Year 1. Final report prepared by Battelle Ocean Sciences for United States Army Corps of Engineers and [U.S.] Environmental Protection Agency, Region III. Philadelphia, PA. Work assignment 1-135. 67 pp. + appendices.

Foster, K.L. 1992. Dredged Material Ocean Dumping Reference Document. Report prepared by Battelle Ocean Sciences for [U.S.] Environmental Protection Agency Office of Wetlands, Oceans, and Watersheds. Washington, D.C. Work Assignment 3-114. 60 pp.

Foster, K.L. and K.W. Buchholz. 1992. Cumulative Impact Analysis of Alternative Flood Control Projects Based on: American River Watershed Investigation [USACE] FEIS and Folsom River Dam and Reservoir [USACE] DEIS. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Federal Activities Region IX. Work Assignment 3-307. 50 pp.

Foster, K. L. and K.W. Buchholz. 1991. Summary of Ocean-Dumping Activities Under Permits Issued by the [U.S.] Environmental Protection Agency. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection. Work Assignment 2-58. 26 p.

J.S. Bonner, D. Hernandez, K.L. Foster, and S. Dowhan. 1991. Determination of Sludge Settling Rates. Report prepared by Texas A&M University Department of Environmental Engineering, Battelle Ocean Sciences, and SAIC for Environmental Protection Agency Office of Wetlands, Oceans, and Watersheds. Work Assignment 2-108. 53 pp.

K.L. Foster. 1991. Fish and Shellfish Tissue Contaminant and Risk Assessment Studies Fact Sheet. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Wetlands, Oceans, and Watersheds. Work Assignment 2-215. 6 pp.

N. O'Mara, R.L. Peddicord, K. Foster, S. Ossoff. 1990. Dredged Material Disposal Strategy Document. Draft final report prepared by Battelle Ocean Sciences and EA Engineering for Environmental Protection Agency Office of Marine and Estuarine Protection. Work Assignment 1-01. 103 pp.

Foster, K.L. 1990. Pre-Operational Assessment - Annex III of the London Dumping Convention. Prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection. Work Assignment 58. 19 pp.

Foster, K.L. 1990. Comparison of the U.S. Approach to Waste Disposal with the Approach Described in the TNO Paper • An Appraisal of Marine Waste Dumping Criteria Based on Risk Analysis and Ecological Effects. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection, Washington, DC. Work Assignment 1-58. 24 pp.

Foster, K.L. and C.D. Hunt. 1990. Summary of Monitoring and Research at the 106-Mile Deepwater Municipal Sludge Site. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection. Work Assignment 1-58. 24 pp.

Foster, K.L. 1990. Summary of • Assessing Human Health Risks from Chemically Contaminated Fish and Shellfish: A Guidance Manual. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection . Work Assignment 1-58. 10 pp.

Foster, K.L. 1990. Pre-Operational Assessment • Annex III of the London Dumping Convention. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection. Work Assignment 1-58. 19 pp.

Buchholz, K.W., and K.L. Foster. 1990. EPA and the Environmental Aspects of Dredged-Material Disposal. Lecture notes prepared by Battelle Ocean Sciences for Environmental Protection Agency Marine and Estuarine Protection. Work Assignment 2-121. 89 pp.

Milliman, J., and K.L. Foster. 1990. Final Environmental Impact Report for the Identification of Dredged-Material Disposal Site(s) in Cape Cod Bay, Massachusetts. 206 pp. + appendices.

Foster, K.L. 1990. The Buzzards Bay Project Synthesis Report on the Toxic Chemical in Buzzards Bay: Sources, Fates, and Effects. Executive summary to be included in the Buzzards Bay CCMP to be authored by EPA and the State of Massachusetts. 3 pp.

Buchholz, K.W., K.L. Foster, V.R. Gibson, S. Hill, H.K. Trulli. 1989. Initial Draft Water-Use Activity Impact Reports for Delaware Inland Bays Water-Use Plan and Assessments of Marine Impacts. Report prepared by Battelle Ocean Sciences for Environmental Protection Agency Office of Marine and Estuarine Protection and State of Delaware Department of Environmental Resources and Environmental Control Division of Water Resources. Work Assignment 42. 126 pp.

Overholtz, W.J., S.A. Murawski, and K.L. Foster. 1989. Impact of predatory fish, marine mammals, and seabirds on the pelagic fish ecosystem of the Northeastern USA. ICES Symp.: Multispecies Models Paper No. 10.

Foster, K.L. 1987. Status of winter flounder *(Pseudopleuronectes americanus)* Stocks in the Gulf of Maine, Southern New England and Middle Atlantic areas. NEFC, Woods Hole Laboratory Ref. Doc. 87-13.

Gabriel, W.L. and K.L. Foster. 1986. Preliminary assessment of winter flounder *(Pseudopleuronectes americanus* Walbaum) on Georges Bank. NEFC, Woods Hole Laboratory Ref. Doc. 86-16.

Foster, K.L. 1982. An Application of a Multispecies Cohort Analysis to Six Georges Bank Fish Stocks. M.S. Thesis, University of Rhode Island, Kingston, R.I.

Foster, Karen L. 1982. An Application of a Multispecies Cohort Analysis to Six Fish Stocks Located on Georges Bank. ICES C.M. 1982/G:37.

Lange, A.M.T., and K.L. Johnson. 1981. Dorsal mantle length, total weight relationships of squids *(Loligo pealei* and *Illex illecebrosus)* from the Atlantic coast of the United States. NOAA Tech. Rep. NMFS SSRF-745.

Johnson, K.L. 1979. Yield-per-recruit analysis for summer flounder *(Paralichthys dentatus).* Woods Hole Laboratory Ref. Doc. No. 79-34.

Tibbetts-Lange, A.M.T. and K.L. Johnson. 1979. Dorsal mantle length, total weight relationships of squids *(Loligo pealei* and *Illex illecebrosus)* from the Northwest Atlantic off the coast of the United States. NAFO Res. Doc. 79/4.

Lange, A.M.T. and K.L. Johnson. 1978. Dorsal mantle length, total weight relationships of squid *(Loligo pealei* and *Illex illecebrosus)* from the Northwest Atlantic off the Coast of the United States. Woods Hole Laboratory Ref. Doc. No. 78-51.

Pertinent Presentations:

NOAA, NMFS, Northeast Fisheries Center Research Meeting. Preliminary analysis of feeding heterogeneities related to lengths of three piscivorous fish. February 1988. Galilee, Rhode Island.

National Estuary Program Science '91 Symposium. Delaware Artificial Reef Study - Year 1. February 25 - 27, 1991. Sarasota, Florida.

Thayer Academy Regional Junior High Science Fair. Keynote Speaker. May 11, 1991.

5th International Conference on Artificial Habitats for Fisheries. Mitigation Potential of Habitat Replacement: Concrete Artificial Reef in Delaware Bay - Preliminary Results. November 3 - 7, 1991. Long Beach, CA.

Invited Expert Artificial Reef Panelist for Second Southern California 300-Acre Artificial Kelp Reef Workshop. Sponsored by Southern California Edison. April 6 - 7, 1992. Long Beach, California.

Women's Fisheries Network. Guest Speaker. From Fisheries Science to Contract Research. September 22, 1992. Boston, MA.

International Conference on Ecological System Enhancement Technology for Aquatic Environments. Fish Community and feeding habits at a pre-fabricated concrete artificial reef in Delaware Bay, U.S.A. October 1995. Tokyo, Japan.

International Conference on Ecological System Enhancement Technology for Aquatic Environments. Benthic community enhancement by a pre-fabricated concrete artificial reef in lower Delaware Bay. October 1995. Tokyo, Japan.

Public Meeting on Expansion of the Mud Dump Site. Fish and shellfish resources. Freeport, New York. April 1996.

Public Meeting on Expansion of the Mud Dump Site. Fish and shellfish resources. Montauk, New Jersey. May 1996.

GC/MS ANALYST

Julie Fredriksson **Battelle Duxbury**

Research Scientist

Education:

B.A. Chemistry, Wheaton College, Norton, MA — 1986

Qualifications:

Ms. Fredriksson joined Battelle in 1986. She has over 14 years experience in the field of environmental analytical chemistry and has assumed a variety of roles during her employment at Battelle. Her primary responsibilities include project management and participation in various methods development related activities. Ms. Fredriksson has more than 12 years of gas chromatography/mass spectrometry (GC/MS) experience specializing in the analysis of environmental samples for trace organic contaminants.

Professional Positions:

Research Scientist, Battelle Duxbury MA, 1996 to present
Laboratory Research Specialist, Department of Toxicology, North Carolina State University, Raleigh NC, 1993 – 1995

Relevant Experience:

Project Manger – Responsible for management of monitoring, dredging, and hydrocarbon related studies including proposal preparation, workplan development, budget/schedule tracking, and deliverable preparation.

GC/MS Facility Manager – Responsible for training staff, overseeing data quality, coordinating workloads, and ensuring overall system/facility efficiency.

GC/MS Analyst/Task Leader – Analysis of various matrices including water, effluent, soil, sediment, tissue, and petroleum products for trace level PAH, phthalates, pesticide, and PCB congeners by GC/MS following NOAA NS&T and EPA EMAP methods. Responsible for data reduction and QC data package preparation.

Laboratory Research Specialist – Supervised and trained graduate students and laboratory staff in the preparation and trace level analysis of environmental samples for organic contaminants (PAH, pesticides, and PCBs) using GC/MS and GC/ECD. Participated in methods development activities.

Lab Technican – Responsible for preparing environmental samples (soils, sediments, tissues, waters) in the laboratory for PAH, pesticides, and PCB.

QUALITY ASSURANCE OFFICER

Mark Guilmain **Battelle Duxbury**

Quality Assurance Coordinator

Education:

Aquaculture Technology, Salem State College (1998)
Indroduction to Business Management, University of Massachusetts (1997)
Marine Toxicology, University of Massachusetts Graduate Program (1997)
Biological Oceananic Processes, University of Massachusetts Graduate Program (1996)
Pollution Prevention, Tufts University Graduate Program (1995)
Site Remediation Technology, Tufts University Graduate Program (1994)
University of Massachusetts, BS Zoology (1992)

Qualifications:

Mr. Guilmain joined Battelle in May, 2000 and brings a broad background of technical experience to his present position as Quality Assurance Coordinator. With 7 years' experience as an environmental chemist he was appointed Quality Assurance Officer for the Massachusetts Department of Environmental Protection, Division of Watershed Management. Mr. Guilmain joined Battelle's Quality Assurance Unit as the analytical laboratory Quality Assurance Coordinator.

As Quality Assurance Coordinator for Battelle, Mr. Guilmain is responsible for management of the Quality Assurance Unit analytical activities. Mr. Guilmain ensures implemention of the site Quality Management Plan at Battelle, and is responsible for maintaining state certifications for Battelle's analytical laboratory, for preparing and reviewing standard operating procedures, identifying and mitiating staff training, conducting laboratory inspections of both Battelle and subcontractor activities, and verifying reported data through data audits.

Mr. Guilmain coordinates, supervises, and participates in all QA activities associated with the collection of environmental data. He implements Battelle's requirement that all environmental data be audited by the Quality Assurance Unit and therefore has extensive experience in auditing analytical chemistry. He is congizant of state and federal QA programs related to the collection and analysis of environmental data.

Professional Positions:

Quality Assurance Coordinator, Quality Assurance Unit, Battellle. 2000 - present. Responsible for the management of the Quality Assurance Unit activities for the analytical chemistry laboratory, performing data audits, laboratory inspections, and review of analytical standard operating procedures.

Quality Assurance Officer, Massachusetts Department of Environmental Protection (DEP), Division of Watershed Protection. 1999 - 2000. Responsible for the development of the Quality Assurance Program at the DEP including environmental monitoring data validation, writing and reviewing field and laboratory standard operating procedures and providing technical support and review for the preparation of Quality Assurance Project Plans.

Environmental Chemist, Massachusetts Water Resource Authority. 1996 - 1999. Responsible for final validation and review of organic laboratory environmental data reports, for performing GC/MS low level method development, for tracking trends in environmental data and implementing strategies to correct deviations, for writing and reviewing laboratory standard operating procedures and statistical spreadsheets, and for training staff on data reporting and GC/MS maintenance/analysis procedures.

Environmental Analyst ,Camp Dresser & McKee Engineering. 1992 - 1996. Analysis and method development of drinking water and wastewater by GC/MS using EPA Methods 625, 8270 and 500 series. Compiling and entering environmental laboratory data into LIMS database for review. Training staff on data reporting and GC/MS maintenance/analysis procedures.

Relevant Experience:

As *Quality Assurance Coordinator* Mr. Guilmain maintains the Quality Assurance Manual that describes the analytical laboratory's procedures and quality control requirements, and he is responsible for maintaining the laboratory's State certifications.

Project QA Officer: In addition to the planning and inspection phases of each project, a major role of the QA Office at Battelle is the verification that all reported data are accurate, traceable, and collected according to the project QA plan and associated SOPs. Mr. Guilmain coordinates and participates in this essential activity. His expertise ranges from the audit of analytical chemistry data, to verification of data contained in database systems. He functions as the QA Officer for several projects and is the lead auditor for many.

Project QA Officer: Mr. Guilmain has experience in support of extrinsic inspections conducted at Battelle. He coordinates preparation, assists laboratory personnel in corrective actions to audit findings, and prepares written reports of findings to management. Recent field sampling and analytical laboratory inspections include an EPA Oceans and Coastal Protection Division *Enterococcus* beach monitoring study.

Project QA Officer for the EPA National Fish Study: Mr. Guilmain is responsible for managing and coordinating all project QA activities. Specific responsiblities include: reviewing and approving the Quality Assurnace Project Plan (QAPP) and updating it as needed, verifying that project activities are conducted according to the QAPP and administering a laboratory training program for both QA and technicial staff to ensure the QAPP requirements are understood and implemented. In addition, he is responsible for ensuring project audits and inspections are conducted, participating in or coordinating client on-site inspections and audits and preparing periodic status reports of QA activities and audit results for the Battelle Operations Manager.

SAMPLE PREPARATION CHEMIST

Beth Kitson **Battelle Duxbury**

Research Technician

Education:

B.A. Medical Technology, Northeastern University, 1966

Qualifications:

Ms. Kitson has been a laboratory technician at Battelle since 1979 and has worked in the Organic Prep laboratory since 1998. Ms. Kitson is experienced in organic extractions of water, soil/sediment and tissue samples, and in the preparation of laboratory standards.

Professional Positions:

Research Technician. Organic Prep laboratory 1998-present
Researcher. Trace Metals Laboratory 1988-1988 Trace Metals sample preparation and analyst.
Medical Technologist (ASCP). New England Deaconess Hospital Chemistry Laboratory. Performed routine and special chemistry procedures. 1965 to 1970.

Relevant Experience:

Research Technician Preparation of flounder, lobster and mussels for PAH, PCB and pesticide analysis for MWRA monitoring program.

Research Technician. Preparation of water and soil samples for PAH analysis for WA 3-14 and 4-14.

Research Technician Preparation of water samples for PAH analysis for Delaware City Refinery.

Research Technician Preparation of bivalve tissues for PAH, PCB and pesticide analysis for NOAA NS&T Mussel Watch program.

Research Technician Preparation of soil samples for hydrocarbon analysis for Rhizosphere Enhanced Bioremediation program.

Research Technician Prepared soil samples for PAH, PCB and pesticide analysis for USACE studies of Housatonic River, Clinton Harbor and North Cove.

Research Technician Preparation of prep laboratory standards.

Researcher. Preparation and analysis of mercury and other metals in effluent, sediment, and lobster and fish tissues being collected for the MWRA Harbor and Outfall Monitoring project.

Researcher. Preparation and analysis of sediment samples for criteria metals as part of ocean dredged material disposal site (ODMDS) site assessments.

Researcher. Preparation and analysis of seawater samples for development of site-specific copper criteria for New York/New Jersey Harbor study. Also responsible for total recoverable, acid soluble, dissolved and particulate mercury measurements in water for the New York/New Jersey Harbor waste load allocation investigations. Served as field chemist assisting in water column sampling for these studies.

Researcher: Successfully extended the application of automated hydride arsenic and selenium measurements to include sediment and tissue matrices.

Field Chemist of the ultra clean trace metals samples in NY harbor and tributaries which established previously undocumented low levels of ambient metals.

Researcher. Performed ICP/AES analysis of porewaters for the St John's River Monitoring Program and tissues for the San Juan Bay Estuary Study.

Researcher. Assisted in development of Toxicity Identification/Reduction Evaluation (TIE/TRE) methods for industrial effluents. Also prepared fractions and measured chloride and fluoride of industrial effluents.

Experienced in conduct of short-term chronic toxicological tests using hexavalent chromium analyses on reference toxicant. Also experienced in methods for large-scale culture and harvesting of *Mysid and Cyprinodon,* and *Champia* and *Isochrysis* used for toxicology testing.

Additional Training and Licenses:

Completed short course and attended several workshops for instruction and method development discussions for flow-injection analysis (FIAS) of As, Se, Hg, and Sb.

Completed a technical writing course at Bridgewater State College in June 1996.

DIOXIN QUALITY ASSURANCE OFFICER

Charles D. Lawrie **Battelle Columbus**

Quality Assurance Manager

Education:

M.S., Marine, Estuarine, and Environmental Sciences, University of Maryland, 1990
B.S., Biological Science, University of Illinois at Chicago, 1987

Qualifications:

Mr. Lawrie currently serves as Quality Assurance (QA) Coordinator in Battelle's Environmental Protection Division and AgriFood Division. In this capacity, he provides QA guidance to a variety of environmental research programs and is also responsible for coordinating all internal and external QA audits and inspections. Mr. Lawrie has over 10 years experience administrating Good Laboratory Practices (40 CFR Part 160) both as a GLP study director and as a Quality Assurance Manager. Since arriving at Battelle two years ago he has established a Quality Assurance Program within the Atmospheric Sciences and Applied Technology department as specified in EPA Order 5360.1. He routinely administers QA and QC programs required in environmental monitoring, environmental surveys, and analytical laboratory systems. He also reviews standard operating procedures (SOPs) and conducts performance and data audits to ensure compliance of facilities and operations with Quality Management Plans (QMPs) and Quality Assurance Project Plans (QAPPs). An environmental chemist with 10 years' experience, Mr. Lawrie holds a Master's degree in Marine, Estuarine, and Environmental Science, specializing in Environmental Chemistry. He has experience conducting audits, providing technical leadership during environmental laboratory certifications, and providing training to technical staff in the use of quality and improvement tools. His areas of expertise include analytical chemistry, environmental toxicology, and experimental design.

Relevant Experience:

Human and Ecological Exposure Monitoring Research (HEEMR) Projects: Mr. Lawrie is the Battelle Quality Manager on all the Task Orders pertaining to the EPA HEEMR project. These studies target a wide variety of chemical agents and study designs concerning human exposure studies. As the Quality Manager Mr. Lawrie's administrates all quality aspects of this project including Quality Assurance Project Plan (QAPP) review and technical system and data audits to ensure that staff follow QAPPs, Standard Operating Procedures (SOPs), and pertinent regulations and methods.

AgriFood Projects: Mr. Lawrie administers the quality assurance program of a large agricultural research department specializing in agrochemical product development under the EPA Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA). This department is compliant with the Good Laboratory Practices and all pertinent regulations of FIFRA. Mr. Lawrie administrates all the Battelle quality aspects of this department including protocol reviews, technical system and data audits to ensure that staff adhere to the GLPs, protocols, SOPs, and pertinent regulations and methods.

Environmental Technology Verification (ETV) Program: Mr. Lawrie is the Battelle Quality Manager for the EPA's ETV program for the Advanced Monitoring System pilot. This program provides independent facility testing of new and existing environmental technologies to enhance their commercial appeal. The nature of this program requires an extremely high level of quality assurance activities. Mr. Lawrie's administrates all the Battelle quality aspects of this pilot program including Battelle Quality Management Plan (QMP) preparation, QAPP review, and technical system and data audits to ensure that staff adheres to the QMP, QAPPs, SOPs, and pertinent regulations and methods.

Dioxin Projects: Battelle operates an advanced dioxin laboratory specializing in high-resolution mass spectrometry. Clients are typically federal and state agencies requiring dioxin analysis according to established EPA methods, which often requires certification under the respective state's guidelines. Mr. Lawrie's administrates all the Battelle quality aspects of this project including Battelle QMP preparation, QAPP review and technical system and data audits to ensure that staff adheres to the QMP, QAPPs, SOPs, and pertinent regulations and methods.

ORGANICS LABORATORY MANAGER

Robert Lizotte **Battelle Duxbury**

Research Scientist

Education:

B.S. Biology, Allegheny College, 1987

Qualifications:

Mr. Lizotte joined Battelle in 1990 and has a total of 11 years of experience in the field of environmental chemistry. His responsibilities include lab oversight and scheduling. He also ensures proper procedural techniques are employes throughout the lab. His anlaytical responsiblities are preparation and analysis with various detectors in gas chromatography of environmental samples in various matrices. These detectors include GC/FID, PID, FPD, and ECD. Analytes include petroleum hydrocarbons, polynuclear aromatic compounds, sterols, pesticides, butyltins, and polychlorinated biphenyls. Further duties include sample preparation, analytical standard preparation, interpretation of GC/FPD, GC/FID and GC/ECD chromatograms, instrumentation troubleshooting and maintenance, and final preparation of data packages. He also acts a project manager, involving client contact, preparation of reports, and preparation of workplans. Mr. Lizotte is Lab Manager of the organics laboratory at Battelle.

Professional Positions:

Supervisor of the Hydrocarbon Laboratory, Enseco Incorporated, Cambridge, Massachusetts. Duties included client contact, instrument scheduling and maintenance, and assigning work to technicians.

Relevant Experience:

Ecological and Human Exposure Measurement and Monitoring

Research Scientist: Performed project management tasks and analysis for a study in China Bay (Hong Kong) examining possible butyltin contamination.

Research Scientist: Performed project management tasks and analysis for the Consortium of Tributyltin Manufacturers for the Long Term Tributyltin Monitoring Project.

Research Scientist: Conducted butyltin analysis and prepared data report for a study conducted for EPA's Oceans and Coastland Protection Division at the 106-mile dump site.

Research Scientist: Performed PCB congener and pesticide analysis in support of the NOAA NRDA study at a Superfund site in San Francisco Bay (excess of 200 tissue and sediment samples).

Research Scientist: Performed PCB congener and pesticide analysis for US Army Corps of Engineers Oakland Harbor Navigation project, Richmond Harbor improvement, and J.F. Baldwin channel. An excess of 200 tissue samples were analyzed in these projects.

Research Scientist: Performed PCB congener and pesticide analysis for a U.S. Navy Superfund site. Analyzed samples and reported data for over 250 samples.

Research Scientist: Performed PCB congener and pesticide analysis for the NOAA Mussel Watch projects (1990 to 1994). Analyzed samples and reported data for over 500 tissue samples.

Research Scientist: Performed Massachusetts Water Pollution Performance Evaluation study for PCB and pesticides in 1992 through 1994.

Research Scientist: Analyzed and reported petroleum hydrocarbon data in many Exxon studies including two intercalibration exercises.

Research Scientist: Analyzed and reported data petroleum hydrocarbon data for an industrial oil and gas research program.

Trained two people from Thailand on the extraction, instrumentation, and calculation of the EPA Method 418.1 IR analysis.

SAMPLE CUSTODIAN/SAMPLE PREPARATION CHEMIST

Mark Misita **Battelle Columbus**

ResearchTechnician

Education:

A.A.S., Hazardous Materials Technology, Ohio University, 1996

Qualifications:

Mr. Misita is currently responsible for overseeing the daily activities in Battelle's dedicated PCDD/PCDF sample preparation laboratory. His duties include maintaining supply inventories, scheduling standard and reagent preparation and extract cleanup activities to coordinate with project schedules, and calibration and maintenance of all equipment used in sample preparation activities. Mr. Misita has demonstrated success in PCDD/PCDF sample preparation using EPA Methods 1613, 8290, 23, and TO9. Mr. Misita has also participated in an internal research and development project to evaluate draft Method 1668 for toxic PCBs and has also developed preparation procedures for the determination of PCBs in sewage sludge, incinerator scrubber water, and emission samples from sewage incinerator stationary sources. He is also experienced in sample preparation for PAH and metals analysis and has extensive hazardous materials training.

Relevant Experience:

PCDD/PCDF Analyses to Determine Coastal Environment Quality. Prepared over 1200 sediment and tissue samples from multiple sites along the East Coast for PCDD/PCDF analysis according to Method 8290 and 1613. The analyses were conducted to determine environment quality and the quality of dredged material proposed for ocean disposal. Samples prepared for these programs have consistently met or exceeded data quality objectives.

PCBs in Sewage Sludge, Incinerator Emissions, and Scrubber Water. Mr. Misita led method development for the extraction of toxic PCBs by a modification of Method 1668. This method was then applied to the analysis of sewage sludge, emissions, and scrubber water from a sewage sludge incineration facility.

PCDD/PCDF in Wastewater Treatment Plant Sludges. Prepared wastewater treatment plant sludge samples from various municipalities in Ohio for PCDD/PCDF analysis according to modified Method 8290 procedures. Preparation involved additional cleanup columns for especially filthy samples.

PCDD/PCDF in Drinking Water. Drinking water samples were extracted and analyzed for a government agency. Detection limits were in the part-per-quadrillion range (less than 10 pg/Liter)

Evaluation of Influent and Effluent Water for a Water Treatment Facility. Pre-discharge water samples from a water treatment facility were extracted and analyzed for PCDD/PCDF using Method 8290 with modifications from Method 1613.

PCDD/PCDF in Air Samples. Air samples, sampled with PUF cartridges, were extracted and analyzed for a government agency using Method TO9.

PCDD/PCDF and Coplanar PCBs in Cage Wash Rinses. Prepared water samples from animal cage wash rinses for the simultaneous determination of PCDD/PCDF and coplanar PCBs. Results of these analyses were used to clear the rinse water for disposal.

Evaluation of Method 1668 for Toxic PCBs (Oct. 1995 Draft). Prepared samples for an internal research and development project to evaluate Draft Method 1668. An initial precision and recovery sample set using the tissue reference matrix (corn oil) was prepared.

PAH Studies. Participated in the development of a sample preparation method to determine PAH in resin samples. Also prepared XAD traps for collecting air samples used in PAH analysis programs. Method development of PAH in food samples using accelerated extraction and clean-up techniques such as accelerated solvent extraction (ASE), super critical fluid extraction (SFE), solid phase extraction (SPE) and immunoassay techniques. PUF and XAD clean-up and trap packing

Inorganic Sample Preparation. Prepared soil samples for determination of metals following procedures in Method 3050.

Additional Training and Licences:

General Health and Safety Procedures
Hazardous Communications
Good Laboratory Practice Regulations (Mid-January 1998)
OSHA 8-Hour Refresher Training
OSHA 40-Hour HAZWOPER Training
First Responder Awareness
First Responder Operations Level
Industrial Safety
CPR/First Aid

SAMPLE PREPARATION

Hein (Henry) Pham **Battelle Columbus**

Technician

Education:

B.A., Chemistry, Ohio State University, 1999

Qualifications:

Mr. Pham is currently responsible for performing the daily activities in Battelle's dedicated PCDD/PCDF sample preparation laboratory. His duties include ordering supplies, reagent preparation, and performing sample extraction and extract cleanup activities to coordinate with project schedules. Mr. Pham has demonstrated success in PCDD/PCDF sample preparation using EPA Methods 1613, 8290, 23, and TO9. Mr. Pham has also participated in the setup and demonstration of EPA Method 1668A for PCB in water samples and the development of a modified Method 1668 for the determination of PCBs in sewage sludge.

Relevant Experience:

PCDD/PCDF Analyses to Determine Coastal Environment Quality. Prepared over 200 sediment and tissue samples from multiple sites along the East Coast for PCDD/PCDF analysis according to Method 8290 and 1613. The analyses were conducted to determine environment quality and the quality of dredged material proposed for ocean disposal. Samples prepared for these programs have consistently met or exceeded data quality objectives.

PCBs in Sewage Sludge, Incinerator Emissions, and Scrubber Water. Mr. Pham participated in method development for the extraction of toxic PCBs by a modification of Method 1668. This method was then applied to the analysis of sewage sludge, emissions, and scrubber water from a sewage sludge incineration facility.

PCDD/PCDF in Drinking Water. Drinking water samples were extracted and analyzed for a government agency. Detection limits were in the part-per-quadrillion range (less than 10 pg/Liter)

Evaluation of Influent and Effluent Water for a Water Treatment Facility. Pre-discharge water samples from a water treatment facility were extracted and analyzed for PCDD/PCDF using Method 8290 with modifications from Method 1613.

PCDD/PCDF in Air Samples. Air samples, sampled with PUF cartridges, were extracted and analyzed for a government agency using Method TO9.

Additional Training and Licences:

General Health and Safety Procedures
Hazardous Communications

DIOXIN LABORATORY MANAGER

Mary E. Schrock **Battelle Columbus**

Principle Research Scientist

Education:

B.S., (Summa Cum Laude) Chemistry, Kent State University, 1986

Qualifications:

Ms. Schrock's primary responsibility is managing large PCDD/PCDF analysis programs for various government and industrial clients. Ms. Schrock has overseen the analysis of several thousand PCDD/PCDF samples and has consistently provided clients with data packages that meet or exceed quality assurance requirements and are on time and within budget. Ms. Schrock has more than 13 years of experience creating and optimizing analytical methods for separating, identifying, and quantifying trace levels of organic compounds in environmental samples and consumer products, including over 8 years in the analysis of PCDD/PCDF compounds by GC/HRMS. Ms. Schrock has provided data packages meeting the quality control criteria of EPA Methods 8290, 1613, 23, and TO9. In addition, Ms. Schrock led the development of procedures to simultaneously determine PCDD/PCDF and coplanar PCBs, and also led an internal evaluation of Draft Method 1668 for toxic PCBs.

Relevant Experience:

PCDD/PCDF Analyses to Determine Coastal Environment Quality. Managed programs from multiple sites along the east coast to evaluate more than 1,000 sediment and tissue samples from bioaccumulation studies to determine environment quality and the quality of dredged material proposed for ocean disposal. These data packages have consistently met or exceeded data quality objectives and have been performed within time and budget constraints.

Toxic PCBs in Sewage Sludge Incinerator Emissions. Assisted in the development of HRMS methods for determining toxic PCBs in raw sewage sludge, sewage sludge incinerator emissions, and incinerator scrubber water. These methods were then applied to the analysis of samples from a sewage sludge incinerator facility to assist in source characterization.

Support for the Dioxin Workgroup of the Canada-U.S Binational Toxics Strategy (BNS). Researched the current literature and worked with contacts at EPA to develop several reports presenting an overview of dioxin sources and providing direct answers to stakeholder questions concerning dioxin inventories with respect to their quality, remaining uncertainties, and points of contention. The reports were well received by both EPA and the multi-stakeholder dioxin workgroup.

Simultaneous Determination of PCDD/PCDF and Coplanar PCBs. Led the development of sample preparation and GC/HRMS analysis procedures to simultaneously determine PCDD/PCDF and coplanar PCBs. This single procedure has allowed clients to evaluate not only PCDD/PCDF, but also the dioxin like coplanar PCBs in their environmental samples at little additional cost.

Evaluation of Method 1668 for Toxic PCBs (Oct. 1995 Draft). Led an internal research and development project to evaluate Draft Method 1668. Coordinated preparation and analysis efforts to evaluate procedures for tissues using corn oil as the reference matrix.

Remedial Investigation and Ecological Assessment for Surface Water and Sediments at the McCormick and Baxter Superfund Site. Managed the PCDD/PCDF analysis task of this program, which included analyzing 100 tissue and sediment samples for PCDD/PCDF and coplanar PCBs. EPA Method 8290 was adapted for simultaneous determination of PCDD/PCDF and coplanar PCBs. Tissue types included a variety of fish, crayfish, and worms from bioaccumulation test studies. TCDD detection limits less than 1 pg/g were obtained. Battelle received an overall "excellent" rating from EPA Region IX for work supporting this Superfund program.

Draft Guidance Document on Evaluation of Dredged Material Contaminated with PCDD/PCDF. Provided technical expertise on the analysis of PCDD/PCDF and the use of EPA approved PCDD/PCDF analytical methods for this draft guidance document, which is being prepared to assist regulators involved with dredged material testing.

Technical Support to the Exploratory Statistical Analysis of Dioxin Data in Meat and Poultry. Provided dioxin analysis expertise to support an exploratory statistical analysis of beef, pork, and poultry data generated as part of the U.S. EPA Dioxin Exposure Initiative.

Dioxin/Furan Analysis of Lobster and Fish Tissues. As Project Manager, oversaw the analysis of lobster muscle and hepatopancreas tissues and tissues from fish collected in the New York Bight Apex and nearby areas for chlorinated dioxins and furans. Study requirements included achieving a 1 pg/g MDL under EPA Method 8290 and analyzing extensive quality control samples to ensure precision and accuracy of the data. Several hundred samples were analyzed under these projects. The lobster and fish data were used by NOAA to develop information on potential contamination of organisms captured by recreational and commercial fishermen in the area.

Study of Toxic Emissions from Coal-Fired Power Plants. Coordinated and participated in the PCDD/PCDF analysis of emission from coal-fired power plants to help characterize toxic emissions for the U.S. Department of Energy.

PCDD/PCDF in Kuwait Air Samples. Organized and participated in the analysis of ambient air samples from Kuwait for the U.S. Army Environmental Hygiene Agency. This study provided key data in determining air quality in the vicinity of oil fires.

PBDD/PBDF Characterization of Commercial Products. Modified and optimized GC/HRMS methods for determining polybrominated dibenzo-p-dioxins and dibenzofurans in flame retardants in support of product registration.

PCB Analysis of Air Samples from a PCB Fire. Provided GC/ECD determination of polychlorinated biphenyls (PCB) in ambient air samples collected during a PCB fire.

Additional Training and Licences:

Opusquan Training Course; Fisons Instruments – 1994
Basic Operation of the VG Autospec Mass Spectrometer - Fisons Instruments - 1994
ACS Course - Mass Spectrometry: Principles and Practice; Michigan State University - 1991
Fundamentals of Chromatographic Analysis; Kent State University - 1988
Basic Capillary GC; Northeastern University; Chrompak - 1987

Professional Affliations:

Ms. Schrock is a member of the American Chemical Society, Phi Beta Kappa, and Iota Sigma Pi. She was the recipient of a Lubrizol Foundation Scholarship in 1984 and an Honors College Scholarship during the period 1982-86. She received an Undergraduate Award in Analytical Chemistry and was recognized as Outstanding Junior Class Chemistry Major in 1985.

Pertinent Publications:

Schrock, M.E.; Barrows, E.S.; Rosman, L.B.; "Biota-to-Sediment Accumulation Factors for TCDD and TCDF in Worms from 28-Day Bioaccumulation Tests," Chemosphere, Vol. 34, Nos 5-7, pp. 1333-1339, 1997.

Schrock, M.E.; Armbruster, M.J.; Riggs, K.B.; Tabor, J.E.; Doherty, A.K.; Lorber, M. "Simultaneous Determination of PCDD/PCDF and Dioxin-Like PCBs in Edible Vegetable Oils," Dioxin '96 - 16th International Meeting, Amsterdam, The Netherlands, August 1996.

Riggs, K.B.; Roth, A., Kelly, T.J.; Schrock, M.E.; "Ambient PCDD/PCDF Levels In Montgomery County, Ohio: Comparison to Previous Data and Source Attribution," Dioxin '96 - 16th International Meeting, Amsterdam, The Netherlands, August 1996.

Schrock, M.E.; Barrows, E.S.; Rosman, L.B.; "TCDD/TCDF Levels in Bioaccumulation Test Tissues and their Corresponding Sediments," Second SETAC World Congress, Vancouver, British Columbia, Canada, November 1995.

Schrock, M.E.; Barrows, E.S.; Rosman, L.B.; "TCDD/TCDF Levels in Bioaccumulation Test Tissues and their Corresponding Sediments," Dioxin '95 - 15th International Meeting, Edmonton, Alberta, Canada, August 1995.

Riggs, K.B.; Brown, T.D.; Schrock, M.E.; "PCDD/PCDF Emissions from Coal-Fired Power Plants," Dioxin '95 - 15th International Meeting, Edmonton, Alberta, Canada, August 1995.

Tsai, C.; Maxwell (Schrock), M.; et al. "Co-crystallization of Interferon-Inducing Drug Ethidium Bromide with Nucleic Acids," The Ohio Journal of Science, 80, 38, 1984.

SAMPLE CUSTODIAN/DATA MANAGER

Carolynn R. Suslick **Battelle MSL**

Technician III: Marine Chemistry

Education:

A.A.S. Associate of Applied Sciences, Engineering, Peninsula College, 1995
Pursuing an Environmental Science BA degree, Western Washington University, 1997-present

Relevant Experience:

Ms. Suslick has a thorough understanding of the requirements of the sample preparation laboratory at the Marine Sciences Laboratory. She designed and presently manages the sample log-in system, and is responsible for monitoring incoming sample conditions, proper storage, and chain-of-custody accuracy. Ms. Suslick is also responsible for preparation of environmental samples for chemical analyses. This includes filtering, pH measurement, pH adjustment, salinity measurement, acid cleaning, packaging, and shipping.

Ms. Suslick is the Chemistry Data Manager for the Marine Chemistry Group at Battelle Marine Sciences Laboratory. This includes the processing of both Organic and Inorganic data as requested by Project Managers, electronic download of ICP-MS raw data, evaluation and determination of ICP-MS data for mass selection and quality control, and maintenance of the Chemistry Group Technical Files. Ms. Suslick is experienced in data reduction, manipulation, graphing, storage, and writing macros for complex data manipulation. She gives direction to staff in the preparation of data reports or data packages, assists in the development of presentation materials, and maintains control charts for the Chemistry Group. Ms. Suslick has a thorough working knowledge of the spreadsheet and database programs necessary to comply with client requirements and she is proficient with various types of electronic data transfer protocol, often interacting with clients to facilitate the transfer of data. Ms. Suslick is presently developing a modified laboratory information management system (LIMS) tailored to the special needs of the Marine Chemistry Group. She is also coordinating an effort for the connection of the Sequim facility and the Duxbury facility to share joint laboratory information

As an intern in the Clallam County Water Quality Department, Ms. Suslick designed and prepared a Visual Reference for the Long Range Planning Team depicting the Septic History of the Dungeness Bay Study Project. Her research included cross referencing the historical physical files with the computer database management system currently in use, interacting with various County agencies and special interest groups, and on site exploration of the Study area.

While attending Peninsula College, Carolynn assisted the Buccaneer Newspaper in setting up a computer accounting system. During her seven years as an Administrative Secretary/Office Manager with Cape Flattery School District, Ms. Suslick was responsible for monitoring the Clallam Bay school budget, reviewing purchases, hiring substitutes and preparing monthly payroll and employee files.

GC/HRMS ANALYST

Joe Tabor **Battelle Columbus**

Master ResearchTechnician

Education:

Attended Miami University, Oxford, Ohio, Chemistry

Qualifications:

Mr. Tabor has over 15 years experience in the operation and maintenance of high resolution double focusing magnetic sector mass spectrometers and is currently responsible for the upkeep and maintenance of Battelle's high resolution mass spectrometers dedicated to dioxin and related compound research. In addition, Mr. Tabor has provided GC/HRMS method development and analysis support to a variety of projects requiring determination of PCDD/PCDF and coplanar PCBs. Projects for which Mr. Tabor has led HRMS analysis efforts include the analysis of tissues and sediments to evaluate dredged material for ocean disposal, air samples to characterize stack emissions and ambient air, and chemical products to aid in product registration. Mr. Tabor has provided quality analytical data to meet the requirements of EPA Methods 8290, 1613, 23, 1668, and TO-9. Matrices include tissue (fish, lobster, clams, worms), air, water, soil, sediment, sludge, and wipes from PCB fires and building contamination.

Mr. Tabor has instituted several innovative analytical techniques to assure high quality GC/HRMS data including instrumental procedures to minimize contamination potential and background PCDD/PCDF levels and has also prepared automated data reduction routines to increase efficiency in sample data reporting. Additionally, Mr. Tabor has worked with the Dioxin Sample Preparation Laboratory to build and modify equipment to prepare extracts for cleanup procedures. He is currently involved in an internal research project to evaluate supercritical fluid extraction (SFE) and accelerated solvent extraction (ASE) of PCDD/PCDF from a variety of matrices as an alternative to the conventional Soxhlet extraction method.

Relevant Experience:

PCBs in Sewage Sludge, Incinerator Emissions, and Scrubber Water. Mr. Tabor led method development for the HRGC/HRMS analysis of toxic PCBs by a modification of Method 1668. This method was then applied to the analysis of sewage sludge, emissions, and scrubber water from a sewage sludge incineration facility.

PCDD/PCDF in Drinking Water. Drinking water samples were analyzed for a government agency. Detection limits were in the part-per-quadrillion range (less than 10 pg/Liter)

Evaluation of Influent and Effluent Water for a Water Treatment Facility. Pre-discharge water samples from a water treatment facility were analyzed for PCDD/PCDF using Method 8290 with modifications from Method 1613.

Method 23 Modification. Mr. Tabor modified Method 23 for the analysis of air samples to screen for mono-, di-, and tri-chlorinated dioxins and furans in addition to the usual tetra- octa- chlorinated compounds.

PCDD/PCDF Analyses to Determine Coastal Environment Quality. Mr. Tabor has analyzed thousands of sediments and tissues from bioaccumulation studies for PCDD/PCDF to determine environment quality and the quality of dredged material proposed for ocean disposal.

Dioxin/Furan Analysis of Lobster and Fish Tissues. Mr. Tabor led analytical efforts for the analysis of several hundred lobster muscle and hepatopancreas tissues and tissues from recreationally important fish collected in the New York Bight Apex and nearby areas for chlorinated dioxins and furans. Study requirements included achieving a 1 pg/g MDL under EPA Method 8290 and analyzing extensive quality control samples to ensure precision and accuracy of the data The lobster and fish data were used by NOAA to develop information on potential contamination of organisms captured by recreational and commercial fishermen in the area.

Remedial Investigation and Ecological Assessment for Surface Water and Sediments at the McCormick and Baxter Superfund Site. Mr. Tabor provided HRMS analysis of 100 tissue and sediment samples for PCDD/PCDF and coplanar PCBs. Tissue types included a variety of fish, crayfish, and worms from bioaccumulation test studies. TCDD detection limits less than 1 pg/g were obtained. Battelle received an overall "excellent" rating from EPA Region IX for work supporting this Superfund program.

Study of Toxic Emissions from Coal-Fired Power Plants. Provided PCDD/PCDF analyses of emissions from coal-fired power plants to help characterize toxic emissions for the U.S. Department of Energy.

PCDD/PCDF in Kuwait Air Samples. Provided HRMS PCDD/PCDF analyses of ambient air samples from Kuwait for the U.S. Army Environmental Hygiene Agency. This study provided key data in determining air quality in the vicinity of oil fires.

PBDD/PBDF Characterization of Commercial Products. In support of product registration, Mr. Tabor has modified and optimized GC/HRMS methods for determining polybrominated dibenzo-p-dioxins and dibenzofurans in flame retardants.

Prior to joining Battelle, Mr. Tabor was responsible for Chemical Samples' Co., Columbus, Ohio, qualitative analytical capability for 12 years. He handled all equipment purchases, supplies, and maintenance, as well as method development and day to day operations. His expertise involved packed and capillary column systems, as well as modification and adaption of instrumentation.

Additional Training and Licences:

Opusquan Training Course - Fisons Instruments - 1994
Basic Operation of the VG Autospec Mass Spectrometer - Fisons Instruments - 1994
High Resolution Mass Spectrometry Training - VG Instruments - 1990
Interpretation of Mass Spectra - ACS Audio Course

Pertinent Publications:

Schrock, M.E.; Armbruster, M.J.; Riggs, K.B.; Tabor, J.E.; Doherty, A.K.; Lorber, M. "Simultaneous Determination of PCDD/PCDF and Dioxin-Like PCBs in Edible Vegetable Oils", Dioxin '96 - 16th International Meeting, Amsterdam, The Netherlands, August 1996.

Shafer, K. H., A. Bjorseth, J. Tabor, and R. J. Jakobsen, "Advancing the Chromatography of GC/FT-IR to WCOT Capillary Columns", HRC and CC, Vol. 3 (2/1980) 87-88.

Shafer, K. H., T. L. Hayes, and J. Tabor, "The Use of a Small FT-IR System for GC/FT-IR/MS Analysis", SPIE, Vol. 289, p. 160 (1981).

TASK LEADER – DIOXIN/FURAN/HCX

Karen L. Tracy **Battelle Columbus**

Research Scientist

Education:

B.S., Biology, Valparaiso University, Valparaiso, Indiana
M.B.A., Business, Indiana University Northwest, Gary, Indiana

Qualifications:

Ms. Tracy currently serves as supervisor for the polyhalogenated dibenzo-p-dioxin and dibenzofuran (PCDD/PCDF) analysis programs. Ms. Lesniak has over 10 years experience in the environmental field. Ms. Lesniak has provided data packages meeting the quality control criteria of SW846 Method 8290, and EPA Methods 1613 and TO9 along with coordinating the analysis of sediment, tissue, and water samples using 8290/1613 and air samples for Method TO9 for both industrial and government clients. Instrumental techniques with which Ms. Tracy is familiar include low resolution GC/MS for both volatile and base-neutral organic compounds. Ms. Tracy has over eight years experience conducting analysis for volatile organic compounds by GC/MS using SW846 Methods 8240, 8260, and 624, Drinking Water Method 524, and US EPA SOW90 for the Contract Laboratory Program. She also has conducted analysis for semi-volatile organic compounds by GC/MS using SW846 Method 8270. Ms. Tracy also has reviewed data for polychlorinated biphenyls, gasoline range organics, diesel range organics, herbicide, pesticide, and glycol analyses.

Relevant Experience:

Dioxin/Furan Analysis of Waters and Sediments from a Waste-Waster Discharge Plant. As project manager, Ms. Lesniak oversaw the analysis of influent, effluent, and sea water, and sediment samples from a waste water treatment plant for chlorinated dioxins and furans.

PCDD/PCDF Analyses of Niagara River Sediments. Coordinated the PCDD/PCDF analysis of sediments from several locations of the Niagara River for the U.S. EPA.

Sewage Sludge Incinerator Study. As Laboratory Coordinator for this project Ms. Lesniak coordinated in-house analysis for PCBs and PCDD/PCDF as well as the subcontracted analyses for Proximate/Ultimate and Poly Aromatic Hydrocarbons by HRMS for stack emission, scrubber water, and sludge samples.

PCDD/PCDF Analyses to Determine Coastal Environmental Quality. Reviewed data packages for sediment and marine tissue samples from bioaccumulation studies to determine the quality of dredged material proposed for ocean disposal.
Prior to joining Battelle, Ms. Tracy was responsible for the operations of the Organic Department for

DLZ Laboratories, Incorporated in Columbus, Ohio. She was responsible for GC/MS, GC/FID, and GC/ECD groups for both volatile and semi-volatile analyses as well as the organic preparation laboratory. These responsibilities included scheduling both staff and equipment, review of all data generated, meeting turn around times, and providing answers to client concerns.

Additional Training and Licences:

Project Management Seminar; Battelle – 1999

ATTACHMENT C

Raw Data

Raw Data Elements

Data Package Elements	PCB	Dioxin/Furan/HCX
Inventory Sheet	✓	✓
QA/QC Narratives	✓	✓
Sample Custody/Receipt Data		
Airbills	✓	✓
Custody forms	✓	✓
Sample tags (if available)	Will provide if not adhered to sample bottles	
Sample receipt/log-in sheets	✓	✓
Miscellaneous shipping/receiving records	✓	✓
Internal lab sample transfer/tracking records	✓	✓
Sample Data		
Final data tables (summary data; field and QC)	✓	✓
Tentatively identified compounds summary form		
Total ion chromatograms	✓	✓
Raw spectra of target compound and background subtracted spectrum of target compound for each sample		
Mass spectra of all reported TICs/three best library matches for each sample		
Chromatograms (both columns, if applicable)	✓	✓
GC integration reports	✓	✓
Pesticide identification summary form		
For Pest/PCBs confirmed by GC/MS, copies of raw spectra		
GPC sample chromatograms	✓	
Manual worksheets	✓	✓
Sample preparation records	✓	✓
Sample acquisition logsheets [1]	✓	✓
ICP/MS raw data		
Furnace AA raw data		
Mercury raw data		
Cyanide raw data		
Other analytical raw data		
Standards Data		
MDL study final tables		✓
Initial calibration reports	✓	✓
Continuing calibration reports (including pesticide degradation checks)	✓	✓
Chromatograms and quant reports for all GC/MS standards	✓	✓
Pesticide analyte resolution summary form		
Pesticide calibration verification summary form		
Pesticide analytical sequence summary form		
GC chromatograms and data system print outs for all GC standards		
For pesticides/Aroclors confirmed by GC/MS		
GPC standard chromatograms	✓	
Florisil cartridge check summary form		
Instrument detection limits summary data		
ICP Interelement correction factors summary form		
ICP linear ranges summary form		
CRDL standards for AA and ICP summary form		
Standards preparation forms	✓	✓

Raw Data Elements (continued)

Data Package Elements	PCB	Dioxin/Furan/HCX
QC Data		
Tune reports	✓	✓
SIS recovery data	✓	✓
MS/MSD recovery data	✓	✓
Method blank data	✓	✓
Internal standard areas/RTs (quant reports, above)	✓	✓
QC raw data (RICs, chromatograms, quant reports)	✓	✓
ICP interference check sample summary form		
Spike sample recovery summary form [2]	✓	✓
Post digest spike sample recovery data		
Sample duplicate precision data	✓	✓
LCS recovery data	✓	✓
Standard addition results		
ICP serial dilutions summary form		
QC raw data – ICP, Furnace, mercury		
QC sample preparation records	✓	✓
Miscellaneous Data		
Copies of preparation/analysis logbooks	✓	✓
Screening records		
All instrument output from screening activities		
Preparation logs raw data	✓	✓
Percent solids data – ***Only applicable to Nestling Samples***	✓	✓
Other records (QAPP, project memorandums)	✓	✓

[1] Battelle assumes that acquisition logsheets are records of GC oven conditions under which samples are analyzed.
[2] Battelle assumes that Spike Sample Recovery Summary Forms are summary report tables with recovery results

ATTACHMENT D

Example of Validation Check List Used at Battelle Duxbury and Columbus

Data Validation Checklist for Data Packages – Battelle Duxbury

Calibrations

- ☐ Spot check the response factors for the initial calibration (1/column and 1 mean) and verify that the standard concentrations are correct and that the calibration meets the SOP or work plan
- ☐ Check that mid-check standards bracket all reported samples, and that results meet the SOP or work plan criteria (frequency and acceptance level).
- ☐ Verify that the correct method file is used to quantify each sample
- ☐ Check that Q-deletes have been deleted correctly.

Data Tables

- ☐ Check transcription of weight data onto the report tables.
- ☐ Check any standard amounts and dilutions entered on the quant reports.
- ☐ Verify the report table reporting units and header information.
- ☐ Check that reported analytes are those called for by the work plan.
- ☐ Spot-check at least one data point for each sample. Recalculate from scratch using all calibration and prep factors.
- ☐ Check qualifiers and MDLs vs work plan.
- ☐ Spot-check surrogate recoveries, focusing on samples with differing dilutions, etc. and check vs work plan criteria.
- ☐ If required, verify that reported pesticides and PCBs have been confirmed.
- ☐ Calculate QC data (LCS, MS, SRM, etc.) and check qualifiers vs work plan.

Documentation

- ☐ Flag questions, document ambiguities and errors in the data package.
- ☐ Review the work plan to ensure that all work plan specifications have been met.

I certify that the validation procedures checked above have been completed:

Validated By:	**Date:**

Data Review Checklist for Data Packages – Battelle Columbus

for
Samples Analyzed on:
OPUS Filename:

Initials/Date	Activity
	Check transcription of extract no., sample ID, and sample weight from prep LRB into OPUS forms for each sample.
	Check to see that all 2,3,7,8-isomers are accounted for: concentration reported, flagged for reason not used, or obviously not present in each sample.
	Double check that there are no ethers contributing to furans in each sample.
	Review lock mass checks for any variations, which could affect reported results.
	Flag any recoveries outside 40-135%/25-150% on OPUS form. Tag flagged forms with post-its for ease in highlighting final forms and report.
	Return data to analyst for corrections.
	Analyst makes corrections, updates, reprints and resaves OPUS data.
	Corrected OPUS data saved as ASCII text file. **ASCII text file Filename:**
	ASCII file pulled over to PC
	ASCII file imported to Quattro Pro or Excel **QPRO or Excel Filename:**
	Format spreadsheet data
	Flag in spreadsheet file any recoveries outside 40-135%/25-150% as on OPUS forms.
	Add and flag second column confirmation results in spreadsheet file.
	Check calculations for EMPCs.
	Review all LRBs used for project, check for completeness of information, ensure that the proper correction techniques have been used, sign the bottom of the page, and make a copy of the pages needed for the study file.
	Verify that the original COC and the miniaturized copy of the COC in the sample log-in LRB are identical.
	Spot-check the calculations of concentrations.
	Verify that the daily calibrations, the window set mixture, and the percent valley are within the QC ranges allowed by the method.
	Print 2 copies of each finalized QPRO sample sheet. Place one in the OPUS printouts for each sample. Collect the others for the final report.
	Check to see if the QC check sample records have been updated within the last quarter. Add any new QC sample results from this project.

ATTACHMENT E

Pertinent SOPs

BATTELLE COLUMBUS SOPs

OCT 30 2000

STANDARD OPERATING PROCEDURE (SOP) FOR THE ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) USING HIGH RESOLUTION GAS CHROMATOGRAPHY/HIGH RESOLUTION MASS SPECTROMETRY (HRGC/HRMS) USING MODIFIED METHODS 8290 AND 1613

Originated by: Joseph E Tabor Date: 10/26/00

Approved by: Mary Schmoll Date: 10/26/00
Technical Reviewer

Approved by: Karen Riggs Date: 10/27/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

Charles P. Lawrie Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for HRGC/ HRMS analysis of samples for PCDD/PCDF. These analyses follow general guidelines described in EPA Methods 8290 and 1613, with some minor modifications/improvements. The purpose of this SOP is to provide a description of PCDD/PCDF sample analysis activities using modified Methods 8290 and 1613 procedures and covers the following:

- Chromatographic/mass spectrometric conditions and data acquisition parameters
- Calibration
- Analysis
- Calculations
- System performance criteria.

III. References

A. SW-846, Method 8290. Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, 1994.
B. EPA Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994, EPA 821-B-94-005.
C. VG Opusquan 2.0 Reference Manual, Issue 4, March 1995
D. Private communication from John Bill, Fisons Instruments, 04-20-95

IV. Definitions

VG, Fisons, and Micromass all refer to the same company, and may be used interchangeably.

V. Procedures

A. Personnel Qualifications
Personnel assigned to laboratory activities meet the educational, work experience, and training requirements for their positions. Records on personnel qualifications and training are maintained in personnel files accessible for review during audit activities. Training is conducted in accordance with standard operating procedures and is available to all laboratory personnel. Employees must demonstrate proficiency at specific tasks and this capability is documented and kept in a central file.

B. Apparatus and Materials
All calibration, column performance, window defining, recovery standards, internal standards spiking solutions, PARs, SRMs, etc. are obtained from commercial sources such as Cambridge Isotope Labs.

C. Instrument or Method Calibration

The GC/HRMS instrumentation is calibrated according to Method 1613, Section 10.0 (see Appendix A), with the following exceptions: the calibration solution concentrations are those specified in Method 1613, Table 4 (see Appendix A) with one additional calibration standard at concentrations equivalent to ½ the level of Method 1613's lowest calibration point.

Using the option in Method 1613, Section 10.2 (see Appendix A), only 1 µl of calibration solution or sample extract is injected per run. The samples are injected on-column.

A mid-level standard from a secondary source such as Wellington EPA-1613CS3 (or equivalent) shall be run immediately following each initial calibration curve as a verification check for the concentrations of the calibration standard solutions.

For DB-5 continuing calibration analyses, a combination solution made by Cambridge Isotope Labs, composed of calibration solution 3, window defining mixture, and tetra dioxin GC column performance mixture, is injected at the beginning and end of each 12 hour run period. This allows determination of calibration and column performance in a single run. Check the continuing calibration results against EPA Method 1613, Table 6 "VER" requirements (see Appendix A). Flag any analyte which fails this criteria. Corrective action, which may include re-tuning and re-analysis of the calibration solution or analysis of a new initial calibration curve or use of the continuing calibration response factor in calculating sample concentrations, will be taken. All corrective actions taken will be fully documented and addressed in the sample report.

D. Sample Preparation and Analysis

1. Sample Preparation

For sample preparation procedures, see SOP ASAT.II-002, *SOP for Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Sample Preparation Using Modified Methods 8290 and 1613.*

2. Sample Analysis

The GC/MS parameters listed in Method 1613, Section 10.1 (see Appendix A) are followed with the following exceptions. The GC column listed in Method 1613, Section 6.9.1 (see Appendix A) is used, but the temperature program has a different initial temperature (140°C) to allow the solvent peak to elute slowly enough to not trip the source ion gauge. The upper temperature of the ramp is held to 320°C rather than 330°C to accommodate the upper temperature limit of the column.

The ions monitored are those listed in Appendix B. A 1µL injection volume is used.

E. Data Acquisition, Calculations, and Data Reduction
Calculations are carried out using Opusquan, a software program designed for dioxin/furan analysis by VG/Micromass Co. Ltd. These calculations are the same as specified in Method 1613, Sections 17.1 to 17.4 (see Appendix A).

Estimated detection limit is calculated by measuring the sum of the heights of a native peak at the predicted retention time, times 2.5, divided by the total area of its internal standard ions, using the equation:

EDL = (F * Ni * Si * A/H * Qs) / (RRF *As * S)

Where
F = the user factor (dl_factor) in the form "fullrun" which will be set equal to 2.5 by the analyst.
Ni = the sum of the noise level of the analyte ions
Si = the sum of the "min_sig_to_noise" keyword value for each of the analyte ions
A/H = the mean area: height ratio of all ions of this analyte's internal standard
Qs = the internal standard amount
RRF = the mean relative response factor of the analyte
As = the total area of all internal standard ion peaks
S = the weight of the sample

A method blank is analyzed and processed using Opusquan in the "blank" mode. The noise factor for the natives in this blank run is then subtracted from subsequent runs, which are processed in the "quantitation" mode to obtain an accurate detection limit for each analyte in each run.

F. Computer Hardware and Software
Calculations will be carried out using Opusquan, a software program designed for dioxin/furan analysis by VG/Micromass Co. Ltd.

G. Quality Control and Quality Assurance (System Performance Criteria)
A combination calibration solution 3/window defining mixture/column performance mixture is injected at the beginning and end of each 12-hour period. This is to ensure adequate resolution of the isomeric peaks, to ascertain that the windows are set correctly to see all the isomers in each congener group, and to verify that the HRMS is adequately tuned. A PFK resolution check is also hard-copied at the beginning and end of each GC/HRMS analysis batch for non-regulatory work and every 12 hours for regulatory work to verify mass resolution. The analyst will provide an initial review/assessment of the data generated, according to SOP ASAT.II-010. A QC check sample (either CIL EDF-2526 or CIL EDF-2513) will be analyzed at least quarterly and the results maintained in the QC check sample binder.

VI. Revision History

1. This SOP was reformatted from SOP0802-01-01.
2. Added Appendices of referenced methods.
3. Version –02 adds criteria for second source calibration standard analyzed with the initial calibration, analysis of a QC check sample quarterly, and clarification on the frequency of PFK resolution checks. Many Method 8290 references were replaced with equivalent references from Method 1613.

APPENDIX A

Method 1613 References

6.8.1.1 Vacuum source for rotary evaporator equipped with shutoff valve at the evaporator and vacuum gauge.

6.8.1.2 A recirculating water pump and chiller are recommended, as use of tap water for cooling the evaporator wastes large volumes of water and can lead to inconsistent performance as water temperatures and pressures vary.

6.8.1.3 Round-bottom flask—100 mL and 500 mL or larger, with ground-glass fitting compatible with the rotary evaporator.

6.8.2 Kuderna-Danish (K-D) Concentrator

6.8.2.1 Concentrator tube—10 mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground-glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

6.8.2.2 Evaporation flask—500 mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012 or equivalent).

6.8.2.3 Snyder column—Three-ball macro (Kontes K-503000-0232, or equivalent).

6.8.2.4 Boiling chips

6.8.2.4.1 Glass or silicon carbide—Approximately 10/40 mesh, extracted with methylene chloride and baked at 450°C for one hour minimum.

6.8.2.4.2 Fluoropolymer (optional)—Extracted with methylene chloride.

6.8.2.5 Water bath—Heated, with concentric ring cover, capable of maintaining a temperature within ±2 °C, installed in a fume hood.

6.8.3 Nitrogen blowdown apparatus—Equipped with water bath controlled in the range of 30-60°C (N-Evap, Organomation Associates, Inc., South Berlin, MA, or equivalent), installed in a fume hood.

6.8.4 Sample vials

6.8.4.1 Amber glass—2-5 mL with fluoropolymer-lined screw-cap.

6.8.4.2 Glass—0.3 mL, conical, with fluoropolymer-lined screw or crimp cap.

6.9 Gas Chromatograph—Shall have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and shall meet all of the performance specifications in Section 10.

6.9.1 GC column for CDDs/CDFs and for isomer specificity for 2,3,7,8-TCDD—60 ±5 m long x 0.32 ±0.02 mm ID; 0.25 µm 5% phenyl, 94% methyl, 1% vinyl silicone bonded-phase fused-silica capillary column (J&W DB-5, or equivalent).

9.8 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

10.0 Calibration

10.1 Establish the operating conditions necessary to meet the minimum retention times for the internal standards in Section 10.2.4 and the relative retention times for the CDDs/CDFs in Table 2.

10.1.1 Suggested GC operating conditions:

Injector temperature:	270°C
Interface temperature:	290°C
Initial temperature:	200°C
Initial time:	Two minutes
Temperature program:	200-220°C, at 5°C/minute
	220°C for 16 minutes
	220-235°C, at 5°C/minute
	235°C for seven minutes
	235-330°C, at 5°C/minute

NOTE: All portions of the column that connect the GC to the ion source shall remain at or above the interface temperature specified above during analysis to preclude condensation of less volatile compounds.

Optimize GC conditions for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, IPR and OPR aliquots, and samples.

10.1.2 Mass spectrometer (MS) resolution—Obtain a selected ion current profile (SICP) of each analyte in Table 3 at the two exact m/z's specified in Table 8 and at ≥10,000 resolving power by injecting an authentic standard of the CDDs/CDFs either singly or as part of a mixture in which there is no interference between closely eluted components.

10.1.2.1 The analysis time for CDDs/CDFs may exceed 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 and a lock-mass m/z from PFK is used for drift correction. The lock-mass m/z is dependent on the exact m/z's monitored within each descriptor, as shown in Table 8. The level of PFK metered into the HRMS during analyses should be adjusted so that the amplitude of the most intense selected lock-mass m/z signal (regardless of the descriptor number) does not exceed 10% 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.

Appendix A

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source necessitating increased frequency of source cleaning.

10.1.2.2 If the HRMS has the capability to monitor resolution during the analysis, it is acceptable to terminate the analysis when the resolution falls below 10,000 to save reanalysis time.

10.1.2.3 Using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 304 (from TCDF). For each descriptor (Table 8), monitor and record the resolution and exact m/z's of three to five reference peaks covering the mass range of the descriptor. The resolution must be greater than or equal to 10,000, and the deviation between the exact m/z and the theoretical m/z (Table 8) for each exact m/z monitored must be less than 5 ppm.

10.2 Ion Abundance Ratios, Minimum Levels, Signal-to-Noise Ratios, and Absolute Retention Times—Choose an injection volume of either 1 µL or 2 µL, consistent with the capability of the HRGC/HRMS instrument. Inject a 1 µL or 2 µL aliquot of the CS1 calibration solution (Table 4) using the GC conditions from Section 10.1.1. If only 2,3,7,8-TCDD and 2,3,7,8-TCDF are to be determined, the operating conditions and specifications below apply to analysis of those compounds only.

10.2.1 Measure the SICP areas for each analyte, and compute the ion abundance ratios at the exact m/z's specified in Table 8. Compare the computed ratio to the theoretical ratio given in Table 9.

10.2.1.1 The exact m/z's to be monitored in each descriptor are shown in Table 8. Each group or descriptor shall be monitored in succession as a function of GC retention time to ensure that all CDDs/CDFs are detected. Additional m/z's may be monitored in each descriptor, and the m/z's may be divided among more than the five descriptors listed in Table 8, provided that the laboratory is able to monitor the m/z's of all the CDDs/CDFs that may elute from the GC in a given retention-time window. If only 2,3,7,8-TCDD and 2,3,7,8-TCDF are to be determined, the descriptors may be modified to include only the exact m/z's for the tetra- and penta-isomers, the diphenyl ethers, and the lock m/z's.

10.2.1.2 The mass spectrometer shall be operated in a mass-drift correction mode, using perfluorokerosene (PFK) to provide lock m/z's. The lock-mass for each group of m/z's is shown in Table 8. Each lock mass shall be monitored and shall not vary by more than ±20% throughout its respective retention time window. Variations of the lock mass by more than 20% indicate the presence of coeluting interferences that may significantly reduce the sensitivity of the mass spectrometer. Reinjection of another aliquot of the sample extract will not resolve the problem. Additional cleanup of the extract may be required to remove the interferences.

10.2.2 All CDDs/CDFs and labeled compounds in the CS1 standard shall be within the QC limits in Table 9 for their respective ion abundance ratios; otherwise, the mass spectrometer shall be adjusted and this test repeated until the m/z ratios fall within the limits specified. If the adjustment alters the resolution of the mass spectrometer, resolution shall be verified (Section 10.1.2) prior to repeat of the test.

10.2.3 Verify that the HRGC/HRMS instrument meets the minimum levels in Table 2. The peaks representing the CDDs/CDFs and labeled compounds in the CS1 calibration standard must have signal-to-noise ratios (S/N) greater than or equal to 10.0. Otherwise, the mass spectrometer shall be adjusted and this test repeated until the minimum levels in Table 2 are met.

10.2.4 The absolute retention time of $^{13}C_{12}$-1,2,3,4-TCDD (Section 7.12) shall exceed 25.0 minutes on the DB-5 column, and the retention time of $^{13}C_{12}$-1,2,3,4-TCDD shall exceed 15.0 minutes on the DB-225 column; otherwise, the GC temperature program shall be adjusted and this test repeated until the above-stated minimum retention time criteria are met.

10.3 Retention-Time Windows—Analyze the window defining mixtures (Section 7.15) using the optimized temperature program in Section 10.1. Table 5 gives the elution order (first/last) of the window-defining compounds. If 2,3,7,8-TCDD and 2,3,7,8-TCDF only are to be analyzed, this test is not required.

10.4 Isomer Specificity

10.4.1 Analyze the isomer specificity test standards (Section 7.15) using the procedure in Section 14 and the optimized conditions for sample analysis (Section 10.1.1).

10.4.2 Compute the percent valley between the GC peaks that elute most closely to the 2,3,7,8-TCDD and TCDF isomers, on their respective columns, per Figures 6 and 7.

10.4.3 Verify that the height of the valley between the most closely eluted isomers and the 2,3,7,8-substituted isomers is less than 25% (computed as 100 x/y in Figures 6 and 7). If the valley exceeds 25%, adjust the analytical conditions and repeat the test or replace the GC column and recalibrate (Sections 10.1.2 through 10.7).

10.5 Calibration by Isotope Dilution—Isotope dilution calibration is used for the 15 2,3,7,8-substituted CDDs/CDFs for which labeled compounds are added to samples prior to extraction. The reference compound for each CDD/CDF compound is shown in Table 2.

10.5.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (RR) (labeled to native) vs. concentration in standard solutions is plotted or computed using a linear regression. Relative response is determined according to the procedures described below. Five calibration points are employed.

10.5.2 The response of each CDD/CDF relative to its labeled analog is determined using the area responses of both the primary and secondary exact m/z's specified in Table 8, for each calibration standard, as follows:

Appendix A

$$RR = \frac{(A1_n + A2_n)\ C_l}{(A1_l + A2_l)\ C_n}$$

where,

$A1_n$ and $A2_n$ = The areas of the primary and secondary m/z's for the CDD/CDF.

$A1_l$ and $A2_l$ = The areas of the primary and secondary m/z's for the labeled compound.

C_l = The concentration of the labeled compound in the calibration standard (Table 4).

C_n = The concentration of the native compound in the calibration standard (Table 4).

10.5.3 To calibrate the analytical system by isotope dilution, inject a volume of calibration standards CS1 through CS5 (Section 7.13 and Table 4) identical to the volume chosen in Section 10.2, using the procedure in Section 14 and the conditions in Section 10.1.1 and Table 2. Compute the relative response (RR) at each concentration.

10.5.4 Linearity—If the relative response for any compound is constant (less than 20% coefficient of variation) over the five-point calibration range, an averaged relative response may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five-point calibration range.

10.6 Calibration by Internal Standard—The internal standard method is applied to determination of 1,2,3,7,8,9-HxCDD (Section 17.1.2), OCDF (Section 17.1.1), the non-2,3,7,8-substituted compounds, and to the determination of labeled compounds for intralaboratory statistics (Sections 9.4 and 15.5.4).

10.6.1 Response factors—Calibration requires the determination of response factors (RF) defined by the following equation:

$$RF = \frac{(A1_s + A2_s)\ C_{is}}{(A1_{is} + A2_{is})\ C_s}$$

where,

$A1_s$ and $A2_s$ = The areas of the primary and secondary m/z's for the CDD/CDF.

$A1_{is}$ and $A2_{is}$ = The areas of the primary and secondary m/z's for the internal standard

C_{is} = The concentration of the internal standard (Table 4).

C_s = The concentration of the compound in the calibration standard (Table 4).

NOTE: There is only one m/z for $^{37}Cl_4$-2,3,7,8-TCDD. See Table 8.

10.6.2 To calibrate the analytical system by internal standard, inject 1.0 µL or 2.0 µL of calibration standards CS1 through CS5 (Section 7.13 and Table 4) using the procedure in Section 14 and the conditions in Section 10.1.1 and Table 2. Compute the response factor (RF) at each concentration.

10.6.3 Linearity—If the response factor (RF) for any compound is constant (less than 35% coefficient of variation) over the five-point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five-point range.

10.7 Combined Calibration—By using calibration solutions (Section 7.13 and Table 4) containing the CDDs/CDFs and labeled compounds and the internal standards, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 15.3) by analyzing the calibration verification standard (VER, Table 4). Recalibration is required if any of the calibration verification criteria (Section 15.3) cannot be met.

10.8 Data Storage—MS data shall be collected, recorded, and stored.

10.8.1 Data acquisition—The signal at each exact m/z shall be collected repetitively throughout the monitoring period and stored on a mass storage device.

10.8.2 Response factors and multipoint calibrations—The data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibration curves. Computations of relative standard deviation (coefficient of variation) shall be used to test calibration linearity. Statistics on initial performance (Section 9.2) and ongoing performance (Section 15.5) should be computed and maintained, either on the instrument data system, or on a separate computer system.

11.0 Sample Preparation

11.1 Sample preparation involves modifying the physical form of the sample so that the CDDs/CDFs can be extracted efficiently. In general, the samples must be in a liquid form or in the form of finely divided solids in order for efficient extraction to take place. Table 10 lists the phases and suggested quantities for extraction of various sample matrices.

For samples known or expected to contain high levels of the CDDs/CDFs, the smallest sample size representative of the entire sample should be used (see Section 17.5).

For all samples, the blank and IPR/OPR aliquots must be processed through the same steps as the sample to check for contamination and losses in the preparation processes.

11.1.1 For samples that contain particles, percent solids and particle size are determined using the procedures in Sections 11.2 and 11.3, respectively.

11.1.2 Aqueous samples—Because CDDs/CDFs may be bound to suspended particles, the preparation of aqueous samples is dependent on the solids content of the sample.

11.1.2.1 Aqueous samples visibly absent particles are prepared per Section 11.4 and extracted directly using the separatory funnel or SPE techniques in Sections 12.1 or 12.2, respectively.

16.3 The ratio of the integrated areas of the two exact m/z's specified in Table 8 must be within the limit in Table 9, or within ±10% of the ratio in the midpoint (CS3) calibration or calibration verification (VER), whichever is most recent.

16.4 The relative retention time of the peak for a 2,3,7,8-substituted CDD or CDF must be within the limit in Table 2. The retention time of peaks representing non-2,3,7,8-substituted CDDs/CDFs must be within the retention time windows established in Section 10.3.

16.5 Confirmatory Analysis—Isomer specificity for 2,3,7,8-TCDF cannot be achieved on the DB-5 column. Therefore, any sample in which 2,3,7,8-TCDF is identified by analysis on a DB-5 column must have a confirmatory analysis performed on a DB-225, SP-2330, or equivalent GC column. The operating conditions in Section 10.1.1 may be adjusted to optimize the analysis on the second GC column, but the GC/MS must meet the mass resolution and calibration specifications in Section 10.

16.6 If the criteria for identification in Sections 16.1 through16.5 are not met, the CDD or CDF has not been identified and the results may not be reported for regulatory compliance purposes. If interferences preclude identification, a new aliquot of sample must be extracted, further cleaned up, and analyzed.

17.0 Quantitative Determination

17.1 Isotope Dilution Quantitation—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, concentration, and gas chromatography. Relative response (RR) values are used in conjunction with the initial calibration data described in Section 10.5 to determine concentrations directly, so long as labeled compound spiking levels are constant, using the following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_n + A2_n)\ C_l}{(A1_l + A2_l)\ RR}$$

where,

C_{ex} = The concentration of the CDD/CDF in the extract, and the other terms are as defined in Section 10.5.2.

17.1.1 Because of a potential interference, the labeled analog of OCDF is not added to the sample. Therefore, OCDF is quantitated against labeled OCDD. As a result, the concentration of OCDF is corrected for the recovery of the labeled OCDD. In instances where OCDD and OCDF behave differently during sample extraction, concentration, and cleanup procedures, this may decrease the accuracy of the OCDF results. However, given the low toxicity of this compound relative to the other dioxins and furans, the potential decrease in accuracy is not considered significant.

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17.1.2 Because $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used as an instrument internal standard (i.e., not added before extraction of the sample), it cannot be used to quantitate the 1,2,3,7,8,9-HxCDD by strict isotope dilution procedures. Therefore, 1,2,3,7,8,9-HxCDD is quantitated using the averaged response of the labeled analogs of the other two 2,3,7,8-substituted HxCDD's: 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD. As a result, the concentration of 1,2,3,7,8,9-HxCDD is corrected for the average recovery of the other two HxCDD's.

17.1.3 Any peaks representing non-2,3,7,8-substituted CDDs/CDFs are quantitated using an average of the response factors from all of the labeled 2,3,7,8-isomers at the same level of chlorination.

17.2 Internal Standard Quantitation and Labeled Compound Recovery

17.2.1 Compute the concentrations of 1,2,3,7,8,9-HxCDD, OCDF, the ^{13}C-labeled analogs and the ^{37}C-labeled cleanup standard in the extract using the response factors determined from the initial calibration data (Section 10.6) and the following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_s + A2_s)\ C_{is}}{(A1_{is} + A2_{is})\ RF}$$

where,

C_{ex} = The concentration of the CDD/CDF in the extract, and the other terms are as defined in Section 10.6.1.

NOTE: There is only one m/z for the ^{37}Cl-labeled standard.

17.2.2 Using the concentration in the extract determined above, compute the percent recovery of the ^{13}C-labeled compounds and the ^{37}C-labeled cleanup standard using the following equation:

$$Recovery\ (\%) = \frac{Concentration\ found\ (\mu g/mL)}{Concentration\ spiked\ (\mu g/mL)} \times 100$$

17.3 The concentration of a CDD/CDF in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids (Section 11.5.1), as follows:

$$Concentration\ in\ solid\ (ng/kg) = \frac{(C_{ex} \times V_{ex})}{W_s}$$

where,

C_{ex} = The concentration of the compound in the extract.
V_{ex} = The extract volume in mL.
W_s = The sample weight (dry weight) in kg.

17.4 The concentration of a CDD/CDF in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted (Section 11.4 or 11.5), as follows:

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$$\textit{Concentration in aqueous phase (pg/L)} = \frac{(C_{ex} \times V_{ex})}{V_s}$$

where,

C_{ex} = The concentration of the compound in the extract.
V_{ex} = The extract volume in mL.
V_s = The sample volume in liters.

17.5 If the SICP area at either quantitation m/z for any compound exceeds the calibration range of the system, a smaller sample aliquot is extracted.

17.5.1 For aqueous samples containing 1% solids or less, dilute 100 mL, 10 mL, etc., of sample to 1 L with reagent water and re-prepare, extract, clean up, and analyze per Sections 11 through 14.

17.5.2 For samples containing greater than 1% solids, extract an amount of sample equal to 1/10, 1/100, etc., of the amount used in Section 11.5.1. Re-prepare, extract, clean up, and analyze per Sections 11 through 14.

17.5.3 If a smaller sample size will not be representative of the entire sample, dilute the sample extract by a factor of 10, adjust the concentration of the instrument internal standard to 100 pg/µL in the extract, and analyze an aliquot of this diluted extract by the internal standard method.

17.6 Results are reported to three significant figures for the CDDs/CDFs and labeled compounds found in all standards, blanks, and samples.

17.6.1 Reporting units and levels

17.6.1.1 Aqueous samples—Report results in pg/L (parts-per-quadrillion).

17.6.1.2 Samples containing greater than 1% solids (soils, sediments, filter cake, compost)—Report results in ng/kg based on the dry weight of the sample. Report the percent solids so that the result may be corrected.

17.6.1.3 Tissues—Report results in ng/kg of wet tissue, not on the basis of the lipid content of the sample. Report the percent lipid content, so that the data user can calculate the concentration on a lipid basis if desired.

17.6.1.4 Reporting level

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TABLE 2. RETENTION TIME REFERENCES, QUANTITATION REFERENCES, RELATIVE RETENTION TIMES, AND MINIMUM LEVELS FOR CDDS AND CDFS

CDD/CDF	Retention time and quantitation reference	Relative retention time	Minimum level[1] Water (pg/L; ppq)	Solid (ng/kg; ppt)	Extract (pg/µL; ppb)
Compounds using $^{13}C_{12}$-1,2,3,4-TCDD as the injection internal standard					
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF	0.999–1.003	10	1	0.5
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD	0.999–1.002	10	1	0.5
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF	0.999–1.002	50	5	2.5
2,3,4,7,8-PeCDF	$^{13}C_{12}$-2,3,4,7,8-PeCDF	0.999–1.002	50	5	2.5
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD	0.999–1.002	50	5	2.5
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD	0.923–1.103			
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.976–1.043			
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.989–1.052			
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.000–1.425			
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.011–1.526			
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD	1.000–1.567			
Compounds using $^{13}C_{12}$-1,2,3,7,8,9-HxCDD as the injection internal standard					
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	0.999–1.001	50	5	2.5
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	0.997–1.005	50	5	2.5
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	0.999–1.001	50	5	2.5
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-2,3,4,6,7,8,-HxCDF	0.999–1.001	50	5	2.5
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	0.999–1.001	50	5	2.5
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8,-HxCDD	0.998–1.004	50	5	2.5
1,2,3,7,8,9-HxCDD	—[2]	1.000–1.019	50	5	2.5
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	0.999–1.001	50	5	2.5
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	0.999–1.001	50	5	2.5
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	0.999–1.001	50	5	2.5
OCDF	$^{13}C_{12}$-OCDD	0.999–1.008	100	10	5.0
OCDD	$^{13}C_{12}$-OCDD	0.999–1.001	100	10	5.0
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.944–0.970			
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.949–0.975			
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.977–1.047			
$^{13}C_{12}$-2,3,4,6,7,8,-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.959–1.021			
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.977–1.000			
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.981–1.003			
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.043–1.085			

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TABLE 2. RETENTION TIME REFERENCES, QUANTITATION REFERENCES, RELATIVE RETENTION TIMES, AND MINIMUM LEVELS FOR CDDS AND CDFS

CDD/CDF	Retention time and quantitation reference	Relative retention time	Minimum level[1] Water (pg/L; ppq)	Solid (ng/kg; ppt)	Extract (pg/µL; ppb)
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.057–1.151			
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.086–1.110			
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.032–1.311			

[1]The Minimum Level (ML) for each analyte is defined as the level at which the entire analytical system must give a recognizable signal and acceptable calibration point. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

[2]The retention time reference for 1,2,3,7,8,9-HxCDD is[3] C_{12}-1,2,3,6,7,8-HxCDD, and 1,2,3,7,8,9-HxCDD is quantified using the averaged responses for $^{13}C_{12}$-1,2,3,4,7,8-HxCDD and $^{13}C_{12}$-1,2,3,6,7,8-HxCDD.

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TABLE 4. CONCENTRATION OF CDDS/CDFS IN CALIBRATION AND CALIBRATION VERIFICATION SOLUTIONS [1] (section 15.3)

	CDD/CDF	CS2 (ng/mL)	CS3 (ng/mL)	CS4 (ng/mL)	CS5 (ng/mL)
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
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
$^{13}C_{12}$-PeCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100
$^{13}C_{12}$-OCDD	200	200	200	200	200
Cleanup Standard					
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
Internal Standards					
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

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TABLE 6. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS WHEN ALL CDDS/CDFS ARE TESTED[1]

CDD/CDF	Test Conc. (ng/mL)	IPR[2,3] s (ng/mL)	IPR[2,3] X (ng/mL)	OPR (ng/mL)	VER (ng/mL)
2,3,7,8-TCDD	10	2.8	8.3–12.9	6.7–15.8	7.8–12.9
2,3,7,8-TCDF	10	2.0	8.7–13.7	7.5–15.8	8.4–12.0
1,2,3,7,8-PeCDD	50	7.5	38–66	35–71	39–65
1,2,3,7,8-PeCDF	50	7.5	43–62	40–67	41–60
2,3,4,7,8-PeCDF	50	8.6	36–75	34–80	41–61
1,2,3,4,7,8-HxCDD	50	9.4	39–76	35–82	39–64
1,2,3,6,7,8-HxCDD	50	7.7	42–62	38–67	39–64
1,2,3,7,8,9-HxCDD	50	11.1	37–71	32–81	41–61
1,2,3,4,7,8-HxCDF	50	8.7	41–59	36–67	45–56
1,2,3,6,7,8-HxCDF	50	6.7	46–60	42–65	44–57
1,2,3,7,8,9-HxCDF	50	6.4	42–61	39–65	45–56
2,3,4,6,7,8-HxCDF	50	7.4	37–74	35–78	44–57
1,2,3,4,6,7,8-HpCDD	50	7.7	38–65	35–70	43–58
1,2,3,4,6,7,8-HpCDF	50	6.3	45–56	41–61	45–55
1,2,3,4,7,8,9-HpCDF	50	8.1	43–63	39–69	43–58
OCDD	100	19	89–127	78–144	79–126
OCDF	100	27	74–146	63–170	63–159
$^{13}C_{12}$-2,3,7,8-TCDD	100	37	28–134	20–175	82–121
$^{13}C_{12}$-2,3,7,8-TCDF	100	35	31–113	22–152	71–140
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	39	27–184	21–227	62–160
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	34	27–156	21–192	76–130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	38	16–279	13–328	77–130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	41	29–147	21–193	85–117
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	38	34–122	25–163	85–118
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	43	27–152	19–202	76–131
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	35	30–122	21–159	70–143
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	40	24–157	17–205	74–135
$^{13}C_{12}$-2,3,4,6,7,8,-HxCDF	100	37	29–136	22–176	73–137
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	35	34–129	26–166	72–138
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	41	32–110	21–158	78–129
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	40	28–141	20–186	77–129
$^{13}C_{12}$-OCDD	200	95	41–276	26–397	96–415
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.6	3.9–15.4	3.1–19.1	7.9–12.7

[1] All specifications are given as concentration in the final extract, assuming a 20 µL volume.
[2] s = standard deviation of the concentration.
[3] X = average concentration.

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TABLE 7A. LABELED COMPOUND RECOVERY IN SAMPLES WHEN ONLY TETRA COMPOUNDS ARE TESTED

Compound	Test Conc. (ng/mL)	Labeled compound recovery (ng/mL) [1]	(%)
$^{13}C_{12}$-2,3,7,8-TCDD	100	31-137	31-137
$^{13}C_{12}$-2,3,7,8-TCDF	100	29-140	29-140
$^{37}Cl_4$-2,3,7,8-TCDD	10	4.2-16.4	42-164

[1] Specification given as concentration in the final extract, assuming a 20 µL volume.

TABLE 8. DESCRIPTORS, EXACT M/Z's, M/Z TYPES, AND ELEMENTAL COMPOSITIONS OF THE CDDs AND CDFs

Descriptor	Exact M/Z [1]	M/Z Type	Elemental Composition	Substance [2]
1	292.9825	Lock	$C_7 F_{11}$	PFK
	303.9016	M	$C_{12} H_4 {}^{35}Cl_4 O$	TCDF
	305.8987	M+2	$C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O$	TCDF
	315.9419	M	$^{13}C_{12} H_4 {}^{35}Cl_4 O$	TCDF[3]
	317.9389	M+2	$^{13}C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O$	TCDF[3]
	319.8965	M	$C_{12} H_4 {}^{35}Cl_4 Q$	TCDD
	321.8936	M+2	$C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl Q$	TCDD
	327.8847	M	$C_{12} H_4 {}^{37}Cl_4 Q$	TCDD[4]
	330.9792	QC	$C_7 F_{13}$	PFK
	331.9368	M	$^{13}C_{12} H_4 {}^{35}Cl_4 Q$	TCDD[3]
	333.9339	M+2	$^{13}C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl Q$	TCDD[3]
	375.8364	M+2	$C_{12} H_4 {}^{35}Cl_5 {}^{37}Cl O$	HxCDPE
2	339.8597	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O$	PeCDF
	341.8567	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF
	351.9000	M+2	$^{13}C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O$	PeCDF
	353.8970	M+4	$^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF [3]
	354.9792	Lock	$C_9 F_{13}$	PFK
	355.8546	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl Q$	PeCDD
	357.8516	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 Q$	PeCDD
	367.8949	M+2	$^{13}C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl Q$	PeCDD [3]
	369.8919	M+4	$^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 Q$	PeCDD [3]
	409.7974	M+2	$C_{12} H_3 {}^{35}Cl_5 {}^{37}Cl O$	HpCDPE
3	373.8208	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF
	375.8178	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O$	HxCDF
	383.8639	M	$^{13}C_{12} H_2 {}^{35}Cl_6 O$	HxCDF [3]
	385.8610	M+2	$^{13}C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF [3]
	389.8157	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl Q$	HxCDD
	391.8127	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 Q$	HxCDD

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TABLE 8. DESCRIPTORS, EXACT M/Z's, M/Z TYPES, AND ELEMENTAL COMPOSITIONS OF THE CDDs AND CDFs

Descriptor	Exact M/Z [1]	M/Z Type	Elemental Composition	Substanee [2]
	392.9760	Lock	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_3{}^{37}ClO$	HxCDD [3]
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDD [3]
	430.9729	QC	C_9F_{17}	PFK
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
4	407.7818	M+2	$C_{12}H{}^{35}Cl_3{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H{}^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H{}^{35}Cl_7O$	HpCDF [3]
	419.8220	M+2	$^{13}C_{12}H{}^{35}Cl_6{}^{37}ClO$	HpCDF [3]
	423.7766	M+2	$C_{12}H{}^{35}Cl_6{}^{37}ClO$	HpCDD
	425.7737	M+4	$C_{12}H{}^{35}Cl_5{}^{37}Cl_2O$	HpCDD
	430.9729	Lock	C_9F_{17}	PFK
	435.8169	M+2	$^{13}C_{12}H{}^{35}Cl_6{}^{37}ClO$	HpCDD [3]
	437.8140	M+4	$^{13}C_{12}H{}^{35}Cl_5{}^{37}Cl_2O$	HpCDD [3]
	479.7165	M+4	$C_{12}H{}^{35}Cl_7{}^{37}Cl_2O$	NCDPE
5	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	Lock	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDD [3]
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDD [3]
	513.6775	M+4	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE

[1] Nuclidic masses used:

H = 1.007825 C = 12.00000 ^{13}C = 13.003355 F = 18.9984
O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

[2]
TCDD = Tetrachlorodibenzo-*p*-dioxin
PeCDD = Pentachlorodibenzo-*p*-dioxin
HxCDD = Hexachlorodibenzo-*p*-dioxin
HpCDD = Heptachlorodibenzo-*p*-dioxin
OCDD = Octachlorodibenzo-*p*-dioxin
HxCDPE = Hexachlorodiphenyl ether
OCDPE = Octachlorodiphenyl ether
DCDPE = Decachlorodiphenyl ether
TCDF = Tetrachlorodibenzofuran
PeCDF = Pentachlorodibenzofuran
HxCDF = Hexachlorodibenzofuran
HpCDF = Heptachlorodibenzofuran
OCDF = Octachlorodibenzofuran
HpCDPE = Heptachlorodiphenyl ether
NCDPE = Nonachlorodiphenyl ether
PFK = Perfluorokerosene

[3] Labeled compound.

[4] There is only one m/z for $^{37}Cl_4$-2,3,7,8,-TCDD (cleanup standard).

Appendix A

TABLE 9. THEORETICAL ION ABUNDANCE RATIOS AND QC LIMITS

Number of Chlorine Atoms	M/Z's Forming Ratio	Theoretical Ratio	QC Limit [1]	
			Lower	Upper
4 [2]	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 [3]	M/(M+2)	0.51	0.43	0.59
7	(M+2)/(M+4)	1.05	0.88	1.20
7 [4]	M/(M+2)	0.44	0.37	0.51
8	(M+2)/(M+4)	0.89	0.76	1.02

[1] QC limits represent ±15% windows around the theoretical ion abundance ratios.
[2] Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard).
[3] Used for $^{13}C_{12}$-HxCDF only.
[4] Used for $^{13}C_{12}$-HpCDF only.

APPENDIX B

Mass Descriptors for Modified Method 8290 and 1613

Appendix B

Experiment: DIOXIN (5 Functions)

Operator : User
Date : 19-OCT-2000 12:12:15
Instrument : AutoSpec

Function 1

Type	: SIR Voltage
Calibration file used	: TEST1005C_1
High mass	: 375.8
Low mass	: 293.0
Resolution	: 10000
Ionisation mode	: EI+
Accelerating Voltage	: 8000.0V
Magnet 1 control	: Current
Start Time	: 19:00
End Time	: 28:25
Fast lock	: On
Number of channels	: 12
Cycle time (ms)	: 1060

Channel	Mass	Ch Time (ms)	I/ch Time (ms)
1 (Lock)	292.9825	80	20
Primary Span Lock (Peaks) 2.00			
Secondary Span Lock (Peaks) 2.00			
Lock Level (mV) 0			
Step Lock (Peaks) 0.020			
2	303.9016	80	10
3	305.8987	80	10
4	315.9419	80	10
5	317.9389	80	10
6	319.8965	80	10
7	321.8936	80	10
8	327.8847	80	10
9	330.9792	80	10
10	331.9368	80	10
11	333.9339	80	10
12	375.8364	50	10

Septum flow : Off

Appendix B

Experiment: DIOXIN (5 Functions)

Operator : User
Date : 19-OCT-2000 12:12:15
Instrument : AutoSpec

Function 2

Type	: SIR Voltage
Calibration file used	: TEST1005C_2
High mass	: 409.8
Low mass	: 339.9
Resolution	: 10000
Ionisation mode	: EI+
Accelerating Voltage	: 8000.0V
Magnet 1 control	: Current
Start Time	: 28:25
End Time	: 37:30
Fast lock	: On
Number of channels	: 11
Cycle time (ms)	: 1000

Channel	Mass	Ch Time (ms)	I/ch Time (ms)
1	339.8597	80	20
2	341.8567	80	10
3	351.9000	80	10
4	353.8970	80	10
5 (Lock)	354.9792	80	10
Primary Span Lock (Peaks) 2.00			
Secondary Span Lock (Peaks) 2.00			
Lock Level (mV) 0			
Step Lock (Peaks) 0.020			
6	354.9792	80	10
7	355.8546	80	10
8	357.8516	80	10
9	367.8949	80	10
10	369.8919	80	10
11	409.7974	80	10

Septum flow	: Off
Repeats	: 1

Appendix B

Experiment: DIOXIN (5 Functions)

Operator : User
Date : 19-OCT-2000 12:12:15
Instrument : AutoSpec

Function 3

Type	: SIR Voltage
Calibration file used	: TEST1005C_3
High mass	: 445.8
Low mass	: 373.8
Resolution	: 10000
Ionisation mode	: EI+
Accelerating Voltage	: 8000.0V
Magnet 1 control	: Current
Start Time	: 37:30
End Time	: 43:30
Fast lock	: On
Number of channels	: 11
Cycle time (ms)	: 1000

Channel	Mass	Ch Time (ms)	I/ch Time (ms)
1	373.8208	80	20
2	375.8178	80	10
3	383.8639	80	10
4	385.8610	80	10
5	389.8157	80	10
6	391.8127	80	10
7 (Lock)	392.9760	80	10
Primary Span Lock (Peaks)	2.00		
Secondary Span Lock (Peaks)	2.00		
Lock Level (mV)	0		
Step Lock (Peaks)	0.020		
8	401.8559	80	10
9	403.8529	80	10
10	430.9729	80	10
11	445.7555	80	10

Septum flow	: Off
Repeats	: 1

Appendix B

Experiment: DIOXIN (5 Functions)

Operator : User
Date : 19-OCT-2000 12:12:15
Instrument : AutoSpec

Function 4

Type	: SIR Voltage
Calibration file used	: TEST1005C_4
High mass	: 479.7
Low mass	: 407.8
Resolution	: 10000
Ionisation mode	: EI+
Accelerating Voltage	: 8000.0V
Magnet 1 control	: Current
Start Time	: 43:30
End Time	: 48:00
Fast lock	: On
Number of channels	: 11
Cycle time (ms)	: 1000

Channel	Mass	Ch Time (ms)	I/ch Time (ms)
1	407.7818	80	20
2	409.7789	80	10
3	417.8253	80	10
4	419.8220	80	10
5	423.7766	80	10
6	425.7737	80	10
7 (Lock)	430.9729	80	10
Primary Span Lock (Peaks)	2.00		
Secondary Span Lock (Peaks)	2.00		
Lock Level (mV)	0		
Step Lock (Peaks)	0.020		
8	430.9729	80	10
9	435.8169	80	10
10	437.8140	80	10
11	479.7165	80	10

Septum flow	: Off
Repeats	: 1

Appendix B

Experiment: DIOXIN (5 Functions)

Operator : User
Date : 19-OCT-2000 12:12:15
Instrument : AutoSpec

Function 5

Type	: SIR Voltage
Calibration file used	: TEST1005C_5
High mass	: 513.7
Low mass	: 441.7
Resolution	: 10000
Ionisation mode	: EI+
Accelerating Voltage	: 8000.0V
Magnet 1 control	: Current
Start Time	: 48:00
End Time	: 52:00
Fast lock	: On
Number of channels	: 9
Cycle time (ms)	: 1000

Channel	Mass	Ch Time (ms)	I/ch Time (ms)
1	441.7428	100	20
2 (Lock)	442.9728	100	10
Primary Span Lock (Peaks) 2.00			
Secondary Span Lock (Peaks) 2.00			
Lock Level (mV) 0			
Step Lock (Peaks) 0.020			
3	442.9728	100	10
4	443.7399	100	10
5	457.7377	100	10
6	459.7348	100	10
7	469.7779	100	10
8	471.7750	100	10
9	513.6775	100	10

Septum flow	: Off
Repeats	: 1

OCT 3 0 2000

STANDARD OPERATING PROCEDURE (SOP) FOR POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) SAMPLE PREPARATION USING MODIFIED METHODS 8290 AND 1613

Originated by: _____ Date: 10/30/00

Approved by: Karen L. Tracy _____ Date: 10/30/00
Technical Reviewer

Approved by: Karen Riggs _____ Date: 10/30/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

Charles D. Laurie _____ Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for preparing samples for PCDD/PCDF analysis. These procedures follow general guidelines described in EPA Methods 8290 and 1613, with some minor modifications/improvements. The purpose of this SOP is to provide a description of PCDD/PCDF sample preparation activities using modified Method 8290 and Method 1613 procedures and covers the following:

- Personnel qualifications
- Sample collection, handling and preservation
- Sample extraction and internal standard spiking
- Extract cleanup
- Final concentration activities.

III. References

A. SW-846, Method 8290. Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRCG/HRMS), Revision 0, 1994.

B. EPA Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994, EPA 821-B-94-005.

C. SOP ASAT.II-005-00. Standard Operating Procedure (SOP) for Polychlorinated Dibenzo-p-dioxin/Polychlorinated dibenzofuran (PCDD/PCDF) Desiccating Agent and Adsorbent Preparation and Storage.

D. SOP ASAT.II-006-00. Standard Operating Procedure (SOP) for Polychlorinated dibenzo-p-dioxin/Polychlorinated dibenzofuran (PCDD/PCDF) Standards and Reagents Preparation and Storage for Modified Method 8290 Analysis.

IV. Definitions

All references in this section are to SW846 Method 8290 unless otherwise indicated.

V. Procedures

A. Personnel Qualifications

Personnel assigned to laboratory activities meet the educational, work experience, and training requirements for their positions. Records on personnel qualifications and training are maintained in personnel files accessible for review during audit activities. Training is conducted in accordance with standard operating procedures and is available to all laboratory personnel. Employees must demonstrate proficiency at specific tasks and this capability is documented and kept in a central file.

B. Sample Collection, Handling, and Preservation

Samples will be treated as described in Method 1613 Section 8.0 (see Appendix A) or as required by the client.

C. Sample Extraction and Spiking

The procedures for tissue, sediment, and water samples are described below. A quality control (QC) sample (Cambridge Isotope standard reference material EDF-2526 or EDF-2513) is extracted with field samples at a minimum of once every quarter. Procedures for other matrices will be distributed to staff in project specific workplans.

1. Sediment and Tissue Samples

   a. All glassware are detergent-washed, rinsed, solvent-rinsed (methanol, dichloromethane) and muffled overnight at 450°C. Soxhlets are assembled with 250-mL round bottom flasks (RB) and are also pre-extracted with the extraction solvent for three hours prior to use. This extract is archived until sample analysis is complete.

   b. The samples are mixed well and weighed into muffled 4-oz jars. For sediments, 10 g wet weight is typically used. For tissues, 20 g wet weight is required if lipid content is being determined; otherwise, 10 g wet weight is used. The samples are mixed with 5 to 10 g Varian Hydromatrix drying agent. The sample/Hydromatrix mixture is allowed to dry with occasional stirring until the mixture is free flowing and powdery. A method blank is prepared by adding Hydromatrix without sample to ensure that laboratory procedures and reagents are free from contamination.

   c. Approximately 1/4 inch of silica gel is added to the bottom of a pre-extracted glass thimble. The sample/Hydromatrix mixture is then added to the thimble. All samples are spiked with 1 ml of IS spiking solution. The matrix spike samples are spiked with 1 ml precision and recovery (PAR) spiking solution. The method detection limit verification samples (MDL) are spiked with 250 µL PAR spiking solution. Boiling chips are added, the Soxhlets are assembled, and the extractions started. Sediments are extracted using toluene. Tissues are extracted using a 50/50 solution of dichloromethane (DCM) and hexane.

   d. After 18 hours of extraction, the Soxhlets are allowed to cool and drain back into the 250-mL RB. New boiling chips are added and the extracts are concentrated to approximately 20 ml using Snyder columns.

2. Water Samples

   a. Water Samples with < 1% Solids

      1. All glassware are detergent-washed, rinsed, solvent-rinsed (methanol, dichloromethane) and muffled overnight at 450°C.

      2. The samples are allowed to come to room temperature.

3. A permanent marker is used to mark the sample volume on each sample container. The samples are then visibly inspected for solids.

4. If the samples are visually determined to be at or below 1% solids, the samples are transferred to a DCM rinsed 2-L separatory-funnel with 3 x 20-mL DCM rinses of the sample container.

5. A method blank, for water with < 1% solids, is prepared by adding 1 L reagent water and 60 ml of DCM to a DCM rinsed 2-L separatory-funnel.

6. All samples are spiked with 1 ml internal standard (IS) spiking solution while they are still in the sample container.

7. Matrix spike samples are also spiked with 1 ml PAR spiking solution.

8. Method detection limit (MDL) samples are also spiked with 250 µL PAR spiking solution.

9. The separatory-funnels are shaken for 2 minutes with periodic venting.

10. The phases are allowed to separate for 20 minutes and the bottom layer (DCM) is drained through a funnel containing approximately 15 g of DCM rinsed sodium sulfate into a 200-mL turboTube.

11. The sodium sulfate is then rinsed with an additional 20 ml of DCM and also collected in the 200-mL turboTube.

12. The 60 ml DCM extraction is repeated 2 more times, each time draining the DCM through the sodium sulfate into the 200-mL turboTube.

13. Using a TurboVap, the extracts are solvent exchanged into 4 ml of hexane.

14. The sample containers are filled to the marked volume with DI water. The DI water is then poured into a graduated cylinder and the volume is recorded

b. Waters with > 1% Solids

1. All glassware are detergent-washed, rinsed, solvent-rinsed (methanol, dichloromethane) and muffled overnight at 450°C.

2. The samples are allowed to come to room temperature.

3. A permanent marker is used to mark the sample volume on each sample container. The samples are then visibly inspected for solids.

4. If the samples are visually determined to be above 1% solids, each sample is filtered through a separate muffled 0.45 µm glass fiber filter.

5. All samples are spiked with 1 ml IS spiking solution while they are still in the sample container.

6. Matrix spike samples are also spiked with 1 ml PAR spiking solution while they are still in the sample container.

7. Method detection limit (MDL) samples are also spiked with 250 µL PAR spiking solution while they are still in the sample container.

8. Samples are shaken vigorously after they have been spiked with the appropriate spiking solutions and then allowed to sit at room temperature for 1 to 2 hours to allow the spike solutions to distribute through out the sample.

9. When samples have been received without any headspace in the sample container, approximately 20% of the sample is transferred to a glass, muffled secondary container before any of the spiking solutions are added to the sample container. The remaining sample is then spike according to the procedure above.

10. The glass fiber filter is wetted and sealed with reagent grade water onto the filtering apparatus. The samples are slowly poured through the glass fiber filters taking care not to disturb any settled solids at the bottom of the sample container. When the majority of the sample has passed through the filter, the remaining portion of the sample in the container is swirled to re-suspend the solids and then also poured through the glass fiber filter. A small amount of reagent grade water is used to rinse the sample container to ensure the complete transfer of solids to the filter.

11. However, if the solid content in the samples is visually determined to be too great for the 0.45 µm glass fiber filter, the samples are centrifuged in order to separate the water layer from the solids layer. The aqueous layer is then poured through a muffled glass fiber filter that has been wetted with reagent grade water. The sediment portion is transferred into a muffled 4-oz glass jar via a muffled stainless steal spatula and the centrifuge tube is rinsed with reagent grade water to remove any remaining solids. The centrifuge tube rinse water is poured through the same muffled glass fiber filter as the decanted aqueous layer. The solid

fraction is combined with Hydromatrix to form a free flowing mixture and then combined with the glass fiber filter in a Soxhlet apparatus. The combined solid fraction/glass fiber filter is extracted using the sediment extraction procedure above (step 1) without the addition of any spike solutions.

12. After the extraction has been completed, the concentrated solids extract is solvent exchanged using a rotoVap into 4 ml of hexane.

13. A method blank, for waters with > 1% solids, is prepared by pouring 1 L reagent water through a glass fiber filter, Soxhlet extracting the filter as described in the previous step. The 1 L of filtered reagent water and 60 ml of DCM are added to a DCM rinsed 2-L separatory-funnel.

14. The separatory-funnels are shaken for 2 minutes with periodic venting.

15. The phases are allowed to separate for 20 minutes and the bottom layer (DCM) is drained through a funnel containing approximately 15 g of DCM rinsed sodium sulfate into a 200-mL turboTube.

16. The sodium sulfate is then rinsed with an additional 20 ml of DCM and also collected in the 200-mL turboTube.

17. The 60 ml DCM extraction is repeated 2 more times, each time draining the DCM through the sodium sulfate into the 200-mL turboTube.

18. Using a TurboVap, the extracts are solvent exchanged into 4 ml of hexane.

19. The sample containers are filled to the marked volume with DI water. The DI water is then poured into a graduated cylinder and the volume is recorded

20. The water and solid fractions are combined before proceeding to any cleanup procedures.

D. Extract Cleanup

1. Partition

If the sample extract is suspected to be of high lipid content, the optional bulk acid silica cleanup (step 4 below) can be used prior to acid/base partitioning. If bulk acid cleanup is used, the cleanup standard (CS) referred to below must be added prior to the bulk acid silica cleanup rather than after transfer to the separatory funnels.

Acid/Base Partitioning:

a. The concentrated extracts are transferred to a hexane rinsed 125-mL separatory funnel using muffled pasteur pipettes with 3 x 4-mL hexane rinses of the round bottom (RB).

b. The sample extracts are spiked with 1 ml of cleanup standard (CS) spiking solution and brought up to approximately 50 ml with hexane.

c. The extracts are partitioned against one 30-mL aliquot and various 20-mL aliquots of concentrated sulfuric acid, until there is no visible color or a maximum of 4 acid washes has been reached.

d. The extracts are washed with 20 ml of 5% sodium chloride solution (NaCl), 15 ml of 20% potassium hydroxide (KOH) solution (for tissues, the first 20 ml 5% NaCl wash and the 20% KOH wash are omitted) and 2 x 20-mL of 5% NaCl solution.

e. The washed extracts are drained through a funnel containing hexane rinsed sodium sulfate with 3 x 6-mL hexane rinses of the separatory funnel and are collected in a 250-mL RB or a 200-mL turboTube.

f. The sodium sulfate is then rinsed with 20 ml hexane and collected in the same RB or turboTube.

If the extract is still discolored after the samples have been through the partitioning procedure, then follow with the optional acid silica column cleanup procedure (step 5 below) prior to performing the stack column cleanup procedure.

2. Silica/Alumina Column Cleanup (Stack Column Cleanup)

a. The sample extracts are rotovapped or turboVapped to near dry in a 46°C water bath and brought up in 4 ml of hexane.

b. Acid/base silica columns and alumina columns are prepared by cutting the tops from 25-mL muffled pipettes, plugging with muffled glass wool, adding activated silica to the 22-mark, base silica to the 18-mark, silica to the 16-mark, acid silica to the 1-mark and approximately ½ inch of muffled sodium sulfate on top.

c. Basic alumina columns are prepared by cutting the tops from 25-mL muffled pipettes, plugging with muffled glass wool; adding 6 g activated basic alumina and approximately ½ inch of muffled sodium sulfate on top.

d. Acid/base silica columns and alumina columns are rinsed separately with 20 ml of hexane. As the last of the hexane rinse approaches the top of the packing, the acid/base silica columns are stacked on top of the alumina columns. The sample extracts are applied to the columns using muffled Pasteur pipettes with 3 x 4-mL hexane rinses of the turboTube or RB. The stacked columns are eluted with 100 ml of hexane and collected in a muffled 125-mL glass jar. As the last of the 100 ml hexane eluant passes through the stacked columns the columns are separated and the alumina column is eluted with 40 ml DCM/hexane (50/50) and collected in a turboTube.

e. If the acid layer in the acid / base silica column is badly discolored through the entire acid silica layer, the stacked silica / alumina column cleanup is repeated once more.

3. Carbon Column Cleanup

a. The sample extracts are turboVapped to near dryness in a 46°C water bath and brought up in 1 ml of hexane.

b. Carbon/Celite columns are prepared by cutting the top and bottom from a 10 ml muffled pipette and fire polishing both ends, plugging one end with muffled glass wool, adding 0.55 g of carbon/celite mixture and plugging with muffled glass wool.

c. The columns are rinsed with 5 ml of toluene, 2 ml of 15:4:1 (DCM/methanol/toluene), 1 ml of 1:1 (cyclohexane/DCM), and 5 ml of hexane. Each rinse is added as each previous rinse approaches the top of the packed bed in order to avoid any mixing and dilution of solvents.

d. The sample extracts are applied using muffled Pasteur pipettes with 2 x 1-mL, 1 x 2-mL and 2 x 3-mL hexane rinses of the TurboVap tube.

e. The column is then rinsed with 2 ml of 1:1 and 2 ml of 15:4:1. As the last of the 15:4:1 passes through the column, the column is inverted and eluted with 30 ml of toluene. The eluate is collected in a 125-mL RB.

4. Bulk Acid Silica Cleanup (optional)

a. Extracts are transferred to a 500 ml Erlenmeyer flask that contains an $1^1/_2$ inch stir bar. The RB is rinsed 3 times with 4 ml of hexane and the rinses are added to the Erlenmeyer flask.

b. The extract volume is brought up to 200 ml by using hexane to wash down the walls of the Erlenmeyer flask.

c. The flask is placed upon a stir plate, and the stir plate is started.

d. Five 10 g aliquots of acid silica are added to the extract and the extracts are stirred for 2 hours.

e. After the 2 hours of stirring, the hexane layer is decanted into a 500 ml RB (or 200 ml turboTube) through $NaSO_4$ that has been pre-rinsed with hexane.

f. 50 ml of hexane are added to the acid silica in the Erlenmeyer flask by washing the walls of the Erlenmeyer flask, stirred for an additional 10 minutes, and then this hexane layer is decanted in the RB (or turboTube) combining it with the original layer of hexane. This procedure is repeated two more times.

g. The NaSO4 is then rinsed with 20 ml of hexane. The hexane is also collected in the same RB (or turboTube) as the sample extract.

Procedures for the use of less acid silica are taken from Method 1613, Section 13.7.2.4 (see Appendix A).

5. Acid Silica Column Cleanup (optional)
   a. The sample extracts are rotovapped or turbovapped to near dry in a 46°C water bath and brought up in 4 ml of hexane.

   b. Acid silica columns are prepared by cutting the tops from 25-mL muffled pipettes, plugging with muffled glass wool, and activated silica to the 22-mark, acid silica to the 1-mark and approximately 1/2 inch sodium sulfate on top.

   c. The column is rinsed with 20 ml hexane. As the last of the hexane rinse approaches the top of the bed packing, a turboTube is placed under the column and the sample extracts are applied using muffled Pasteur pipettes with 3 x 4-mL hexane rinses of the RB or turboTube.

   d. The column is eluted with 90 ml of hexane and collected in the turboTube.

   If the acid silica layer is badly discolored, the acid silica column cleanup is repeated.

E. Final Concentration

1. The sample extracts are rotovapped to near dry in a 48°C water bath and brought up in 1 ml of hexane.

2. Muffled concentrator tubes are rinsed with DCM by vortexing for approximately 30 seconds and then the rinse is discarded.

3. After the concentrator tubes have dried, 20 µL of nonane is pipetted into the tubes using an Eppendorf pipette. The tubes are lightly tapped to remove any air bubbles and the meniscus is marked on the tube using permanent marker.

4. 200 µL of hexane is then pipetted into the tubes using an Eppendorf pipette and the tubes are lightly tapped to remove any air bubbles. The meniscus of the hexane is then marked on the tube using permanent marker.

5. The concentrator tubes are labeled with sample Ids using a permanent marker.

6. Leaving the solvents in the tubes, the sample extracts are transferred using muffled Pasteur pipettes with 3 x 1-mL hexane rinses of the RB.

7. The sample extracts are then blown down under nitrogen in a 45°C sand bath to the marked hexane meniscus.

8. The RB is then rinsed with 1 x 1-mL DCM and transferred to the concentrator tube, being sure to rinse the walls of the tube with the DCM rinses. This is also blown down under nitrogen to the marked hexane meniscus.

9. The RB is then rinsed with 1 x 0.5-mL DCM and transferred to the concentrator tube, being sure to rinse the walls of the tube with the DCM rinses. This rinse is also blown down to the marked hexane meniscus.

10. The sample extracts are then spiked with 10 µL RS spiking solution, vortexed for approximately 30 seconds, capped, and a sample information archive label affixed to each concentrator tube. If not going directly to analysis, the tubes containing the extracts are capped and stored in a freezer.

11. Prior to analysis, the labeled concentrator tubes are removed from the freezer and allowed to come to room temperature.

12. Label the pre-assembled GC vials that contain 300-µL inserts. There must be a gap in the labels on the vials for liquid volume inspection.

13. Put squeeze balls on pipettes. Visually inspect the concentrator tubes to ensure that at least a 200 µL volume is contained. If not, adjust to the marked line with DCM.

14. Vortex the concentrator tubes, making sure that the liquid rinses the side walls. 200 µL will reach about halfway up the tube.

15. Transfer the contents of the concentrator tubes to the correct GC vial inserts. It is not possible with a disposable to completely transfer all of the material (about 10 μL will remain).

16. Cap the GC vial and place it in a GC vial container. Continue with the remaining samples. When all original samples have been transferred, start the nitrogen blow down procedure.

17. On an N-Evap, push the Teflon tubing onto the ¼" metal tube/valve assemblies, and install new glass pipettes.

18. Check to see that the nitrogen is turned on at the tank, and that the pressure is set to 40 psi on the second stage of the tank regulator, and approximately 5 pi on the regulator just before the N-Evap.

19. Uncap the GC vials and place them in the sample holders, with the labeled gap towards you so that the solvent level may be inspected. Push the pipettes down until the tip just rests inside the vial opening. Check each blowdown valve to see that the flow is correct, so that evaporation occurs, but the level of the liquid is not greatly deformed. Blow the sample extracts down until they are almost dry.

20. Adjust the sample volume to 20 μL with nonane using a syringe. Vortex the GC vial and store it in the GC vial container. If the samples are not going directly to the instrument for analysis, store them in a freezer.

VI. Revision History

1. The extraction of a quarterly QC sample was added.
2. The procedure for spiking IS in water samples with < 1% solids was updated.

Appendix A

Method 1613 References

Appendix A

determined. In this case, an isomer-specificity test standard containing the most closely eluted isomers listed in Table 5 (CIL EDF-4033, or equivalent) may be used.

7.16 QC Check Sample—A QC Check Sample should be obtained from a source independent of the calibration standards. Ideally, this check sample would be a certified reference material containing the CDDs/CDFs in known concentrations in a sample matrix similar to the matrix under test.

7.17 Stability of Solutions—Standard solutions used for quantitative purposes (Sections 7.9 through 7.15) should be analyzed periodically, and should be assayed against reference standards (Section 7.8.3) before further use.

8.0 Sample Collection, Preservation, Storage, and Holding Times

8.1 Collect samples in amber glass containers following conventional sampling practices (Reference 16). Aqueous samples that flow freely are collected in refrigerated bottles using automatic sampling equipment. Solid samples are collected as grab samples using wide-mouth jars.

8.2 Maintain aqueous samples in the dark at 0-4°C from the time of collection until receipt at the laboratory. If residual chlorine is present in aqueous samples, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine (Reference 17). If sample pH is greater than 9, adjust to pH 7-9 with sulfuric acid.

Maintain solid, semi-solid, oily, and mixed-phase samples in the dark at <4°C from the time of collection until receipt at the laboratory.

Store aqueous samples in the dark at 0-4°C. Store solid, semi-solid, oily, mixed-phase, and tissue samples in the dark at <-10°C.

8.3 Fish and Tissue Samples

8.3.1 Fish may be cleaned, filleted, or processed in other ways in the field, such that the laboratory may expect to receive whole fish, fish fillets, or other tissues for analysis.

8.3.2 Fish collected in the field should be wrapped in aluminum foil, and must be maintained at a temperature less than 4°C from the time of collection until receipt at the laboratory.

8.3.3 Samples must be frozen upon receipt at the laboratory and maintained in the dark at <-10°C until prepared. Maintain unused sample in the dark at <-10°C.

8.4 Holding Times

8.4.1 There are no demonstrated maximum holding times associated with CDDs/CDFs in aqueous, solid, semi-solid, tissues, or other sample matrices. If stored in the dark at 0-4°C and preserved as given above (if required), aqueous samples may be stored for up to one year. Similarly, if stored in the dark at <-10°C, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year.

Appendix A

8.4.2 Store sample extracts in the dark at <-10°C until analyzed. If stored in the dark at <-10°C, sample extracts may be stored for up to one year.

9.0 Quality Assurance/Quality Control

9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 18). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

If the method is to be applied to sample matrix other than water (e.g., soils, filter cake, compost, tissue) the most appropriate alternate matrix (Sections 7.6.2 through 7.6.5) is substituted for the reagent water matrix (Section 7.6.1) in all performance tests.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve separations or lower the costs of measurements. These options include alternate extraction, concentration, cleanup procedures, and changes in columns and detectors. Alternate determinative techniques, such as the substitution of spectroscopic or immuno-assay techniques, and changes that degrade method performance, are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for the analytes of interest.

9.1.2.1 Each time a modification is made to this method, the analyst is required to repeat the procedure in Section 9.2. If the detection limit of the method will be affected by the change, the laboratory is required to demonstrate that the MDL (40 CFR Part 136, Appendix B) is lower than one-third the regulatory compliance level or one-third the ML in this method, whichever is higher. If calibration will be affected by the change, the analyst must recalibrate the instrument per Section 10.

9.1.2.2 The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modifications.

9.1.2.2.2 A listing of pollutant(s) measured, by name and CAS Registry number.

Appendix A

13.7.1.1 Prepare the column as given in Section 7.5.4.

13.7.1.2 Pre-elute the column with 100 mL of hexane. Drain the hexane layer to the top of the column, but do not expose the sodium sulfate.

13.7.1.3 Load the sample and rinses (Section 12.4.1.9.2) onto the column by draining each portion to the top of the bed. Elute the CDDs/CDFs from the column into the apparatus used for concentration (Section 12.4.1.7) using 200 mL of hexane.

13.7.1.4 Concentrate the cleaned up extract (Sections 12.6 through 12.7) to constant weight per Section 12.7.3.1. If more than 500 mg of material remains, repeat the cleanup using a fresh anthropogenic isolation column.

13.7.1.5 Redissolve the extract in a solvent suitable for the additional cleanups to be used (Sections 13.2 through 13.6 and 13.8).

13.7.1.6 Spike 1.0 mL of the cleanup standard (Section 7.11) into the residue/solvent.

13.7.1.7 Clean up the extract using the procedures in Sections 13.2 through 13.6 and 13.8. Alumina (Section 13.4) or Florisil (Section 13.8) and carbon (Section 13.5) are recommended as minimum additional cleanup steps.

13.7.1.8 Following cleanup, concentrate the extract to 10 µL as described in Section 12.7 and proceed with the analysis in Section 14.

13.7.2 Acidified silica gel (Reference 28)—Procedure alternate to the anthropogenic isolation column (Section 13.7.1) that is used for removal of lipids from the Soxhlet/SDS extraction (Section 12.4.1).

13.7.2.1 Adjust the volume of hexane in the bottle (Section 12.4.1.9.2) to approximately 200 mL.

13.7.2.2 Spike 1.0 mL of the cleanup standard (Section 7.11) into the residue/solvent.

13.7.2.3 Drop the stirring bar into the bottle, place the bottle on the stirring plate, and begin stirring.

13.7.2.4 Add 30-100 g of acid silica gel (Section 7.5.1.2) to the bottle while stirring, keeping the silica gel in motion. Stir for two to three hours.

NOTE: 30 grams of silica gel should be adequate for most samples and will minimize contamination from this source.

STANDARD OPERATING PROCEDURE (SOP) FOR INTERNAL QA INSPECTION AND CORRECTIVE ACTION PROCEDURES FOR POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) AND RELATED COMPOUND ANALYTICAL PROGRAMS

Originated by: Charles V. Jaworski Date: 2/15/00

Approved by: Mary Schrock Date: 2/15/00
Technical Reviewer

Approved by: Karen Riggs Date: 2/21/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

Charles V. Jaworski Date: 2/21/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for internal Quality Assurance (QA) inspections and corrective action procedures for PCDD/PCDF and related compound analysis programs.

III. References

None

IV. Definitions

Critical Phases – Essential study events or procedures, which are observed by the Quality Assurance Officer or his/her designee to assess the integrity of a project.

Project Manager – The individual responsible for the overall conduct of a project.

Management –The individual defined as at least one level above the project manager for the purposes of routing Quality Assurance issues.

QA Officer –The individual assigned by management responsible for all aspect of quality assurance for the Atmospheric Science and Applied Technology department. This individual will be independent from the Project Manager and report directly to Management.

V. Procedures

A. Audits

Internal systems audits involve the evaluation of facilities, equipment, processes, and data packages for conformance to Battelle standards and project specific objectives. The Quality Assurance (QA) Officer or his/her designee conducts these audits. Any such audits or reviews are documented. To ensure that all issues have been addressed, the auditor also reviews all corrective actions taken.

1. Critical Phase Audits

The QA Officer will conduct critical phase inspections, when required by the project manager, to assess that facilities, equipment, personnel, methods, practices, records, and quality control, as they apply to regulated projects, are in conformance to approved protocols, SOPs, regulations, sponsor requirements, and Battelle policy.

Prior to conducting critical phase inspections in the laboratory or field, the QA Officer will review the method, current versions of applicable SOPs, personnel training records and any special sponsor requirements. All critical phase observations will be conducted in the immediate area where the work is taking place, unless requested by the project manager, or for safety purposes.

The QA officer will monitor the on-going activity to assure SOPs and/or approved methods are available to the personnel conducting the work and that the work is being done as described in those SOPs or methods.

The QA Officer will also check to see that appropriate calibration, standardization, and inspection of equipment (i.e., refrigerators, freezers, balances, etc.) has occurred prior to use and that all required equipment records are in place and current.

Data records will be examined to make sure that they are current, are being promptly recorded in ink for manual recordings, and that any error corrections are being made by following Good Laboratory Practice (GLP) techniques. The QA Officer will check to see that the procedure being observed is fully and accurately being documented.

All reagents, solutions, control and reference materials will be checked by the QA Officer to insure that they are appropriately labeled.

All findings and observations will be recorded on the form similar to Attachment A and will be kept on file in the Quality Assurance Unit following routing (see section A5).

2. Audits of Data Quality

All data reports, including the Laboratory Record Books (LRBs), will be reviewed by the project manager and/or his/her designee to ensure that the procedures have been fully documented. If required by the project, following the review of the project manager and/or his/her designee, all materials will be sent to the QA Officer for further review. The project manager and/or his/her designee and/or the QA Officer will review all raw data (or a percentage as required by the project/client) for completeness and accuracy. In addition to ensuring that the data adheres to all issues of GLP, if required, the following specific items will be emphasized. This list is not all-inclusive and additional items may be examined:

- verify the concentration and traceability of standard solutions
- ensure traceability of all sample extracts
- Initial calibration and continuing calibrations meet method and/or project requirements
- mass lock checks meet requirements
- transcription verification of extract number and sample weight from the prep LRB into OPUS forms and Excel spreadsheets for each sample
- all compounds of interest are accounted for
- double check for co-eluting ethers for the furans reported and that the compounds have been flagged appropriately
- percent recoveries for internal standards, Standard Reference Material (SRM), and any spike samples meet method and/or project requirements

- percent recoveries are flagged appropriately.

3. Facility Inspections

All laboratory facilities may be inspected annually by the QA Officer to ensure that the facilities adhere to all requirements set forth in the regulations. Equipment records for instruments used to generate data or that affect the integrity of data collected will be examined to assure that written records are being maintained according to SOP. All reagents and samples present in the laboratory will be reviewed to assure proper labeling and that the stated storage requirements are being met. The SOP manuals will be compared with the SOP manual Table of Contents to assess whether they are current.

In addition, a member of the Environmental Safety and Health (ES&H) Department conducts quarterly facility inspections. The laboratories will be inspected for general cleanliness and adherence to safety procedures and requirements. The results of these inspections are communicated to the laboratory personnel verbally and when deemed appropriate, the results may be provided in writing based on the severity of the inspection results.

B. Reports and Corrective Action(s)

Findings of Critical Phase audits, Technical Data audits, and Facility Inspections, by the QA Officer, will be documented on an Audit Comment Sheet similar to Attachment B. This form, together with a Quality Assurance Routing Sheet (similar to Attachment C) will then be sent to the project manager (or facility manager in the case of facility inspections). The project manager will review each finding (if any) and describe on the Audit Comment Sheet the corrective actions taken and sign the routing sheet to attest to the fact that these actions were implemented. The form will then be returned to QA officer who will verify that the corrective actions were adequate and have been implemented (confirmed by signature on the routing sheet). The findings will then be routed to management for review and signature and returned to the QA Officer for retention.

VI. Revision History

Not applicable

Attachment A

Critical Phase Inspection Form

Project Manager:
QA Officer:_____

Study Number:
Date:_____

Compliance Item Cited: See Attached Comments

No Adverse Findings

Phase	Protocol, SOP, Method, Equipment Used

Personnel:

Narrative:

Attachment B

AUDIT COMMENT SHEET

Study Number and Phase:
QA Auditor:

INSPECTION PARAMETER	COMMENT	CORRECTIVE ACTION/RESPONSE

Attachment C

Quality Assurance Routing Sheet

Project Number:

Project Manager:

Activity:

Audit Type:

Project Title:

Sponsor:

Auditor:

Date:

Please complete the attached form indicating CORRECTIVE ACTION TAKEN (IF NEEDED), sign and date this Routing Sheet in the space provided beside your name, and return the entire set when completed to the Quality Assurance Unit no later than _____.

Route To	Signature	Date

STANDARD OPERATING PROCEDURE (SOP) FOR POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) SAMPLE CONTAINER PREPARATION AND SHIPMENT

Originated by: _____ Date: 16 Feb 2000

Approved by: _____ Date: 2/15/00
Technical Reviewer

Approved by: _____ Date: 2/18/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

_____ Date: 2/18/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for preparation and shipment of containers for PCDD/PCDF samples and contains the following:

- Sample container quality
- Shipping instructions for the sample container(s).

III. References

None.

IV. Definitions

Not applicable.

V. Procedures

A. Personnel Qualifications

Personnel assigned to laboratory activities meet the educational, work experience, and training requirements for their positions. Records on personnel qualifications and training are maintained in personnel files accessible for review during audit activities. Training is conducted in accordance with standard operating procedures and is available to all laboratory personnel. Employees must demonstrate proficiency at specific tasks and this capability is documented and kept in a central file.

B. Sample Container

All sample containers are amber glass with Teflon caps and are purchased certified, pre-cleaned (VWR, grade QA or equivalent) and are received along with a Certificate of Analysis. The supplier assigns a lot number to each container that allows the container to be traced to the appropriate Certificate of Analysis.

C. Sample Container Shipping Procedures

Each sample container is individually wrapped in bubblewrap. The wrapped containers are tightly packed into a cooler that is lined with bubble wrap and packing peanuts. Cool packs, at room temperature, are also added to the cooler, if required. A chain of custody form is then placed in a plastic sealed bag and taped to the inside top of the cooler. The cooler is tape/sealed shut and taken to the Battelle shipping department, along with a shipping memo that contains the address and client contact that the cooler is being sent to. The shipping department then sends the cooler and its contents to the client.

VI. Revision History

Not applicable.

STANDARD OPERATING PROCEDURE (SOP) FOR POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) DESICCATING AGENT AND ADSORBENT PREPARATION AND STORAGE

Originated by: _____ Date: 16 Feb 2000

Approved by: _____ Date: 2/15/00
Technical Reviewer

Approved by: _____ Date: 2/18/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

_____ Date: 2/18/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for the preparation and storage of adsorbents and desiccating agents used in sample clean-up activities for PCDD/PCDF samples. These procedures follow general guidelines described in SW846 Method 8290 and EPA Method 1613, with some minor modifications/improvements. This SOP covers the following areas:

- Silica preparation and storage
- Acid silica preparation and storage
- Basic silica preparation and storage
- Alumina preparation and storage
- CarboPack C carbon preparation and storage
- Celite preparation and storage
- CarboPack C carbon/Celite mixture preparation and storage
- Hydromatrix (also known as Hidromatrix) preparation and storage
- Sodium Sulfate preparation and storage

III. References

A. SW-846, Method 8290. Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRCG/HRMS), Revision 0, 1994.

B. EPA Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994, EPA 821-B-94-005.

IV. Definitions

All sections referenced in this SOP are from SW846 Method 8290 unless otherwise indicated.

V. Procedures

A. Personnel Qualifications

Personnel assigned to laboratory activities meet the educational, work experience, and training requirements for their positions. Records on personnel qualifications and training are maintained in personnel files accessible for review during audit activities. Training is conducted in accordance with standard operating procedures and is available to all laboratory personnel. Employees must demonstrate proficiency at specific tasks and this capability is documented and kept in a central file.

B. Silica gel.

Silica gel (ICN Biomedicals, 100-200 mesh, grade 60 A, or equivalent) is activated and cleaned in a manner similar to that called for in Method 8290, Section 5. 2.3 using the following procedure. A muffled Pyrex tube is filled ¾ full with silica and rinsed with 350 mL methanol (EM Science, HPLC grade, or equivalent) and 350 mL dichloromethane (DCM)(B&J Brand, pesticide residue grade, or equivalent), consecutively, using nitrogen to push the solvent through the silica. While still saturated

with DCM, the silica is emptied into a muffled glass thimble, topped with DCM-rinsed muffled glasswool, and extracted with DCM in a Soxhlet apparatus for a minimum of 3 hours. After extraction, the silica is poured into a muffled Pyrex tube, placed under nitrogen, heated to 65 degrees Celsius for 30 minutes, and then activated at 160 degrees Celsius for 1 hour. After activation the silica is allowed to come to room temperature under nitrogen and then is stored in a dessicator in a muffled glass jar with a Teflon-lined screw cap (expiration is 1 year from date of preparation).

C. Acid silica gel

Acid silica gel is prepared in a manner similar to that called for in Method 8290, Section 5.2.5 using the following procedure: 35 grams of activated silica gel (prepared as in V.B above) is weighed into a muffled glass jar; 15 mL of concentrated sulfuric acid is then added to the same jar. The jar is then capped with a Teflon-lined screw cap and shaken until the mixture is free of lumps and free flowing. The mixture is stored in a muffled glass jar with a Teflon-lined screw cap, inside a dessicator (Expiration date is 1 year from preparation).

D. Basic silica gel

Basic silica gel is prepared in a manner similar to that called for in Method 8290, Section 5.2.4 using the following procedure: 35 grams of activated silica gel (prepared as in V.B above) is weighed into a muffled glass jar; 17 mL of 1 N NaOH solution is then added to the same jar. The jar is then capped with a Teflon-lined screw cap and shaken until the mixture is free of lumps and free flowing. The mixture is stored in a muffled glass jar with a Teflon-lined screw cap, inside a dessicator (Expiration date is 1 year from preparation).

E. Alumina

Alumina (Sigma, activity grade 1, type WB-2 basic, or equivalent) is activated in a manner using similar methods called for in Method 1613 B, Section 7.5.2.2. using the following procedure. A muffled Pyrex tube is filled ¾ full with alumina and kept under nitrogen; the tube is then placed in a tube furnace and slowly brought up to 500 °C. The alumina is kept at 500 °C for a minimum of 12 hours while being under nitrogen. After this activation period, the alumina is allowed to come to room temperature under nitrogen. Once cool the alumina is stored in a muffled Erlenmeyer flask, capped with a muffled glass stopper, and placed in an oven at 130 °C. Right before and during use the alumina is stored in a dessicator at room temperature. The alumina is re-activated if three days have passed since it's last activation.

F. CarboPack C Carbon

Carbopack C (Supelco, 60/80 mesh, or equivalent) following Method 1613 B, Section 7.5.3.1. requires no special cleanup prior to use. Stored in dessicator (expiration is 5 years from date of receipt).

G. Celite

Celite (Supelco, 545-AW, reagent grade, or equivalent) following Method 1613 B, Section 7.5.3.2. requires no special cleanup prior to use. Stored in dessicator (expiration is 5 years from date of receipt).

H. CarboPack C carbon/ Celite Mixture

The CarboPack C carbon (Supelco, 60/80 mesh) / Celite (Supelco, 545-AW, reagent grade) mixture is activated in a manner similar to that called for in Method 1613 B, Section 7.5.3.3. with a modification producing a 20 % CarboPack C to Celite mixture (w/w) using the following procedure: the required amounts of CarboPack C carbon and Celite are weighed into the same muffled glass jar to produce enough material to pack the desired number of columns with a small amount of material being left over. The jar is then capped with a Teflon-lined screw cap and rotated until a uniform mixture is obtained. The Teflon-lined screw cap is removed and the mixture is covered with muffled aluminum foil that is punctured with small holes. The mixture is then placed and stored in a muffle oven at 130 °C for a minimum of 6 hours. Prior to use, the carbon/Celite mixture is allowed to come to room temperature inside a dessicator and is capped with a Teflon-lined screw cap (expiration is 1 year from date of preparation).

I. Hydromatrix

Hydromatrix (Varian, Sample Preparation Products, part number 0019-8003, or equivalent) requires no special cleanup prior to use. Once opened the Hydromatrix is stored in a muffled glass jar with a Teflon-lined screw cap, inside a dessicator (expiration is 1 year from date of receipt).

J. Sodium Sulfate

Sodium sulfate (JT Baker, 12-60 mesh, Ultra Resi-Analyzed, or equivalent) is cleaned and dried in a manner similar to Section 5.4.1. by the following procedure: 1 kg is poured into a large muffled glass thimble and rinsed with 500 mL of DCM (B&J Brand, pesticide residue grade, or equivalent). The saturated sodium sulfate is allowed to dry inside the thimble for 1 hour. The partially dried sodium sulfate is poured into a shallow stainless steel tray and covered with muffled aluminum foil or a stainless steel lid and muffled for a minimum of 6 hours at 450 °C. The muffled sodium sulfate is stored in the muffling container at 130 °C. Prior to use, the muffled sodium sulfate is poured into a muffled glass jar with a Teflon-lined screw cap and allowed to come to room temperature inside a dessicator.

VI. Revision History

Not applicable.

STANDARD OPERATING PROCEDURE (SOP) FOR POLYCHLORINATED DIBENZO-P-DIOXIN/POLYCHLORINATED DIBENZOFURAN (PCDD/PCDF) STANDARDS AND REAGENTS PREPARATION AND STORAGE FOR MODIFIED METHOD 8290 ANALYSIS

Originated by: _____ Date: 16 Feb 2000

Approved by: _____ Date: 2/16/00
Technical Reviewer

Approved by: _____ Date: 2/18/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

_____ Date: 2/18/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This SOP describes routine procedures for preparation and storage of standards and reagents for PCDD/PCDF samples prepared using modified Method 8290 (ASAT.II-002). These procedures follow general guidelines described in EPA Method 1613, with some minor modifications/improvements. The descriptions in this SOP cover the following items:

- Internal Standard (IS) Spiking Solution
- Cleanup Standard (CS) Stock Solution
- Cleanup Standard (CS) Spiking Solution
- Precision and Recovery (PAR) Spiking Solution
- Recovery Standard (RS) Stock/Spiking Solution
- Other standards
- Potassium Hydroxide Solution
- Sodium Hydroxide Solution
- Sodium Chloride Solution
- Dichloromethane/Methanol/Toluene – 15:4:1 Solution
- Dichloromethane/Cyclohexane – 1:1 Solution
- Dichloromethane/Hexane – 1:1 Solution

III. References

A. EPA Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994, EPA 821-B-94-005.

IV. Definitions

All references in this section are to EPA Method 1613 unless otherwise indicated.

V. Procedures

A. Personnel Qualifications

Personnel assigned to laboratory activities meet the educational, work experience, and training requirements for their positions. Records on personnel qualifications and training are maintained in personnel files accessible for review during audit activities. Training is conducted in accordance with standard operating procedures and is available to all laboratory personnel. Employees must demonstrate proficiency at specific tasks and this capability is documented and kept in a central file.

B. Standard Solutions

1. Internal Standard (IS) Spiking Solution

The IS spiking solution is made similar to Method 1613 B, Section 7.10.3 by the following procedure. IS stock solution (Cambridge Isotope Laboratories EDF-8999, or equivalent) is diluted 1:50 with acetone (B & J Brand pesticide residue grade, or eqivalent). Every sample, including each quality control (QC) sample, gets a 1 mL spike of IS spiking solution prior to extraction. This is equivalent of 20 µl of stock IS in each 1 mL of IS spiking solution. In order to minimize waste of these standards, the minimum volume possible of the IS spiking solution is prepared for each sample batch. The total

volume of the IS spiking solution prepared for a sample batch needs to be sufficient to deliver a 1 mL aliquot to each sample (including QC samples) with 2 mL being left over. For example, if the total number of samples (including QC samples) is 28, then 30 mL of IS spiking solution would be prepared. This often results in odd volumes of IS spiking solution being needed. Because of this, an accurate final volume for the IS spiking solution is determined using the weight and density of the components rather than by a direct volume measurement.

The IS stock solution volume is determined by taking the total mL of IS spiking solution to be prepared and multiplying by 20µL IS stock/mL. For this example, if 30 mL of IS spiking solution is being prepared, then 600 µL IS stock is needed. The IS stock volume is nominally measured using a calibrated Eppendorf pipettor and the stock is added to a tared, muffled, amber vial with a Teflon-lined screw cap. The weight of IS stock solution is noted in the lab record book. This weight should be within ±0.0003 of the theoretical weight for the volume of IS stock solution multiplied by the density of the solution, otherwise the total solution volume should be adjusted accordingly. For this example, the weight of 600µL of the IS stock solution, with a density of 0.718 g/mL, should read 0.4308 ± 0.0003 g. The theoretical IS spiking solution total weight is determined based on the volume of IS stock delivered and the amount of acetone needed to reach the correct total volume, and their respective densities. The solution is then brought up to the final volume with acetone by accurately reaching the theoretical weight of the IS spiking solution. This is accomplished by using a muffled pasture pipette to slowly add acetone to the IS stock aliquot until the theoretical weight of the solution is obtained within ±0.0003g. In the example for preparing 30 mL of the IS spiking solution, once 0.4308g of the stock IS was added, acetone would then be added until the total solution volume reached 23.6862 ± 0.0003g:

0.600 mL Stock IS (nonane ρ=0.718 g/mL)	Or	(0.4308g)
29.400 mL acetone (acetone ρ=0.791 g/mL)	Or	(23.2554g)
30.000 mL Total	Or	(23.6862g)

Once the final solution volume has been reached, the vial is capped. The capped vial is sealed with Teflon tape and the volume is marked using a permanent marker. The vial is then stored in a freezer. Prior to use, the IS spiking solution is kept capped and sealed and allowed to come to room temperature inside a fume hood. The IS spiking solution is good for a maximum period of five days from the date of preparation.

2. Cleanup Standard (CS) Stock Solution

The CS stock standard is prepared by a 1:50 dilution of CIL stock solution ED-907 (or equivalent) in nonane (Aldrich high purity, or equivalent). Usually only a few milliliters of this dilute solution are needed and, therefore, all volumes can be accurately measured using calibrated pipettors; no weighing is needed. The CS stock solution is prepared in a similar manner as Method 1613, Section 7.11. For example:

80 µL 2,3,7,8-TCDD$^{37}Cl_4$ stock (CIL ED-907)
1000µL + 1000µL + 1000µL + 920µL nonane
4.000 mL Total

The stock solution is measured using a calibrated Eppendorf pipettor and added to an amber vial with a Teflon-lined screw cap. This is brought up to volume with Aldrich high purity nonane (or equivalent) which is also measured using a calibrated Eppendorf pipettor. The vial is capped, then sealed with Teflon tape. The volume is marked using a permanent marker and stored in a freezer. Prior to use, the CS stock solution is kept capped and sealed and allowed to come to room temperature inside a fume hood. The solution is considered valid for a period of five years, at which time an assessment of the solution will be performed. If the assessment of the solution attests to the integrity of the quality and concentration of the stock, the expiration date of the stock may be extended for an additional five years.

3. Cleanup Standard (CS) Spiking Solution
Each sample extract, including QC sample extracts, gets 1 mL of CS spiking solution prior to any cleanup activities. The CS spiking solution is prepared in a similar fashion to the IS spiking solution (V.B.1) except that more of a dilution is necessary. The CS stock solution (from Section V.B.2) is diluted 1:5000 with acetone (B & J Brand pesticide residue grade, or equivalent). A minimum of 30 mL of the CS spiking solution is prepared at all times. In a fashion similar to the IS spiking solution, the CS spiking solution volumes are accurately determined using weights for the CS stock and acetone, and their respective densities rather than by a direct volume measurement. The stock CS volume is nominally determined using a calibrated Eppendorf pipetor and is then added to a tared amber vial with a Teflon-lined screw cap. The weight of CS stock solution is noted in the lab record book. This weight should be within ±0.0003 of the theoretical weight for the volume of CS stock solution multiplied by the density of the solution, otherwise the total solution volume should be adjusted accordingly. The theoretical CS spiking solution total weight is determined based on the volume of CS stock delivered and the amount of acetone needed to reach the correct total volume, and their respective densities. The solution is then accurately brought up to the final volume with acetone by reaching the theoretical weight of the CS spiking solution. This is accomplished by using a muffled pasture pipette to slowly add acetone to the CS stock aliquot until the theoretical weight of the spiking solution is obtained within ±0.0003g. For preparing 30 mL of the CS spiking solution, once 0.0043g of the stock CS is added, acetone would then be added until the total solution volume reached 23.7296 ± 0.0003g.

6 µL Stock CS (nonane ρ=0.718)	Or	(0.0043g)
29.994 µL acetone (acetone ρ=0.791)	Or	(23.7253g)
30.000 mL Total	Or	(23.7296g)

The CS spiking solution is then capped. The capped vial is sealed with Teflon tape and the volume is marked using a permanent marker. The vial is then stored in a freezer. Prior to use, the CS spiking solution is kept capped and sealed and allowed to come to room temperature inside a fume hood. The CS spiking solution is good for a maximum period of five days from the date of preparation. This standard may also be purchased directly from a vendor as long as it is a certified solution.

4. Precision and Recovery (PAR) Spiking Solution
For those samples that require a spike of PAR (matrix spike (MS), matrix spike duplicate (MSD), and method detection limit samples (MDLs), etc.) a 1:200 dilution of CIL stock solution EDF-7999 (or equivalent) is used. Usually only a few milliliters of this dilute solution are needed and, therefore, all volumes can be accurately measured using calibrated pipettors; no weighing is needed.. Each MS/MSD sample gets 1 mL of the PAR spiking solution (the equivalent of 5 µL of CIL EDF-7999), and each MDL sample gets 250 µL of the PAR spiking solution (the equivalent of 1.25 µL of CIL EDF-7999). The PAR spiking solution is prepared in a similar manner as Method 1613, Section 7.14. For example:

15 µL PAR stock (CIL EDF-7999)
1000µL + 1000µL + 985µL acetone
3.000 mL Total

The stock solution is measured using a calibrated Eppendorf pipettor and added to an amber vial with a Teflon-lined screw cap. This is brought up to volume with acetone (B & J Brand pesticide residue grade, or equivalent) which is also measured using a calibrated Eppendorf pipettor. The vial is capped, then sealed with Teflon tape. The volume is marked using a permanent marker and stored in a freezer. Prior to use, the PAR spiking solution is kept capped and sealed and allowed to come to room temperature inside a fume hood. The solution is good for a maximum period of five days from the date of preparation.

5. Recovery Standard (RS) Stock/Spiking Solution
Each sample extract, including QC sample extracts, gets 10 µL of RS standard added prior to analysis. The RS standard is prepared by a 1:250 dilution of CIL stock solutions ED-911(or equivalent) and ED-996 (or equivalent) in nonane (Aldrich high purity, or equivalent). Usually only a few milliliters of this dilute solution is needed and is, therefore, made using pipettors; no weighing is needed. Each sample gets 10 µL of the diluted RS (the equivalent of 0.04 µL of CIL ED-911 and ED-996). The RS spiking

solution is prepared in a similar manner as Method 1613, Section 7.14. For example:

20 µL 1,2,3,4-TCDD$^{13}C_{12}$ stock (CIL ED-911)
20 µL 1,2,3,7,8,9-HxCDD$^{13}C_{12}$ stock (CIL ED-996)
1000µL + 1000µL + 1000µL + 1000µL + 960µL nonane
5.000 mL Total

The stock solution is measured using a calibrated Eppendorf pipettor and added to an amber vial with a Teflon-lined screw cap. This is brought up to volume with Aldrich high purity nonane, which is also measured using a calibrated Eppendorf pipettor. The vial is capped, then sealed with Teflon tape. The volume is marked using a permanent marker and stored in a freezer. Prior to use, the RS spiking solution is kept capped and sealed and allowed to come to room temperature inside a fume hood. The solution is considered valid for a period of five years, at which time an assessment of the solution will be performed. If the assessment of the solutions attests to the integrity of the quality and concentration of the stock, the expiration date of the stock may be extended for an additional five years.

6. Other Standards
Instrument calibration standards, window set mix, and the TCDD and TCDF column performance standards are purchased as certified mixes from CIL.

C. Reagent Solutions

1. Potassium Hydroxide Solution
A 20 % weight per volume (w/v) potassium hydroxide (KOH) solution (Section 7.1.1) is prepared in organic – free reagent water (JT Baker, Ultra Resi-Analyzed, or equivalent). The solution is prepared by weighing 20 grams of KOH (EM Science,GR, pellets, or equivalent) into a muffled glass beaker. The KOH is then transferred to a Teflon bottle and 100 mL of water is slowly added to the bottle. The KOH is allowed to completely dissolve into the water before being used. The 20 % KOH solution is stored in the Teflon bottle with a Teflon cap at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

2. Sodium Hydroxide Solution
A 1 N sodium hydroxide (NaOH) solution sited in Section 7.5.1.3. is prepared in organic-free reagent water (JT Baker, Ultra Resi-Analyzed, or equivalent). The 1 N NaOH solution is prepared by weighing 40 grams NaOH (JT Baker, Baker-analyzed, pellets, or equivalent) into a muffled glass beaker. The NaOH is transferred to a 1-L Teflon bottle and 1 L water is slowly added. The NaOH is allowed to completely dissolve into the water before being used. The 1 N NaOH solution is stored in the Teflon bottle with a Teflon cap at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

3. Sodium Chloride Solution
   A 5 % weight per volume (w/v) sodium chloride (NaCl) solution (Section 7.1.4) is prepared in organic-free reagent water (JT Baker, Ultra Resi-Analyzed, or equivalent). The solution is prepared by weighing 50 grams NaCl (EM Science, GR, crystals, or equivalent) into a muffled glass beaker. The NaCl is transferred to a 4-L reagent bottle and 1 L of water is slowly added. The NaCl is allowed to completely dissolve before being used. The 5 % NaCl solution is stored in the reagent bottle with a Teflon-lined cap at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

4. Dichloromethane/Methanol/Toluene – 15:4:1 Solution
   The 15:4:1 volume per volume (v/v) solution in Section 13.5.2. containing dichloromethane (B&J Brand, pesticide residue grade, or equivalent), methanol (EM Science, HPLC grade, or equivalent), and toluene (JT Baker, Ultra Resi-Analyzed, or equivalent) is made by measuring 150 mL of dichloromethane in a muffled glass graduated cylinder and then pouring the dichloromethane into a muffled glass 250 mL reagent bottle. Methanol (40 mL) is measured in a muffled graduated cylinder and poured into the same reagent bottle containing the dichloromethane. Toluene (10 mL) is measured in a muffled graduated cylinder and poured into the same glass reagent bottle containing the dichloromethane:methanol solution. The solution is capped with a Teflon-lined screw cap and stored at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

5. Dichloromethane/Cyclohexane – 1:1 Solution
   The 1:1 volume per volume (v/v) solution cited in Section 13.5.2. containing dichloromethane (B&J Brand, pesticide residue grade, or equivalent) and cyclohexane (B&J Brand, pesticide residue grade, or equivalent) is made by measuring 100 mL of dichloromethane in a muffled graduated cylinder and then pouring it into a muffled 250-mL glass bottle. Cyclohexane (100 mL)is measured in a muffled graduated cylinder and poured into the glass bottle containing the dichloromethane. The solution is capped with a Teflon-lined screw cap and stored at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

6. Dichloromethane/Hexane – 1:1 Solution
   The 1:1 volume per volume (v/v) solution cited in Section 12.4.1.2 & 13.4.7.2. containing dichloromethane (B&J Brand, pesticide residue grade, or equivalent) and hexane (B&J Brand, pesticide residue grade, or equivalent) is made by measuring 1000 mL of dichloromethane in a muffled graduated cylinder and then pouring it into a muffled 4-L glass bottle. Hexane (1000 mL)is measured in a muffled graduated cylinder and poured into the glass bottle containing the diclorometh ane. The solution is capped with a Teflon-lined screw cap and stored at room temperature. The solution bottle is labeled with storage conditions and an expiration date of 1 year from date of preparation.

VI. Revision History

Not applicable.

OCT 30 2000

STANDARD OPERATING PROCEDURE (SOP)
FOR
THE SAMPLE CHAIN-OF-CUSTODY FOR DIOXIN/FURAN ANALYSIS

Originated by: _____ Date: 10/30/00

Approved by: _____ Date: 10/30/00
Technical Reviewer

Approved by: _____ Date: 10/30/00
Management

Reviewed and Registered by QAU:

_____ Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I. Scope

This standard operating procedure defines chain-of-custody procedures for samples submitted to Battelle for polyhalogenated dibenzo-p-dioxin and dibenzofuran (dioxin/furan) analysis.

II. Purpose

According to Good Laboratory Practices (GLP) Regulations of the Food and Drug Administration (FDA), the Environmental Protection Agency (EPA), and others, handling of samples must be conducted in a manner that ensures that:

- There is proper storage.
- Distribution is made in a manner designed to preclude the possibility of contamination, deterioration, or damage.
- Proper identification is maintained throughout the distribution process.
- The receipt and distribution of each batch is documented.

The above requirements are provided by the chain-of-custody procedures described in this standard operating procedure.

III. References

None.

IV. Definitions

Dioxin Laboratory is a limited access laboratory at Battelle designed exclusively for preparation of samples for dioxin/furan analysis. The Dioxin Laboratory is located in Room 5218.

Sample Preparation LRB is a bound laboratory record book used for documentation of sample preparation procedures.

Sample Name is the sample identification provided by the client for each sample (i.e., sludge AB). The sample name is generally listed on the label affixed to the sample container.

Sample Code is the unique sample identification number provided by Battelle for each sample received for dioxin/furan analysis.

V. Procedures

A. Sample Receipt

1. Samples for dioxin/furan analysis arrive at Battelle through Battelle's Shipping/Receiving Department. Upon notification of sample arrival, a staff member from the Dioxin

Laboratory will retrieve samples from the location of arrival. Alternatively, samples may be submitted directly to the Dioxin Laboratory by a Battelle Staff member.

2. Upon receipt of a sample(s) at the Dioxin Laboratory, the shipping container will be opened and the contents will be inspected to determine if receipt is acceptable. This inspection will include the following and will be recorded in the sample log-in laboratory record book (LRB):
   a.) The method of delivery will be documented and the package will be examined for the presence/absence of custody papers.
   b.) The shipping container will be inspected for the presence/absence and condition of custody seals.
   c.) The temperature of each shipping container will be measured and recorded to document if the samples were maintained at the appropriate temperature during shipment. The temperature of a cooler blank (if available), melt water, or the external temperature of the sample container should be measured and documented (thermometers or probes should never be inserted into a sample container).
   d.) Inspect each sample for the presence/absence and condition of custody seals. Check each sample for breaks or leaks.
   e.) Labels on sample containers will be compared with the accompanying sample documents or chain-of-custody records to ensure that all listed samples have been received. If any samples are missing, mislabeled, broken, or damaged, the Battelle project leader will be informed, who will then inform the client. The external chain-of-custody record will be signed and dated by the staff member receiving the samples. Any discrepancies or problems with the samples will be noted on the chain-of-custody record.
   f.) Aqueous samples that might contain total residual chlorine (TRC) should ideally be treated with sodium thiosulfate ($Na_2S_2O_3$) in the field. If that hasn't occurred, upon receipt in the laboratory the sample should be measured for TRC using a commercial test kit. If TRC is detected then $Na_2S_2O_3$ should be added at a ratio of 80 mg per liter of sample and documented on the COC and in the sample log-in LRB.
   g.) The pH of aqueous samples will be measured and recorded. If the pH is >9, the pH will be adjusted to a pH of 7-9 with sulfuric acid unless the project requirements state specifically otherwise.

3. In addition to sample names, all samples should be labeled with an expiration date and storage conditions required by the method, client, or study protocol. If expiration dates and storage conditions are not listed on the label affixed to the container, the Battelle staff member receiving the samples will affix a second label to each sample container with the expiration date and storage conditions. If expiration dates and storage conditions are not specified by the method, client, or study protocol, the following should be used:

	Expiration Date	Storage
Fish/adipose tissue:	5 years	<-20°C
All other matrices:	5 years	<4°C

4. Receipt of samples will be recorded in a LRB designated for sample receipt. One page will be used for each sample shipment. The following information will be recorded:

- Method of delivery (i.e., FedEx)
- Presence/absence and condition of custody seals on the shipping container and sample jars.
- Measurement for TRC and amount of $Na_2S_2O_3$ added if necessary.
- The temperature of each shipping container.
- The pH of each aqueous sample and adjustments made to sample pH.
- Any discrepancies between sample labels and COC sheets.
- Sample name
- Conditions of samples upon receipt (i.e., intact)
- Each sample will be given a unique log-in code based on the LRB in which the sample receipt is documented (i.e., 43201-10-2). The first group of numbers in this sample code is the LRB number, the second two digit number is the LRB page on which the sample code is documented, and the third number is the line on the LRB page which specifically refers to that sample (only one line is assigned per sample). This number will also be written on each jar.

5. A copy of the completed chain-of-custody records submitted with the samples will be made and attached to the sample receipt LRB. A copy will also be made for the project file. The original chain-of-custody record will be returned to the client if requested, or archived in the project files.

6. Samples will be stored under required conditions within the Dioxin Laboratory until preparation for analysis.

B. Sample Preparation

1. At the initiation of sample preparation for dioxin/furan analysis, each sample (or sample aliquot, if the entire sample is not used for sample preparation) will be assigned a unique sample code. An example of an acceptable code is one based on the LRB in which sample preparation is documented (i.e., 45674-24-5). The first group of numbers in this sample code is the LRB number, the second two digit number is the LRB page on which the sample code is documented, and the third number is the line on the LRB page which specifically refers to that sample (only one line is assigned per sample). Other sample codes are acceptable if they can be made unique for each sample and can be easily documented.

2. Date and name of staff member will be recorded in the sample preparation LRB for all sample preparation activities.

3. The sample extracts resulting from sample preparation will be labeled with the unique sample code and stored at $< 4^{o}C$ in a designated refrigerator within the Dioxin Laboratory, unless other storage conditions are specified by the method, client, or study protocol. A

LRB, referred to as the Extract Chain-of-Custody LRB, is used to document storage of sample extracts ready for analysis. Documentation will include a listing of the sample name, the unique sample code, and the date the extracts were stored and ready for analysis. An example of this documentation is shown in Attachment A.

C. Sample Analysis

1. The analyst will retrieve sample extracts for dioxin/furan analysis from the Dioxin Laboratory. The analyst will record his/her name and the date of extract retrieval on the page in the LRB assigned to that particular sample set (Attachment A).

2. Sample extracts will be brought by the analyst to the analysis laboratory. Extracts will remain in custody of the analyst at all times. At the end of each day, or if the analyst will be absent from the laboratory at any time during the day, sample extracts will be stored in a limited access storage access and documented in study records.

3. Mass spectrometry analysis of sample extracts will be recorded in daily log runs kept with each MS instrument. The log run will list the sample name, the unique sample code, and the MS file name as shown in Attachment B. The sample name, unique sample code, and MS file name will be recorded on each hard copy chromatogram generated by the analysis.

4. A copy of the run log will be entered in the project files at the completion of analysis.

5. At the completion of analysis, sample extracts will be returned to the Dioxin Laboratory for storage until final disposition. This return will be documented in the Extract Chain-of-Custody LRB by the analyst.

D. Sample Division

Samples which need to be divided and submitted for analyses other than dioxin/furan will be handled in the following manner:

1. At the initiation of sample preparation for dioxin/furan analysis, the aliquot(s) required for additional analyses will be removed from the bulk sample.

2. Aliquot removal will be documented in the sample preparation LRB.

3. The aliquot will be labeled with the original sample name and submitted to the laboratory responsible for the additional analyses. Documentation of transfer of sample aliquots to a second laboratory will be recorded in the sample preparation LRB.

4. It will be the responsibility of the laboratory conducting additional analyses to assign a unique sample code to the sample aliquot received for additional analyses, to label the sample with expiration date and storage conditions, and to initiate chain-of-custody procedures for the sample aliquot.

5. Remaining bulk sample will be stored in the Dioxin Laboratory until final disposition.

VI. Revision History

1. Additional sample login details were added in going to ASAT.II-007-01 including measuring cooler temperature upon sample receipt and measuring and adjusting pH of aqueous samples.

Attachment A

BATTELLE LABORATORY RECORD BOOK NO. 47905 99

Work Performed by: Savage Project No. G6920007-01 Date of Work: May 10, 2000

Title or Purpose: Perth Amboy Continued from: —

Samples ready for GC/MS analysis
Stored in Rm 5218, Freezer F-3

Extraction #
48248-17-02
03
04
05
06
07
08
09
10
11
12

Samples to 5282
Freezer 05-10-00

5/10/00

Entered by: Andrew Downs Date: 5/10/06 Continued to: —

Performances of this work observed by:	Disclosed to and understood by me:
Date:	Karen Pennell Date: 5/10/00
Date:	Date:

Attachment B

BATTELLE LABORATORY RECORD BOOK NO. 48279 33

Work Performed by: TABar Project No. G003896-018C Date of Work: 02-23-00

Title or Purpose: USEPA Region II Blue Crab Tissue Continued from: p32

48279338	1	47725-45-12	cp3+ws+cp	(A) (Geo OK)	Lmb Dioxin
48279338	1	48370-11-26	Method Blank		
	2	48370-11-02	111378		
	3	48370-11-03	111379		
	4	48370-11-04	111380		
	5	48370-11-05	111381		
	6	48370-11-06	111401		
	7	48370-11-09	111403		
	8	48370-11-10	111403 Duplicate		
	9	48370-11-11	111408		
	10	48370-11-12	111431		
	11	47725-45-12	cp3+ws+cp (Geo OK)		
	12	48370-11-26	Method Blank		
	13	48370-11-13	111432		
	14	48370-11-14	111437		
	15	48370-11-15	111441		
	16	48370-11-16	111442		
	17	48370-11-17	111444		
	18	48370-11-18	111446		
	19	48370-11-19	111464		
	20	48370-11-20	207280		
	21	48370-11-21	207282		
	22	47725-45-12	cp3+ws+cp (Geo OK)		

Cal Date saved as DIOXCONCAL 07-FEB-00

aqueous Date saved as G003896018C_23-FEB-00

(A) change Comp T to end end T to start at 27:55

Entered by: JT Tabor Date: 02-23-00 Continued to: p34

Performances of this work observed by:	Disclosed to and understood by me:
Date:	Karen Riemide Date: 2/23/00
Date:	Date:

OCT 30 2000

STANDARD OPERATING PROCEDURE FOR DIOXIN/FURAN TECHNICAL DATA REVIEW

Originated by: Karen L. Tracy Date: 10/24/00

Approved by: Mary Dehnert Date: 10/24/00
Technical Reviewer

Approved by: Karen Riggs Date: 10/27/00
Management

Reviewed and Registered by QAU: Charles V. Lawrie Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This Standard Operating Procedure (SOP) describes the general procedures to be followed for the technical review of dioxin/furan analysis data.

III. References

A. SW-846, Method 8290. Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, 1994.
B. EPA Method 1613: Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Revision B, 1994, EPA 821-B-94-005.

IV. Definitions

None.

V. Procedures

A. Analyst Review of Analytical Data

1. Review the initial calibration curve and check the response factor summary sheet. Relative standard deviation (RSD) of response factors (RF) generated by isotope dilution should be $\leq 20\%$. The RSDs for RFs generated by internal standard should be $\leq 35\%$.

2. Check to verify that the PFK resolution checks meet the criteria found in EPA Method 1613B, Section 10.1.2.

3. Verify that the window set solution and column performance check meet the criteria found in EPA Method 1613B, Sections 10.3 and 10.4.

4. Check the continuing calibration results against EPA Method 1613B, Table 6 "VER" requirements. Flag any isomer analyte that fails this criteria and mark with a post-it note.

5. Review all chromatograms and analyte hits for the criteria in Method 1613, Revision B, Section 16.0. If the analyst deems a peak to be a valid hit even though it fails to meet one or more of the criteria found in Method 1613, Revision B, Section 16.0, an estimated maximum possible concentration can be calculated for this analyte following guidance in Method 8290, Revision 0, 1994, Section 7.9.5.2.1 and should be flagged as such in the raw data.

6. Once the analyst has completed review of the data as outlined above, he/she should place his/her initials and the date on the first chromatogram output for each run.

B. Secondary Technical Review of Analytical Data

1. Check the transcription of the extract number, sample identification, and sample weight/ volume from the preparation laboratory record book (LRB) onto the OPUS forms for each sample.

2. Check to see that all 2,3,7,8-isomers are accounted for, concentration reported, flagged for reasons not used, or obviously not present in each sample.

3. Spot-check continuing calibrations. Review any exceedances noted in V.A.4.

4. Double check that there are no co-eluting ethers contributing to the furans in each sample.

5. Check the lock mass check for each function to ensure that there are no variations > 20% that could impact reported results.

6. Flag any internal standard recoveries that are outside 40-135% (or 25-150% depending on project requirements) on the OPUS form. Tag these flagged forms with post-it notes for ease in highlighting final forms and report

7. Check calculations for all Estimated Maximum Possible Concentrations (EMPCs) and spot check the calculations for the concentrations detected.

8. Review HRMS LRB for the criteria listed in Section V.D.1.

9. Return the data to the analyst for corrections, updates, or reprints if there are any. The analyst should resave any data file that has been changed.

10. Review all corrections made by analyst.

11. Repeat steps 1-11 for second column confirmation data.

12. Format electronic spreadsheet data as required by project.

13. Flag any internal standard recoveries that are outside the 40 – 135% recovery limit (or 25 – 150% recovery limit, depending on project requirements) in the spreadsheet.

14. Add and flag any secondary column confirmation results into the spreadsheet.

15. Check that the QC check sample records have been updated within the quarter. If there are any new QC check sample results from the current project, add this information to the binder. The QC check samples will be Cambridge Isotopes standard reference materials (EDF-2526 or EDF-2513). Acceptability criteria are for concentrations in the QC check sample to be within 30% difference of consensus value

16. Document the secondary technical review on the data check list (Attachment A) and include in project records.

C. Technical Review of Dioxin/Furan Data Spreadsheets

1. Spot check to ensure that all calculations in the spreadsheets are correct (e.g. percent relative difference, percent recovery, standard deviation, method detection limit, etc.) and that sample data in the spreadsheet corresponds to the raw data.

2. Verify that correct units are used and that all header information is correct and complete and in the correct format required by the client.

3. Check that any data discussed in the report text is consistent with the data in the report tables.

4. Return to secondary technical reviewer for corrections, updates, or reprints.

5. Review all corrections made.

6. Document this review by initialing the concurrence box on the report internal review block.

D. Technical Review of Preparation Data for Dioxin/Furan Analysis

1. Review all laboratory record books (LRBs) used for the project. Check for completeness and accuracy of information, ensure that proper correction techniques have been used, sign the bottom of the page, and make a copy of the pages needed for the study file. LRBs include: Sample log-in, standard log-in, sample preparation, and extract chain-of-custody (COC).

2. Verify that the original COC and the miniaturized copy of the COC in the sample log-in LRB are identical.

E. Safety

N/A

VI. Revision History

None

Attachment A

Data Review Checklist
for
Samples Analyzed on :
OPUS Filename:

Initials/Date	Activity
	Check transcription of extract no., sample ID, and sample weight from prep LRB into OPUS forms for each sample.
	Check to see that all 2,3,7,8-isomers are accounted for: concentration reported, flagged for reason not used, or obviously not present in each sample.
	Double check that there are no ethers contributing to furans in each sample.
	Review lock mass checks for any variations, which could affect reported results.
	Flag any recoveries outside 40-135%/25-150% on OPUS form. Tag flagged forms with post-its for ease in highlighting final forms and report.
	Return data to analyst for corrections.
	Analyst makes corrections, updates, reprints and resaves OPUS data.
	Corrected OPUS data saved as ASCII text file. **ASCII text file Filename:**
	ASCII file pulled over to PC
	ASCII file imported to Quattro Pro or Excel **QPRO or Excel Filename:**
	Format spreadsheet data
	Flag in spreadsheet file any recoveries outside 40-135%/25-150% as on OPUS forms.
	Add and flag second column confirmation results in spreadsheet file.
	Check calculations for EMPCs.
	Review all LRBs used for project, check for completeness of information, ensure that the proper correction techniques have been used, sign the bottom of the page, and make a copy of the pages needed for the study file.
	Verify that the original COC and the miniturized copy of the COC in the sample log-in LRB are identical.
	Spot-check the calculations of concentrations.
	Verify that the daily calibrations, the window set mixture, and the percent valley are within the QC ranges allowed by the method.
	Print 2 copies of each finalized QPRO sample sheet. Place one in the OPUS printouts for each sample. Collect the others for the final report.
	Check to see if the QC check sample records have been updated within the last quarter. Add any new QC sample results from this project.

Manual Number: BATTELLE COPY

Battelle SOP Number: ASAT. II-011-00

OCT 30 2000

STANDARD OPERATING PROCEDURE
FOR USING ELECTRONIC AND MECHANICAL BALANCES

Originated by: _____ Date: 10/24/00

Approved by: _____ Date: 10/24/00
Technical Reviewer

Approved by: _____ Date: 10/20/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

_____ Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This Standard Operating Procedure (SOP) describes the general procedures to be followed for the operation, calibration, and maintenance of electronic and mechanical balances.

III. References

A. Balance manual, which if obtainable, should be readily available to users.

B. Battelle SOP ASAT.I-002-00, Use of Equipment Logbooks.

IV. Definitions

A. Critical weights are those that are critical to the scientific integrity of the experiment. Examples of critical weights would include the weights of samples, of chemicals used to prepare standards and of weights needed for calculations.

B. Non-critical weights are those that are estimates and/or used in applications in which the weight is not critical to the scientific integrity of the experiment. Recording of non-critical weights is not mandatory. Examples are weights determined for the rough splitting of samples, for balancing of centrifuge tubes, or for estimating weights of bulk reagents.

C. Responsible person is the individual assigned by management to be accountable for operations as described in equipment SOPs, including initiation and archival of Battelle Columbus Operations equipment logbooks.

V. Procedures

A. Operation of Balances Being Used for Determination of Critical Weights

1. No operator shall use the balance until properly trained. It is the responsibility of the user to ensure that the balance is functioning properly.

2. The operator should verify that the balance service interval sticker is current (i.e., the date of next service indication is a future or present date and not a past date). This sticker is provided by a trained service technician who is scheduled to do a yearly calibration of the balance. A balance which has not been properly calibrated should not be used.

3. Verify that the balance is level; if not, adjust the balance by using the built-in bubble indicators (if present).

4. Check the balance accuracy by using at least two different certified verification weights that are near the low and high end of the range to be weighed. The verification weights should have been certified within the last twelve months and should be ASTM class 2 weights or better. To test accuracy, the balance should read the verification weights to at least within 2 percent of their nominal weights unless other specifications are indicated in the equipment logbook. The balance must be checked for accuracy with the verification weights at the beginning of every month and recorded in the equipment log book. Additionally, at least once every day of use the balance should be checked for accuracy

with the verification weights and recorded in the study records. The accuracy of the balance must be rechecked with the verification weights if the balance is turned off or moved to a different platform.

5. Record the nominal and the determined values for the verification weights in the study record and/or equipment log. Check the determined values to make sure they are within the acceptable limits as established for the laboratory.

6. If the balance cannot pass the accuracy test, the particulars of the failure must be recorded in the equipment logbook, the balance must be labeled as out of calibration and not to be used, and the Responsible Person should be notified as soon as possible.

B. Operation of Balances Being Used for Non-Critical Weightings

1. No operator shall use any balance until properly trained on its use.

2. Mechanical balances may not have balance service interval stickers. They do not require accuracy verification with certified weights. It is recommended that these balances be checked for correct operation at zero grams before use and that the check weight identification, if used, and actual weights be documented in the study record or equipment logbook.

C. Maintenance

1. Whenever an operator suspects a balance is not functioning correctly, the operator shall label the balance "out of service" and the Responsible Person will be notified.

2. Balance repairs shall be performed by trained service technicians only.

3. An equipment log will be kept in accordance with Battelle SOP ASAT.I-002-00, Use of Equipment Logs. Equipment logs are required to be archived when the equipment is no longer in use or after the final quarter of 2002 and then every four years.

D. Safety

Care shall be taken not to damage the balance or calibration verification weights with fingerprints or residual chemicals on the pan. The use of gloves and weighing boats are recommended.

VI. Revision History

None

Attachment A

BATTELLE COLUMBUS OPERATIONS

EQUIPMENT LOGBOOK

BATTELLE X/I NUMBER _____

TYPE: _____

MAKE: _____

MODEL: _____

SERIAL NUMBER: _____

DEPARTMENT: _____

LOGBOOK DATES: FROM _____ TO _____

RESPONSIBLE PERSON: _____

SOP REFERENCE: _____

* For equipment comprised of components, use space below for additional X/I.

N/A = Not Applicable

Attachment B

ROUTINE MAINTENANCE - Battelle X/I or Serial No._____

Date	Initials	Describe the Nature of the Maintenance

Frequency/schedule _____
N/A = Not Applicable

Attachment C

NON-ROUTINE MAINTENANCE – Battelle X/I or Serial No._____

Date	Initials	Describe the Nature of the Problem, How & When Discovered, Remedial Action

N/A = Not Applicable

Attachment D

Balance Calibrations

Date	Initials	Ranges/Observed Mass (g)	Notes

*Frequency/schedule_____

N/A = Not application

BATTELLE X#:_____

Manual Number: BATTELLE COPY

Battelle SOP Number: ASAT. II-012-00

OCT 3 0 2000

**STANDARD OPERATING PROCEDURE (SOP)
FOR THE USE OF REFRIGERATORS AND FREEZERS
USED FOR DIOXIN-RELATED PROJECTS**

Originated by: _____ Date: 10/24/00

Approved by: _____ Date: 10/24/00
Technical Reviewer

Approved by: _____ Date: 10/26/00
Management

Reviewed and Registered by the Quality Assurance Coordinator:

_____ Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I./II. Scope/Purpose

This Standard Operating Procedure (SOP) is applicable to the routine operation, monitoring, and maintenance of the refrigerators and freezers for dioxin-related projects.

III. References

Battelle SOP ASAT.I-002-00, Use of Equipment Logbooks

IV. Definitions

None.

V. Procedures

A. Files and Records

1. Completed monthly temperature/maintenance logs will be placed in a binder maintained by the person responsible or designee for the refrigerator(s)/freezer(s) until archived. Temperature/maintenance logs are required to be archived when the equipment is no longer in use or at the end of the last quarter of 2002 and then every four years.

2. The thermometer calibration records or equivalent documentation will be maintained by the responsible person, designee or instrument lab until archived. Thermometer calibration records are required to be archived when the equipment is no longer in use or at the end of the last quarter of 2002 and then every four years.

B. Temperature/Maintenance Log

1. The temperature/maintenance log (similar to Attachment A) shall be posted on the door of the refrigerator/freezer and shall contain the following information:
   a. The month and year of the temperature/maintenance log.

   b. Type of equipment (refrigerator or freezer).

   c. Manufacturer.

   d. BMI or serial number. If these numbers are not available, a permanent unique number will be assigned to the unit.

   e. The location.

   f. The operating range. The assigned operating ranges will be documented on the unit. Common ranges can be as follows: approximately 0-4°C for refrigerators, ≤-20°C for freezers, and ≤-70°C for ultra low freezers.

   g. The monitoring frequency for all refrigerator/freezer units used for dioxin related projects will be every Battelle business day.

h. The thermometer number assigned to the unit (if applicable).

i. The calibration due date of the thermometer assigned to the unit.

j. The person or designee to notify in case of problem or issue.

k. Any additional safety concerns (if applicable).

l. Routine and Non-routine maintenance. This information will be documented on either this log or in the refrigerator/freezer logbook.

2. At the end of each month the temperature log will be placed in A.1 above.

C. Monitoring

1. The temperature shall be checked every Battelle business day by the responsible person or designee. If the responsible person is to be absent, he/she needs to designate an alternate to record the temperature until the responsible person returns.

2. If the monitoring is documented according to Attachment A, temperature recording should be performed as follows:

a. If the temperature of the refrigerator/freezer is within the designated operating range, then the time, temperature, date and initials will be filled in the initial reading space on the temperature log. The second reading set of boxes will be left blank.

b. If the temperature of the refrigerator/freezer is outside the operating range, the time, temperature, date and initials will be recorded in the initial reading space on the temperature log. Within several hours, the temperature will be rechecked and appropriate information will be recorded in the second reading set of boxes.

c. If the temperature is still outside the operating range, the responsible person or designee will be notified. The person notified, along with the date and initials of the person monitoring will be documented.

d. The person responding to a deviation shall document the corrective action/comments, the date, and their initials appropriately.

e. If there is a known mechanical failure for an extended period, the contents of the unit will be moved at the responsible person's discretion. The new location(s), date, and initials of the person(s) transferring the material will be documented on the temperature/maintenance log or in the refrigerator/freezer logbook.

D. Use

1. It will be the responsibility of the person storing the material to place it in a refrigerator/freezer with the correct operating range.

2. If the refrigerator/freezer is equipped with a locking device it shall be kept locked as deemed necessary by the responsible person (e.g. radiolabeled material storage). Keys

or combinations to the refrigerator/freezer shall be made available to the personnel requiring access to the refrigerator/freezer.

E. Calibration and Maintenance

The thermometers shall be calibrated in accordance with SOP ASAT.II-013-00. Units shall be defrosted as needed. Compressor coils shall be maintenanced as needed by service personnel.

F. Safety

1. Certain refrigerator/freezers may represent a safety hazard due to poor ventilation, CO_2, presence of radioactive isotopes, etc. All units shall be properly labeled on the outside door with the appropriate means of notification of such hazards.

2. Refrigerated or frozen flammables must be stored in approved explosion-proof refrigerators/freezers.

3. All personnel entering refrigerators/freezers, including walk-in refrigerators/freezers must be sure that they have provided themselves with adequate protection and precautions such as protective clothing, eyewear, or supplemental ventilation to ensure undue risk is not taken.

G. Quality Control

The temperature/maintenance logs will be reviewed, dated and initialed for compliance with this SOP by the area supervisor or his/her designee on a weekly basis. If a deviation from this SOP is noted, the deviation will be documented and maintained with the refrigerator/freezer logbook.

VI. Revision History

This is the first version of this SOP.

ATTACHMENT A

BATTELLE TEMPERATURE LOG

Month/Year:_____ Operating Range:_____
Type Of Equipment:_____ Calibration Due Date:_____
Type Of Equipment:_____ BMI ID# Or SN:_____
Manufacturer:_____ Monitoring Frequency:_____
Manufacturer:_____ Therm/Chart Rec. ID#:_____
Room #:_____
Responsible Person to Notify:_____ Phone Number:_____

	Initial Reading			Second Reading				Initial Reading			Second Reading		
Date	Time	Temp	Date/Initial	Time	Temp	Date/Initial	Date	Time	Temp	Date/Initial	Time	Temp	Date/Initial
1							17						
2							18						
3							19						
4							20						
5							21						
6							22						
7							23						
8							24						
9							25						
10							26						
11							27						
12							28						
13							29						
14							30						
15							31						
16													

Problem/Responsible Person Notified	*Date/Initial*	**Corrective Action/Comments**	**Date/Initial**

Routine Maintenance:

Non-routine Maintenance:

Reviewed by (Date/Initials):_____ Week 1 Review (Date/Initials):_____

Week 2 Review (Date/Initials):_____ Week 3 Review (Date/Initials):_____

Week 4 Review (Date/Initials):_____ Week 5 Review (date/Initials):_____

OCT 3 0 2000

STANDARD OPERATING PROCEDURE (SOP) FOR THE USE AND CALIBRATION OF DIGITAL AND GLASS THERMOMETERS

Originated by: Karen L Shay Date: 10/26/00

Approved by: Mary Schrock Date: 10/26/00
Technical Reviewer

Approved by: Karen Riggs Date: 10/27/00
Management

Reviewed and Registered by QAU:

Charles P. Laurie Date: 10/30/00

Battelle
505 King Avenue
Columbus, Ohio 43201

I. Scope

This standard operating procedure (SOP) describes the use and calibration of digital and glass thermometers and describes the methods that will be used to cross-calibrate monitoring thermometers against NIST-traceable or standardized (calibrated) thermometers.

II. Purpose

The purpose of this SOP is to describe the use and calibration of digital and glass thermometers that are used to monitor the temperature of refrigerators, freezers, drying ovens, water baths, and shipping containers. This equipment is used primarily for the sample storage, sample and reagent drying, and sample processing.

Digital and glass thermometers are available from retail and scientific suppliers; the selection of a unit should be based on the required accuracy, range, and the intended use of the thermometer. Of specific importance are:

- Units: the thermometer must provide readings in °C (centigrade)
- Accuracy: units intended for monitoring refrigerators, freezers, water baths, or sample condition
  must be accurate to ± 1.0°C.
- Range: the temperature range must bracket the expected range of the intended use of the thermometer.

III. References

None.

IV. Definitions

None.

V. Procedures

Each laboratory thermometer will be identified with a unique identification number (ID). This ID may be the serial number that is stamped on the thermometer itself or some other unique ID. Laboratory thermometers used for routine monitoring of refrigerators, freezers, water baths, etc. should be assigned to a piece of equipment and should not "travel" among units, if possible. In addition, if a thermometer calibrated for one temperature is moved to a different type of unit, the thermometer must be recalibrated prior to use (e.g., if a thermometer assigned to a refrigerator is used to monitor a water bath then the calibration factor determined at 4°C cannot be applied to a water bath at 60°C without verification).

A. Digital Thermometers

1. Operation

The analyst must review the literature provided with the thermometer prior to use to determine proper use of the features. Of critical importance is selection of the correct units if the thermometer has an °F (Fahrenheit) option. Unless specified in the project-specific Quality Assurance Project Plan (QAPP) (or similar document), temperature is always recorded in °C. In most cases the thermometer operation follows these steps:

a. Turn the unit on.
b. Verify that the LED display is complete. This is typically a self-test of the unit as the reading "188.8".
c. Allow the unit to equilibrate. Many units perform an initial self-test.
d. Verify that °C units are selected.
e. Begin temperature monitoring. The response of the thermometer may vary based on the temperature being measured. The analyst must observe the display to determine when the temperature has stabilized.
f. When the digital display is stable (on fluctuation) for 15 seconds, then the temperature reading is considered stable and is recorded.
g. Temperatures are recorded directly on the proper data sheet and in the proper laboratory record book (LRB) if needed.
h. To preserve the battery, turn off the unit if the time between measurements is greater than 15 minutes if possible.

Some thermometers may include a low-battery warning. Back-up batteries should be carried with the unit to avoid the loss of data.

If the temperature-monitoring unit includes a positionable probe, the probe should be placed in the appropriate matrix to avoid sudden temperature fluctuations. These matrices are: water:ethylene glycol (1:1) for temperatures between 4°C and -20°C and sand for temperatures < -20°C.

2. Maintenance

Other than quarterly calibration against a NIST traceable thermometer and cleaning of the probe with water and lint-free paper, no maintenance is required for digital thermometers. Use of a digital thermometer must be discontinued if any part of the LED display fails. Care should be taken to protect the thermometer probe if it protrudes from the unit.

3. Training

Each trainee must read and fully understand this SOP, and then demonstrate to the satisfaction of the Laboratory Manager, or designee, that he/she understands the operation of the thermometer of interest.

4. Safety
   There are no safety considerations associated with the operation of a digital thermometer. The project manager should be consulted for safety issues related to samples or the sampling site.

B. Glass Thermometers

1. Operation
   In most cases thermometer operation follows these steps:
   a. Place the thermometer in the equipment that the measurement is being taken for. If the thermometer is to stay inside a piece of equipment submerge the bulb of the thermometer in a jar filled with a suitable material for maintaining temperature.
   b. Allow the thermometer to equilibrate for a minimum of 20 minutes.
   c. Take the measurement from the top of the meniscus formed by the liquid inside the thermometer and then adjusted by the correction factor for the thermometer.
   d. Temperatures are adjusted for their correction factor and recorded directly on the proper data sheet.

2. Maintenance
   The thermometer must be calibrated once every year against a NIST traceable thermometer. Thermometers which are cracked, broken, or have separated liquid columns cannot provide accurate measurements and should be discarded or returned to the manufacturer for maintenance, unless the thermometer is chilled and all of the liquid returns to the bulb.

3. Training
   Each trainee must read and fully understand this SOP, and then demonstrate to the satisfaction of the Laboratory Manager, or designee, that he/she understands the operation of the thermometer of interest.

4. Safety
   Thermometers are easily broken if extreme caution is not used in handling. Broken thermometers, particularly those with mercury as the column liquid, pose several potential safety hazards. Care must be taken to avoid the sharp edges and any contact with escaping mercury. If a thermometer breaks, (1) a mercury spill kit should be used immediately, (2) the Hazardous Waste Coordinator and/or a Hazardous Waste Handler should be contacted, and (3) personnel exposure should be assessed with the Health and Safety Officer.

C. Calibration of Digital and Glass Thermometers

1. National Institute of Standards and Technology (NIST) Thermometer Calibration
   The NIST thermometer is calibrated annually by a professional thermometer service using approved ATSM methodology. These thermometers are very expensive and should not be used for routine monitoring. The calibration records received with these thermometers should be filed in the "Calibration" section of the Refrigerator Monitoring Log. These correction factors should be applied when performing the calibration described in this section.

2. Digital and Glass Thermometer Calibration
   Each digital thermometer will be calibrated quarterly using a NIST traceable thermometer. Each glass thermometer will be calibrated annually using a NIST traceable thermometer.

   To calibrate:

   a. Place the digital or glass thermometer and the NIST-traceable thermometers in an area which can sustain the temperature of interest according to the following table. In most cases, both thermometers are immersed in a matrix that will not react to the temperature rise and fall associated with opening the equipment door. The matrix used during cross-calibration should be noted on the calibration form (Attachment A).

Area of Use	Desired Temperature	Suggested Matrix for cross-calibration
Freezers	Below -20°C	Place in container of sand
Refrigerators	4°C±2°C	Place in a beaker of water:ethylene glycol (1:1)
Water baths	60°C or 100°C	Water bath
Drying ovens	100°C	Drying oven Place in a beaker of sand

   b. Shut the equipment door and allow the thermometers to stabilize for at least 30 minutes.

   c. Read the NIST-traceable and laboratory thermometers and document the measured temperature to the nearest whole number on the Thermometer Calibration Form (Attachment A). Do not remove either thermometer from the stabilizing matrix during this

time.

d. Perform units conversion, if required, and apply the appropriate calibration factor (if any) to the NIST-traceable thermometer reading.

e. Calculate the difference in the readings between the corrected NIST-traceable thermometer and the laboratory thermometer to the nearest whole number. This is the correction factor that should be applied to all temperature measurements using the laboratory thermometer. This correction factor should not be more than 3°C, or the digital or glass thermometer should be replaced. It should be noted that this range is designated by the Laboratory Manager and is not based on a recognized method. The following example illustrates the procedure.

Thermometer ID	Unit ID	NIST ID	MATRIX	NIST Measurement (Units)	NIST Correction Factor (Units)	Corrected NIST Measurement (Units)	Laboratory Thermometer Measurement (Units)	Correction Factor (Units)	Date/ Initials
Ref. 3	Refrig. 3	ERTCO G-57811	H_2O	20	+0.08	20.08	19	20.08 - 19 = +1	2-23-95 rlb

f. A tag with the calculated correction factor and the expiration date (one year from the date of calibration for glass thermometers and three months from the date of calibration for digital thermometers) should be affixed to the thermometer and noted on the temperature log of the unit the thermometer is monitoring, if applicable.

3. Thermometer Care

NIST traceable thermometers should always be stored in their protective cases. Laboratory thermometers should be secured within the equipment being monitored. Thermometers, which are cracked, broken, or have separated liquid columns cannot provide accurate measurements. In the former two cases the thermometer should be discarded in the appropriate waste stream. In some cases it is possible to re-unite a split column by chilling the thermometer so that all liquid returns to the bulb. If this is unsuccessful the thermometer should be returned to the manufacturer for maintenance (NIST-traceable) or discarded in the appropriate waste stream.

4. Calibration Training

To complete training in the calibration of laboratory thermometers, laboratory personnel should

a. Read this SOP.
b. Observe a trained staff member perform the calibration and documentation procedures (Section V.C).

c. Perform the calibration and documentation procedures under the supervision of a trained person.

VI. Revision History

This is the first version of this SOP.

Attachment A

Thermometer Calibration Form

Thermometer ID	Unit ID	NIST ID	Matrix	NIST Measurement	NIST Correction Factor	Corrected NIST Measurement	Laboratory Thermometer Measurement	Correction Factor	Date/ Initials

[1] Matrix in which thermometers were immersed during calibration.

[2] Correction Factor = [NIST calibrated thermometer Reading (°C)] - [Temperature Reading (°C)]
(°C + 9/5) + 32 = °F