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FINAL ADDENDUM 1 UNIFORM FEDERAL POLICY SAMPLING AND ANALYSIS PLAN SITE  
38 RUM POINT LANDFILL NSWC INDIAN HEAD MD  
01/01/2016  
AGVIQ ENVIRONMENTAL SERVICES

SAP Worksheet #1—Title and Approval Page

**Final  
Addendum 01  
Uniform Federal Policy-Sampling and Analysis Plan  
Site 38 – Rum Point Landfill  
Naval Support Facility Indian Head  
Indian Head, Maryland**

**Contract No. N62470-12-D-7004  
Task Order No. JU05**

Prepared for:

**Department of the Navy  
Naval Facilities Engineering Command  
Washington**

Prepared by:



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**January 2016**

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AGVIQ Project Manager



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NAVFAC Washington – Navy Chemist

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# Executive Summary

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## Introduction

AGVIQ, LLC (AGVIQ) was contracted by the Department of the Navy (Navy), Naval Facilities Engineering Command (NAVFAC) Washington to conduct a Remedial Action at Site 38 – Rum Point Landfill, at Naval Support Facility Indian Head (NSF-IH), Indian Head, Maryland under Contract No. N62470-12-D-7004 (SBRAC II), Task Order No. JU05. A Uniform Federal Policy-Sampling and Analysis Plan (UFP-SAP) was prepared and approved for Site 38 to ensure that environmental data collected in support of the Remedial Action are scientifically sound, of known and documented quality, and suitable for the intended purposes. During review of the Remedial Action Work Plan (RAWP), the U.S. Environmental Protection Agency (EPA) noted the omission of dioxin in post-excavation confirmation samples, and recommended its addition to post-confirmation sampling based on the reported one-time disposal of ash from a thermal treatment tank that was located on Range 3 Burn Point (Engineering Field Activity Chesapeake, 2003). In subsequent partnering discussions, it was agreed that a RAWP Addendum 01, to include a modified UFP-SAP and AHA, would be prepared to include management of ash waste, should it be uncovered while performing the removal action. This UFP-SAP Addendum 01 is provided as an addendum to the existing UFP-SAP to address post excavation confirmation sampling in the event that ash waste is uncovered during implementation of the remedial action at Site 38.

## UFP-SAP Addendum Worksheets

This UFP-SAP Addendum 01 includes the following worksheets:

- Project Management (Worksheets #1, #3, #4, #6, #7, #9, #10, #11, #12, #15)
- Measurements/Data Acquisition (Worksheets #19, #20, #23 to #25, #28, #30)
- Assessment Oversight (Worksheets #31, #32)
- Data Review (Worksheets #34 to #36)

All tables are embedded within the worksheets. Figures are provided in the RAWP. Field standard operating procedures are provided in Attachment A of the approved UFP-SAP, and the Department of Defense Environmental Laboratory Accreditation Program letter for the laboratory performing dioxin analysis is provided in Attachment B.

Upon approval of this UFP-SAP Addendum 01 by the Navy and the regulators, the ash excavation activities may proceed if ash is encountered.

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## Attachments

- A Analytical Methods
- B DoD ELAP Accreditation Letters

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## SAP Worksheet #3A—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Joseph Rail	Remedial Project Manager (RPM)	Naval Facilities Engineering Command (NAVFAC) Washington	202-685-3105	<a href="mailto:joseph.rail@navy.mil">joseph.rail@navy.mil</a>
Travis Wray	Installation Restoration (IR) Program Project Manager	Naval Support Facility Indian Head (NSF-IH)	(301) 744-2262	travis.wray@navy.mil
Cathy Gardner	Construction Manager	NAVFAC Washington, PWD South Potomac, Facility Engineering	301-744-2181	<a href="mailto:cathy.gardner@navy.mil">cathy.gardner@navy.mil</a>
Bob Thomson	RPM	EPA Region III	215-814-3357	<a href="mailto:thomson.bob@epa.gov">thomson.bob@epa.gov</a>
Curtis DeTore	RPM	MDE	410-537-3791	<a href="mailto:cdetore@mde.state.md.us">cdetore@mde.state.md.us</a>
Jim Nicotri	AGVIQ Project Manager (PM)	AGVIQ/CH2M HILL	339-832-4555	<a href="mailto:Jim.nicotri@ch2m.com">Jim.nicotri@ch2m.com</a>
Taylor Sword	AGVIQ Program Quality Manager	AGVIQ	757-217-5725	<a href="mailto:Taylor.sword@tikigag.com">Taylor.sword@tikigag.com</a>
Stephen Matney	AGVIQ Senior Technical Consultant	AGVIQ	757-213-8583	<a href="mailto:smatney@tikigag.com">smatney@tikigag.com</a>
Brenda Martinez	GCAL Project Manager	GCAL	225-769-4900	<a href="mailto:brenda.martinez@gcal.com">brenda.martinez@gcal.com</a>
Juan Acaron	Project Chemist/Data Validator	CH2M HILL	352-384-7002	<a href="mailto:juan.acaron@ch2m.com">juan.acaron@ch2m.com</a>
Joe Calixte	Site Construction Manager	AGVIQ	(757) 328-5568	<a href="mailto:jcalixte@tikigag.com">jcalixte@tikigag.com</a>
Steve Detwiler	Site Quality Control Manager	CH2M HILL	(828) 460-7979	<a href="mailto:steve.detwiler@ch2m.com">steve.detwiler@ch2m.com</a>
Jason Chalk	Site Safety Officer	AGVIQ	757.285-5951	jchalk@tikigag.com
<b>New Additions Below</b>				
Natalie Luciano	Laboratory PM	Eurofins Lancaster Laboratories Environmental, LLC	717-556-7354	<a href="mailto:NatalieLuciano@eurofinsUS.com">NatalieLuciano@eurofinsUS.com</a>
Laura Caulfield	Laboratory QA Manager	Eurofins Lancaster Laboratories Environmental, LLC	717-556-7354	<a href="mailto:LauraCaulfield@eurofinsUS.com">LauraCaulfield@eurofinsUS.com</a>

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## SAP Worksheet #4A—Project Personnel Sign-Off Sheet

The personnel listed below acknowledge their receipt, acceptance, and approval for the listed sections of this UFP-SAP for Remedial Action activities at Site 38 – Rum Point Landfill, NSF-IH, Indian Head, Maryland. The signed version of this document becomes a part of the Administrative Record for the NSF-IH, and a copy will be maintained in AGVIQ’s project file.

### Organization: AGVIQ

Name	Title/Role	Telephone Number (optional)	Signature/E-mail Receipt	Date SAP Read
Jim Nicotri	PM	781-585-5724		
Taylor Sword	Quality Assurance Manager (QAM)	757-213-8599		
Stephen Matney	Senior Technical Consultant	757-213-8583		
Joshua Painter	Health and Safety (H&S) Manager	303-993-9274		
Juan Acaron	Project Chemist /UFP-SAP Primary Author	352-384-7002		
Teresa Offner	UFP-SAP Primary Author	678-592-0024		
Joe Calixte	Site Construction Manager	(757) 328-5568		
Steve Detwiler	Site Quality Control Manager	(828) 460-7979		
Jason Chalk	Site Safety Officer	(757) 285-5951		

### Organization: GCAL Laboratories

Name	Title/Role	Telephone Number (optional)	Signature/E-mail Receipt	Date SAP Read
Brenda Martinez	GCAL Project Manager	225-769-4900		

### Organization: Eurofins Lancaster Laboratories Environmental, LLC

Name	Title/Role	Telephone Number (optional)	Signature/E-mail Receipt	Date SAP Read
Natalie Luciano	Laboratory PM	717-556-7354		
Laura Caulfield	Laboratory QA Manager	717-556-7354		

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## SAP Worksheet #6A—Communication Pathways

The following table adds the communication pathways with the lab performing the dioxin analysis to those in the approved UFP-SAP. All other pathways remain the same and so are not repeated here.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Reporting lab data quality issues	Laboratory QA officer	Laura Caulfield	717-556-7354 <a href="mailto:LauraCaulfield@eurofinsUS.com">LauraCaulfield@eurofinsUS.com</a>	Reports all quality assurance/quality control (QA/QC) issues associated with project field samples for dioxin to the Construction Quality Control Manager and Project Chemist as soon as identified, not to exceed 24 hours.

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## SAP Worksheet #7A—Personnel Responsibilities Table

The following table adds the responsibilities associated with dioxin analysis to those in the approved UFP-SAP. All other personnel responsibilities remain the same and so are not repeated here.

Name	Title/Role	Organizational Affiliation	Responsibilities
Laura Caulfield	Laboratory QA Manager	Eurofins Lancaster Laboratories Environmental, LLC	Responsible for audits, CAs, checks of QA performance within the laboratory

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## SAP Worksheet #9A—Project Scoping Session Participants Sheet

<b>Project Name:</b> Remedial Action for Site 38 – Rum Point Landfill <b>Projected Date(s) of Sampling:</b> Fall 2015 / Winter 2016 <b>PM:</b> Jim Nicotri – AGVIQ		<b>Site Name:</b> Site 38 – Rum Point Landfill <b>Site Location:</b> NSF-IH, Indian Head, Maryland		
<b>Date of Session:</b> September 3 and September 18, 2015 <b>Scoping Session Purpose:</b> Provide direction on sampling approach if dioxin is uncovered during implementation of Remedial Action.				
Name	Title/Project Role	Affiliation	Phone #	E-mail Address
Jim Nicotri	PM	AGVIQ	339-832-4555	<a href="mailto:Jim.nicotri@ch2m.com">Jim.nicotri@ch2m.com</a>
Joseph Rail	RPM	NAVFAC	202-685-3105	<a href="mailto:joseph.rail@navy.mil">joseph.rail@navy.mil</a>
Bob Thomson	RPM	EPA Region 3	215-814-3357	<a href="mailto:thomson.bob@epa.gov">thomson.bob@epa.gov</a>
Martin Gehlhaus	Toxicologist	EPA Region 3	215-814-3359	<a href="mailto:Gehlhaus.Martin@epa.gov">Gehlhaus.Martin@epa.gov</a>
Judy Solomon	Chemist	NAVFAC	757-322-4744	<a href="mailto:judy.solomon@navy.mil">judy.solomon@navy.mil</a>
Juan Acaron	Chemist	CH2M HILL	352-384-7002	<a href="mailto:juan.acaron@ch2m.com">juan.acaron@ch2m.com</a>
Theresa Offner	Geologist	CH2M HILL	(678) 592-0024	<a href="mailto:Teresa.Offner@CH2M.com">Teresa.Offner@CH2M.com</a>

## Site 38

### September 3, 2015

#### Comments/Decisions

A meeting was held on September 3, 2015 to discuss concerns regarding the addition of dioxin as an analytical requirement to the existing RAWP and UFP-SAP for Site 38. Concerns were expressed that delay due to approvals for addenda to the RAWP and UFP-SAP would result in the project not reaching completion due to approaching winter weather and the end of construction season. Because ash reportedly was disposed on a one-time bases but is not known to be present in the landfill and was not observed during previous trenching activities, assuming ash is present and delaying start of the removal action is not the best use of the project budget.

The meeting included a discussion of procedures that would prevent shut-down of the project if ash were discovered. It was decided the path forward should include removal and segregation of the material, if discovered, so that excavation of landfill material could continue. Handling and sampling for waste characterization and transportation and disposal of the ash waste material could then be performed while still continuing to complete the removal action in the landfill footprint. The UFP-SAP Addendum would include only new worksheets (not revisions to all the existing sheets) to include additional sample number and analyses.

#### Action Items

Develop a Technical Memorandum proposing elements for a UFP-SAP Addendum and conduct a meeting with the partnering team to agree on the sampling and analysis approach for dioxin in the event that ash is identified while performing the excavation at Site 38.

## September 18, 2015

### **Comments/Decisions**

A Technical Memorandum describing the path forward for Site 38 was provided to the partnering team. It was agreed during the call that the RAWP Addendum 01 and UFP-SAP Addendum 01 will be prepared simultaneously with implementation of preparatory tasks remedial action (attaining approval of the ESS, base permits, mobilization and pre-construction meetings). Landfill excavation work is not expected to begin until approximately 8 weeks after initial mobilization to the site. During this time period, the RAWP Addendum 01 and UFP-SAP Addendum 01 will be developed and submitted for approval.

### **Action Items**

Develop a **RAWP Addendum 01** to include the following:

- As each layer is excavated, the ash waste will be manually/visually screened to remove any debris or potential MEC items per the approved ESS. If any MEC is identified, the SUXOS will inspect and evaluate the item and determine if it is MPPEH, MEC, or MDAS. If it is determined to be MPPEH or MEC all work will stop and the Navy will be contacted for direction on how to proceed as per the approved ESS. If it is determined to be MDAS, the item will be tagged and placed in the metal container in the support zone for recycling
- After the ash waste area has been cleared of potential MEC, the ash waste will be placed in a lined roll-off container. Once the roll-off is full, it will be inspected by EODTECHDIV and confirmed again to be MEC/MPPEH free. The roll-off will then be covered and moved to the support zone in an area adjacent to but separate from the rest of the stockpiled material. Ash waste containers will be clearly labeled as described in the approved RAWP, Section 3.2.2 (e.g., Waste - pending analysis).
- Collect a composite sample of the ash material from the roll-off and ship to a laboratory for dioxin analysis only to determine if dioxin is present in the ash material and if the concentration is above 1 part per billion (ppb).
- Cover the material in the roll-off; continue with excavation of the landfill, as planned
- If dioxin is not present in the sample collected, the ash material will be stockpiled with other excavated soils and sampled for waste characterization in accordance with the existing Work Plan (TCLP VOCs, TCLP SVOCs, PCBs, etc.) and disposed with other excavated soils from the landfill.
- If dioxin is present in the sample above criteria, the waste will be characterized separately for parameters required by a facility licensed to receive waste containing dioxin at concentrations detected.

Develop a **UFP-SAP Addendum 01** to include the following:

Because the potential for buried ash is based on the statement, “ash may have been disposed one time,” it is assumed that the amount of ash that might be uncovered would be less than or equal to one truckload (no more than 20 cubic yards). The Final Design specifies that the depth of the landfill varies from 1 foot at the edges to approximately 7 feet below ground surface at its deepest point. So the need to analyze confirmation samples for dioxin is based on the following:

- If dioxin-impacted ash does not extend to the base of the landfill (i.e. the excavation depth to native soil), then confirmation samples will not be collected for dioxin analysis.
- If ash does extend to the base of the excavation but dioxin is not detected in the ash, then no dioxin analysis will be performed as part of the confirmation samples.
- If dioxin-impacted ash does extend to the base of the excavation and dioxin is detected in the ash, then the confirmation sample for that grid or grids will be analyzed for dioxin (as outlined below).

- If dioxin is detected in the confirmation sample at concentrations exceeding criteria, then up to 3 background samples will be collected from areas outside of the landfill to evaluate whether the presence of dioxin in the confirmation sample is attributable to the landfill contents or is consistent with background dioxin concentrations.
- Dioxin analysis will be performed using EPA Method 8290A (provided in Attachment A), which will provide concentrations for congeners of dioxin so that the 2,3,7,8 TCDD TEQ can be calculated.
- The 2,3,7,8 TCDD TEQ will be calculated in accordance with the “World Health Organization re-evaluation of dioxin toxic equivalency factors”, April 2007, WHO 2005 TEF update (provided in Attachment A).

Results will be provided to the Indian Head Partnering Team for determination of the path forward.

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## **SAP Worksheet #10A—Conceptual Site Model**

The conceptual site model presented in the RAWP is complete, with the exception of the identification of dioxin as a chemical of potential concern if ash is uncovered in the landfill.

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## SAP Worksheet #11A—Project Quality Objectives/Systematic Planning Process Statements

This section presents the project quality objectives (PQOs) for evaluating dioxin for the Site 38 Remedial Action in the format specified by the UFP-SAP guidance.

### **What is the environmental question that is being answered?**

Problem Definition – The site is an unlined landfill used from an unknown date to December 1989 that potentially contains ash. This UFP-SAP will confirm completion of the selected remedy for soil by verifying removal of waste within the landfill footprint and confirmation that the remaining soil is not impacted by the disposed wastes, including dioxin that may be present from past disposal of ash at the site.

**Environmental Questions to be Answered** - The environmental question/problem to be addressed upon completion of the remedial action at Site 38 is:

Is the landfill closed in a manner that protects human health and the environment in accordance with applicable and relevant State of Maryland solid waste management regulations?

### **Who will use the data?**

The data will be used by the Indian Head Installation Restoration Team (IHIRT) (Navy, EPA Region III, MDE) to determine if the landfill is closed in a manner that protects human health and the environment in accordance with state of Maryland solid waste management regulations, or if additional excavation is required.

AGVIQ will also use the data to prepare a Construction Completion Report, which will document the field activities and analytical results. Chemists will use the data to evaluate overall data quality with respect to subcontracted laboratories.

### **What are the Project Action Limits (PALs)?**

The PALs for post-excavation dioxin sampling data will be the EPA Region 3 Risk Based Concentrations for 2,3,7,8-TCDD for soil. Data will be used to calculate 2,3,7,8-TCDD Toxicity Equivalence Quotient (TEQ) value for congeners with a Toxic Equivalence Factor (TEF) greater than zero, as defined in the “World Health Organization re-evaluation of dioxin toxic equivalency factors”, WHO 2005 TEF update (included in Attachment A).. The data will be given to the IHIRT who will determine if additional excavation is required (see Worksheet #15).

### **What will the data be used for?**

The data will be used to determine if additional excavation is required or not, in accordance with applicable and relevant State of Maryland solid waste management regulations.

### **What types of data are needed (matrix, target analytes, analytical groups, field screening, onsite analytical or offsite laboratory techniques, sampling techniques)?**

This UFP-SAP Addendum 01 provides details for the collection and analyses (For dioxin/furans, Method 8290A) of confirmation soil samples in support of the selected remedy provided in the ROD. Worksheet #17 presents detailed information on the types of data needed for this project. All samples will be analyzed by an offsite laboratory. All samples will be collected in accordance with the standard operating procedures listed on Worksheet #21 and presented in Attachment A of the approved UFP-SAP. Confirmation samples will be analyzed for dioxins/furans in accordance Method 8290A included in Attachment A of the Addendum 01.

## **SAP Worksheet #11A—Project Quality Objectives/Systematic Planning Process Statements (continued)**

### **How “good” do the data need to be in order to support the environmental decision?**

The data will be of the quality and quantity required to meet the project objective of determining whether the remaining soil, post excavation, presents associated human health risks. Additional information associated with the precision, bias, sensitivity, representativeness and comparability of the data is provided in this worksheet and in Worksheets #12, #15, #19, #20, #24, and #28.

### **How will data be used when the laboratory-specific limits of detection are greater than the PALs?**

Worksheet #15 presents analytical methodology and limits. In addition to listing the particular analytes, PALs, and limits, it also identifies where limits of detection (LODs) are greater than PALs. Although this information was considered when planning the analytical protocol for the site and may lead to some uncertainty, it does not prevent conclusions from being drawn with respect to the objectives of the Remedial Action, for the following reasons:

1. Samples collected will be analyzed for dioxins/furans that will be used by the IHIRT to determine if additional excavation is needed or not. Analyzing for the specified analytes is needed to calculate the 2,3,7,8 TCDD TEQ using the latest WHO TEFs which is appropriate for satisfying this objective, as well as making decisions about whether further action is warranted.
2. Even though some LODs may be greater than their respective PALs, detection limits (DLs) are close to and may be less than the applicable PALs. When this occurs, the laboratory instrumentation would report a constituent, if detected, at a concentration greater than its DL; such a result would be J-qualified as estimated because it is less than the limit of quantitation (LOQ). In addition, congeners returning a Non-Detect will use a value of zero “0” in the calculation of the TEQ in accordance with current practice at Indian Head.
3. Any resulting uncertainty will be discussed in the data quality evaluation.

### **How much data should be collected (number of samples for each analytical group, matrix, and concentration)?**

The number and type of post excavation confirmation samples to be analyzed for dioxins/furans is dependent on the PQOs listed at the end of this Worksheet. Detailed information on data collection is provided in Worksheet #17 of the UFP-SAP. The estimated quantities and types of QA/QC samples are detailed in Worksheet #20 of this UFP-SAP Addendum 01.

### **Where, when, and how should the data be collected/generated?**

- Detailed information on when the data will be collected is provided in Worksheet #16 of the UFP-SAP.
- Detailed information on where and how the data will be collected is provided in Worksheet #9, and Worksheets #14 and # 17 of the UFP-SAP.
- All sampling will be performed in general accordance with the procedures described in the SOPs listed on Worksheet #21 of the UFP-SAP. SOPs are provided in Attachment A of the UFP-SAP.

### **Who will collect and generate the data? How will the data be reported?**

- The AGVIQ field team will collect the samples after excavation is complete.
- The samples will be shipped via overnight courier to Eurofins Lancaster Laboratories.
- All chemical data generated will be submitted to AGVIQ. Once received and reviewed by AGVIQ, all chemical data will be validated internally.
- All chemical and field sampling data will be summarized in a table and will be submitted to the IHIRT as an official submittal for review and use in determining if additional excavation is needed or not.

## SAP Worksheet #11A—Project Quality Objectives/Systematic Planning Process Statements (continued)

### How will the data be archived?

Data will be archived in accordance with Navy SBRAC II contractual requirements. The analytical data will be loaded to the Navy Installation Restoration Information Solution (NIRIS) database once complete.

### List the PQOs in the form of if/then qualitative and quantitative statements.

- If ash is uncovered but does not extend to the base of the landfill (i.e. the excavation depth), then confirmation samples will not be analyzed for dioxin/furans.
- If ash is uncovered and does extend to the base of the excavation but dioxin is not detected in the composite ash waste sample (described in the RAWP Addendum 01), then confirmation samples will not be analyzed for dioxin/furans.
- If ash is uncovered and does extend to the base of the excavation and dioxin/furan is detected in the composite ash waste sample (above), then the confirmation sample for that grid or grids will be analyzed for dioxin/furans.
- Further, if dioxin/furan is detected in the confirmation sample at concentrations exceeding criteria, then up to 3 background samples will be collected from areas outside of the landfill to evaluate whether the dioxin/furan in the confirmation sample is attributable to the landfill contents or is consistent with background dioxin/furan concentrations
- If the IHIRT determines that the concentrations of dioxin (2,3,7,8 TCDD TEQ) does not exceed the cleanup criteria or background concentrations, then the remedial action will be considered complete.
- If the IHIRT determines that the concentrations of detected constituents (2,3,7,8 TCDD TEQ) exceed the cleanup criteria and background concentrations, the IHIRT will determine if additional excavation is needed and which grids require additional excavation.

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## SAP Worksheet #12-6—Measurement Performance Criteria Table - Field QC Samples

**Matrix:** Surface and Subsurface Soil

**Analytical Group:** Dioxin

**Concentration Level:** Low (SW-846 8290A)

QC Sample <sup>1</sup>	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicate	Dioxin/Furans	One per 10 field samples	Precision	%RPD ≤20%	S & A
Equipment Rinseate Blank	Dioxin/Furans	1 per day of sampling for decontaminated equipment. 1 per lot for disposable equipment	Bias / Contamination	Target analytes ≤ 1/2 LOQ	S
Temperature Blank	Dioxin/Furans	One per cooler	Accuracy / Representativeness	≤ 6°C	S

<sup>1</sup>MS/MSD is described on Worksheet 28.

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## SAP Worksheet #15-6—Reference Limits and Evaluation Table

**Matrix:** Surface and Subsurface Soil

**Analytical Group:** Dioxin/Furans, Method 8290A

**Concentration Level:** Low

Analyte	CAS No.	WHO 2005 TEF <sup>1</sup>	RSLs <sup>1</sup> Residential Soil Adjusted (pg/g)	PQL Goal (pg/g)	Laboratory Specific Limits (pg/g)			Accuracy Control Limit (%R)		Precision Control Limit (% RPD)
					LOQs	LODs	DLs			
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	1	4.8	4.8	1	0.2	0.1	70	128	25
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4	1	4.8	4.8	5	1	0.4	74	125	
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	0.1	48	48	5	1	0.3	72	131	
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	0.1	48	48	5	1	0.4	74	134	
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	0.1	48	48	5	1	0.3	71	138	
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9	0.01	480	480	5	1	0.5	76	125	
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	0.0003	16000	16000	10	2	1	73	135	
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	0.1	48	48	5	1	0.3	75	135	
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	0.03	160	160	5	1	0.4	77	131	
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	0.3	16	16	5	1	0.3	75	128	
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	0.1	48	48	5	1	0.3	77	130	
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	0.1	48	48	5	1	0.3	73	134	
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	0.1	48	48	5	1	0.5	74	135	
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	0.1	48	48	5	1	0.3	74	133	
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	0.01	480	480	5	1	0.3	73	135	
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	0.01	480	480	5	1	0.4	72	131	
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0	0.0003	16000	16000	10	2	0.5	66	144	
Total Tetrachlorodibenzo-p-dioxin (TCDD)	41903-57-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Total Pentachlorodibenzo-p-dioxin (PeCDD)	36088-22-9	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Hexachlorodibenzo-p-dioxin (HxCDD)	34465-46-8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Heptachlorodibenzo-p-dioxin (HpCDD)	37871-00-4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

RSLs<sup>1</sup>: Based on RSLs Residential Soil Adjusted (November, 2015) for 2,3,7,8-TCDD of 4.8 pg/g.

1 - Note: The comparison above is useful for demonstrating that the concentration level is appropriate given the data needs. For each field sample, a total TEQ is calculated by summing the contribution of each detected result multiplied by that compound's TEF. The Total TEQ is then compared to the screening level for 2,3,7,8-TCDD. TEF factors were taken from the "World Health Organization re-evaluation of dioxin toxic equivalency factors", dated April 2007, WHO 2005 TEF update (included in Attachment A).

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## SAP Worksheet #19A—Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference <sup>1</sup>	Containers (number, size, and type)	Sample Volume <sup>2</sup> (units)	Preservation Requirements (chemical, temperature, light-protected)	Maximum Holding Time <sup>3</sup> (preparation / analysis)
SS, SB	Dioxins/Furans	SW846 8290A/9015119	1 of 4 oz glass soil jar	100 g	4 ± 2 °C	30 days/45 days

SS = Surface Soil

SB = Subsurface Soil

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

<sup>2</sup> Provide the minimum sample volume or mass requirement if it differs from the container volume.

<sup>3</sup> Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

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## SAP Worksheet #20A—Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations <sup>2</sup>	No. of Field Duplicates	No. of Field Triplicates	No. of MS/MSDs <sup>1</sup>	No. of Field Blanks <sup>3</sup>	No. of Equip. Blanks <sup>3</sup>	No. of VOA Trip Blanks <sup>3</sup>	Total No. of Samples to Lab <sup>5</sup>
SS, SB	Dioxins/Furans (see Note 4)	0 to 12 (see Note 5)	0 to 2	0	0 to 1	TBD	0 to 1	TBD	0 to 18

SS = Surface Soil

SB = Subsurface Soil

<sup>1</sup> Although the MS/MSD is not typically considered a field QC, it is included here because location determination is often established in the field.

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

<sup>3</sup> The number of equipment blanks and trip blanks is based on a fundamental assumption of the number of sampling days each site will require. It was assumed that the soil confirmation sampling will occupy a total of one days.

<sup>4</sup> Dioxin/Furans will only be sampled if the conditions on Worksheet 11 are met.

<sup>5</sup> The number of samples is dependent on how many sampling grids the ash waste actually touches. Only the grid in which the ash extends to the floor of the excavation (native soil) will be sampled.

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## SAP Worksheet #23A—Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and / or Number	Date Last Revisited if not Revised	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM	Modified for Project Work? <sup>1</sup> (y/n)
9015119	Determination of Tetra- Through Octa-Chlorinated Dioxins and Furans using HRGC/HRMS by EPA 1613B or SW-846 8290A (Rev. 8, 6/22/15).		Definitive	SS, SB / DIOXIN/FURANS	HRGC/HRMS	Eurofins Lancaster Laboratories Environmental, LLC	None	N
9015412	DFS HRGC/HRMS Preventative and Corrective Maintenance (Rev. 2, 1/23/15).		N/A	Various	HRGC/HRMS	Eurofins Lancaster Laboratories Environmental, LLC	None	N

SS = Surface Soil

SB = Subsurface Soil

<sup>1</sup> If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy QAO.

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## SAP Worksheet #24A—Analytical Instrument Calibration Table

Instrument <sup>3</sup>	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA <sup>2</sup>	SOP Reference <sup>1</sup>
GC/MS (Dioxins/Furans)	GC Column Performance Check	Prior to ICAL or CCV	Peak separation between 2,3,7,8-TCDD and other TCCD Isomers result in a valley $\leq 25\%$ ; ID of all first and last eluters of the eight homologue retention time windows; absolute retention times for switching from one homologous series to the next $\geq 10$ sec. for all components in the mixture.	Correct the problem and re-analyze prior to proceeding.	Analyst, Supervisor	Eurofins SOP 9015119
	ICAL	Prior to sample analysis	Ion Abundance Ratios must be within method acceptance limits. S/N Ratio: $\geq 10$ for all target ions. RSD for each native compound $\leq 20\%$ . RSD $\leq 20\%$ for the RFs for the 9 labeled internal standards (ISs).	Correct problem then repeat ICAL.		
	CCV	At the beginning and end of each 12 hour analytical sequence.	Ion Abundance ratios must be within method acceptance limits; for unlabeled standards, RF within 80-120% of value established in ICAL; for labeled standards, RF within 30% of RF established in ICAL.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last acceptable CCV. End-of-run CCV: If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ RPD (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending CCV RFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new ICAL if it is analyzed within two hours. Otherwise reanalyze.		

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23A).

<sup>2</sup> Name or title of responsible person may be used.

<sup>3</sup> DoD QSM v. 4.2 is the basis for specifications on this table. Specifications are based on the analytical method that will be performed.

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## SAP Worksheet #25A—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

<b>Instrument / Equipment</b>	<b>Maintenance Activity</b>	<b>Testing Activity</b>	<b>Inspection Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Responsible Person<sup>2</sup></b>	<b>SOP Reference<sup>1</sup></b>
Thermo Fisher Scientific HRGC/HR/M	Injection port, column, ion source, others as needed	Calibration Check	Visual	As needed	Initial calibration or calibration verification passes method specifications	Perform additional maintenance prior to instrument calibration or calibration verification	Analyst / Supervisor	9015412

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23A).

<sup>2</sup> Name or title of responsible person may be used.

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## SAP Worksheet #28-6—Laboratory QC Samples Table

**Matrix:** Soil

**Analytical Group:** Dioxins

**Analytical Method/ SOP Reference:** SW846-8290A/ 9015119

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One for each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents, and the analytical system are under control.	Use project-specific criteria, if available. Otherwise, no analytes detected $\geq$ QL for the analyte or $\geq$ 5% of the associated regulatory limit for the analyte or $\geq$ 5% of the sample result for the analyte, whichever is greater, per method.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Analyst / Supervisor	Accuracy/Bias, Contamination	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One for each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents, and the analytical system are under control.	Refer to Worksheet 15-6. In-house statistical laboratory limits were provided since DoD QSM v. 4.2 does not specify.	If, however, any individual precision or accuracy falls outside the acceptable range, system performance is unacceptable for that compound. Correct the problem and re-prepare and reanalyze (Section 9.2 of EPA 1613B).	Analyst / Supervisor	Accuracy/Bias	Refer to Worksheet 15-6. In-house statistical laboratory limits were provided since DoD QSM v. 4.2 does not specify.
Matrix Spike (MS)	One per 20 normal field samples	Same as LCS.	Investigate repeated failures. No corrective action necessary. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.	Analyst / Supervisor	Accuracy/Bias	Same as LCS.
Matrix Spike Duplicate (MSD)	One per 20 normal field samples	Same as MS and refer to Worksheet 15-6.	Same as MS	Analyst / Supervisor	Accuracy/Bias, Precision	Same as MS and refer to Worksheet 15-6.
Internal standards (IS)	Every field sample, standard, and QC sample.	% recovery for each IS in the original sample (prior to dilutions) must be within 40-135%, per method.	Correct problem, then re-prepare and reanalyze the samples with failed IS. Apply Q-flag to all associated analytes if acceptance criteria are not met.	Analyst / Supervisor	Accuracy/Bias	% recovery for each IS in the original sample (prior to dilutions) must be within 40-135%, per method.

**Notes:**

The specifications in this table meet the requirement of DoD QSM 4.2.

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## SAP Worksheet #30A—Analytical Services Table

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization <sup>1</sup> (name and address, contact person, and telephone number)	Backup Laboratory / Organization (name and address, contact person, and telephone number)
SS, SB	Dioxin/Furans	Refer to Worksheet #18	SW846 8290A	7 Calendar-day TAT	Eurofins Lancaster Laboratories Environmental, LLC 2425 New Holland Pike Lancaster, PA 17601 717-656-2300	TBD

SS = Surface Soil  
 SB = Subsurface Soil

<sup>1</sup>If the laboratory is not known at time of SAP submission, put "TBD" in the column as a placeholder.

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## SAP Worksheet #31A—Planned Project Assessments Table

<b>Assessment Type</b>	<b>Frequency</b>	<b>Internal or External</b>	<b>Organization Performing Assessment</b>	<b>Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)</b>
Field Performance Audit	In accordance with SBRAC II program requirements	Int.	AGVIQ	Steve Detwiler Project QC Manager CH2M Hill	Jim Nicotri PM/AGVIQ	Steve Detwiler Project QC Manager CH2M Hill	Taylor Sword Program Quality Manager AGVIQ
Offsite Laboratory Technical Systems Audit	Laboratories must have current DoD ELAP accreditation which will identify the period of performance.	Ext.	Laboratory Accreditation Bureau in accordance with Department of Defense ELAP	TBD Laboratory Accreditation Bureau	Laura Caulfield Laboratory QA Officer Eurofins Lancaster	Laura Caulfield Laboratory QA Officer Eurofins Lancaster	Taylor Sword Activity Quality Manager AGVIQ

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## SAP Worksheet #32A—Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response (Name, Title, Org.)	Time Frame for Response
Field Performance Audit	Checklist and Written Audit Report	Jim Nicotri PM/AGVIQ	Within 1 week of audit	Memorandum	Steve Detwiler Project QC Manager CH2M Hill & Taylor Sword Quality Assurance Manager AGVIQ	Within 1 week of receipt of CA Form
Offsite Laboratory Technical Systems Audit	TBD by Laboratory Accreditation Bureau	Laura Caulfield Laboratory QA Officer Eurofins Lancaster Laboratories Environmental, LLC	Within 2 months of audit	Memorandum	TBD by Laboratory Accreditation Bureau	Within 2 months of receipt of initial notification.

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## SAP Worksheet #34-36A—Data Verification and Validation (Steps I and IIa/IIb) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
<b>Step 1</b>			
Field Notebooks	Field notebooks will be reviewed internally and placed into the project file for archival at project closeout.	Internal	Steve Detwiler CH2M Hill
COCs and Shipping Forms	COC forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the COC will be initialed by the reviewer, a copy of the COC retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	Internal / External	Steve Detwiler CH2M Hill
			Project Chemist: Juan Acaron/CH2M HILL
Sample Condition upon Receipt	Any discrepancies, missing, or broken containers will be communicated to the Project Chemist in the form of laboratory logins.	External	Project Chemist: Juan Acaron/CH2M HILL
Documentation of Laboratory Method Deviations	Laboratory method deviations will be discussed and approved by the Project Chemist. Documentation will be incorporated into the case narrative that becomes part of the final hardcopy data package.	Internal / External	Project Chemist: Juan Acaron/CH2M HILL
Electronic Data Deliverables	Electronic data deliverables will be compared against hardcopy laboratory results (10% check).	External	Project Chemist: Juan Acaron/CH2M HILL
Case Narrative	Case narratives will be reviewed by the data validator during the data validation process. This is verification that they were generated and are applicable to the data packages.	External	Data Validator: Herb Kelly/CH2M HILL
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Laboratory QA Officer: Laura Caulfield/Eurofins Lancaster
Laboratory Data	The data will be verified for completeness by the Project Chemist.	External	Project Chemist: Juan Acaron/CH2M HILL
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the site file. If CAs are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the QA site file. Periodically, and at the completion of site work, site file audit reports and CA forms will be reviewed internally to ensure that all appropriate CAs have been taken and that CA reports are attached. If CAs have not been taken, the site manager will be notified to ensure action is taken.	Internal	PM: Jim Nicotri /AGVIQ
			Project Chemist: Juan Acaron/CH2M HILL
CA Reports	CA reports will be reviewed by the Project Chemist or PM and placed into the project file for archival at project closeout.	External	PM: Jim Nicotri /AGVIQ
			Project Chemist: Juan Acaron/CH2M HILL

## SAP Worksheet #34-36A—Data Verification and Validation (Steps I and IIa/IIb) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
<b>Step IIa</b>			
Laboratory Methods	Ensure the laboratory analyzed samples using the correct methods.	External	Project Chemist: Juan Acaron/CH2M HILL
TCL and TAL (lists)	Ensure the laboratory reported all analytes from each analysis group as described in Worksheet #15A.	External	Project Chemist: Juan Acaron/CH2M HILL
RLs	Ensure the laboratory met the project-designated reporting limits as described in Worksheet #15A. If reporting limits were not met, the reason will be identified and documented.	External	Project Chemist: Juan Acaron/CH2M HILL
Laboratory SOPs	Ensure that approved analytical laboratory SOPs were followed.	External	Data Validator: Herb Kelly/CH2M HILL
<b>Step IIb</b>			
Sample Chronology	Holding times from collection to extraction or analysis and from extraction to analysis will be considered by the data validator during the data validation process.	External	Data Validator: Herb Kelly/CH2M HILL
Raw Data	10 percent review of raw data to confirm laboratory calculations.	External	Data Validator: Herb Kelly/CH2M HILL
Onsite Screening	All non-analytical field data will be reviewed against UFP-SAP requirements for completeness and accuracy based on the field calibration records.	Internal	Steve Detwiler CH2M Hill
Documentation of Method QC Results	Establish that all required QC samples were run and met limits.	External	Data Validator: Herb Kelly/CH2M HILL
Documentation of field QC Sample Results	Establish that all required UFP-SAP QC samples were run and met limits.	Internal	Project Chemist: Juan Acaron/CH2M HILL
			Data Validator: Herb Kelly/CH2M HILL
<b>Step 2b: Analytical Data Validation</b>			
Dioxins	Analytical methods and laboratory SOPs, as presented in this UFP-SAP, will be used to evaluate compliance against QA/QC criteria. QA/QC criteria for field QC samples are presented in Worksheet 12, target compound lists, LOQs, LODs, DLs, and limits for precision and accuracy are presented in Worksheet 15, QA/QC criteria for calibrations are presented in Worksheet 24, and QA/QC criteria for laboratory QC samples are presented in Worksheet 28. Data may be qualified if QA/QC exceedances have occurred. Data qualifiers will be those presented in Region III Modifications to National Functional Guidelines for Organic Data Review (EPA, September, 1994). Guidance from USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (EPA, August, 2014) may also be applicable.	External	Data Validator: Herb Kelly/CH2M HILL

Notes:

- Analytical methods and laboratory SOPs, as presented in this UFP-SAP, will be used to evaluate compliance against QA/QC criteria. QA/QC criteria for field QC samples are presented in Worksheet 12, target compound lists, LOQs, LODs, DLs, and limits for precision and accuracy are presented in Worksheet 15, QA/QC criteria for calibrations are presented in Worksheet 24, and QA/QC criteria for laboratory QC samples are presented in Worksheet 28. Data may be qualified if QA/QC exceedances have occurred. Data qualifiers will be those presented in Region III Modifications to National Functional Guidelines for Organic Data Review (EPA, September, 1994). Guidance from USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (EPA, August, 2014) may also be applicable.
- Internal or external is in relation to the data generator.

# **Attachment A**

## **Analytical Methods**

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## METHOD 8290A

### POLYCHLORINATED DIBENZO-*p*-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH- RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

#### 1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for the detection and quantitative measurement of polychlorinated dibenzo-*p*-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) in a variety of environmental matrices and at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. The following compounds have been determined by this method:

Compound	CAS Registry No. <sup>a</sup>
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin (PeCDD)	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin (HpCDD)	35822-46-9
1,2,3,4,5,6,7,8-Octachlorodibenzo- <i>p</i> -dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0

Total Tetrachlorodibenzo- <i>p</i> -dioxin (TCDD)	41903-57-5
Total Pentachlorodibenzo- <i>p</i> -dioxin (PeCDD)	36088-22-9
Total Hexachlorodibenzo- <i>p</i> -dioxin (HxCDD)	34465-46-8
Total Heptachlorodibenzo- <i>p</i> -dioxin (HpCDD)	37871-00-4
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3

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<sup>a</sup> Chemical Abstract Service Registry Number

1.2 This method requires the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits (MCLs), and other pertinent information. Samples containing concentrations of specific congeneric analytes (PCDDs and PCDFs) considered within the scope of this method that are greater than ten times the upper MCLs must be analyzed by a protocol designed for such concentration levels, e.g., Method 8280. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is described.

1.3 The sensitivity of this method is dependent upon the level of interferences within a given matrix. The calibration range of the method for a 1-L water sample is 10 to 2000 ppq for TCDD/TCDF and PeCDD/PeCDF, and 1.0 to 200 ppt for a 10-g soil, sediment, fly ash, or tissue sample for the same analytes (Table 1). Analysis of a one-tenth aliquot of the sample permits measurement of concentrations up to 10 times the upper MCL. The actual limits of quantitation will differ from the lower MCL, depending on the complexity of the matrix.

1.4 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced in residue analysis and skilled in HRGC/HRMS. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.6 Because of the extreme toxicity of these compounds, the analyst must take necessary precautions to prevent the exposure of laboratory personnel or others to materials known or believed to contain PCDDs or PCDFs. Typical infectious waste incinerators are not satisfactory devices for disposal of materials highly contaminated with PCDDs or PCDFs. A

laboratory planning to use these compounds should prepare a disposal plan. Additional safety instructions are outlined in Secs. 5.0.

## 2.0 SUMMARY OF METHOD

2.1 This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) analytical techniques. See Method 3500 for guidance on other appropriate extraction techniques.

2.2 If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination.

2.3 A designated amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still bottom, fuel oil, chemical reactor residue, fish tissue, or human adipose tissue is spiked with a solution containing specified amounts of each of the nine isotopically ( $^{13}\text{C}_{12}$ ) labeled PCDDs/PCDFs listed in the far left column of Table 2. The sample is then extracted according to a matrix-specific extraction procedure. Aqueous samples that contain 1 percent or more solids and solid samples that show an aqueous phase are filtered, the solid phase (including the filter) and the aqueous phase extracted separately, and the extracts combined before extract cleanup. The extraction procedures are:

- (a) Toluene -- Soxhlet extraction for soil, sediment, fly ash, and paper pulp samples
- (b) Methylene chloride -- liquid-liquid extraction for water samples
- (c) Toluene -- Dean-Stark extraction for fuel oil, and aqueous sludge samples
- (d) Toluene extraction for still bottom samples
- (e) Hexane/methylene chloride or methylene chloride -- Soxhlet extraction for fish tissue samples
- (f) Methylene chloride extraction for human adipose tissue samples
- (g) As another option, all solid samples (wet or dry) may be extracted with toluene using a Soxhlet/Dean Stark extraction system or using pressurized fluid extraction (PFE) (Method 3545) or microwave extraction (Method 3546).
- (h) Water samples may also be extracted using solid-phase extraction (SPE) by Method 3535.

The decision for the selection of an extraction procedure for chemical reactor residue samples is based on the appearance (consistency, viscosity) of the samples. Generally, the samples can be handled according to the procedure used for still bottom (or chemical sludge) samples.

2.4 The extracts are submitted to an acid-base washing treatment and dried. Following a solvent exchange step, the extracts are cleaned up by column chromatography on alumina, silica gel, and activated carbon.

2.4.1 The extracts from adipose tissue samples are treated with silica gel impregnated with sulfuric acid before chromatography on acidic silica gel, neutral alumina, and activated carbon.

2.4.2 Fish tissue and paper pulp extracts are subjected to an acid wash treatment only, prior to chromatography on alumina and activated carbon.

2.5 The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 10 to 50  $\mu\text{L}$  (depending on the matrix) of a nonane solution containing 50  $\text{pg}/\mu\text{L}$  of the

recovery standards  $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and  $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD (Table 2). The former is used to determine the percent recoveries of tetra- and pentachlorinated PCDD/PCDF congeners, while the latter is used to determine the percent recoveries of the hexa-, hepta- and octachlorinated PCDD/PCDF congeners.

2.6 An aliquot of the concentrated extract is injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving power of at least 10,000 (10 percent valley definition).

2.7 The identification of OCDD and nine of the fifteen 2,3,7,8-substituted congeners (Table 3), for which  $^{13}\text{C}$ -labeled standards are available in the sample fortification and recovery standard solutions (Table 2), is based on their elution at their exact retention time (within 0.005 retention time units measured in the routine calibration) and the simultaneous quantitation of the two most abundant ions in the molecular ion region. The remaining six 2,3,7,8-substituted congeners (i.e., 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF, and 1,2,3,4,7,8,9-HpCDF), for which no carbon-labeled internal standards are available in the sample fortification solution, and all other PCDD/PCDF congeners are identified when their relative retention times fall within their respective PCDD/PCDF retention time windows, as established from the routine calibration data, and the simultaneous quantitation of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to  $^{13}\text{C}_{12}$ -OCDD and the simultaneous quantitation of the two most abundant ions in the molecular ion region. Identification also is based on a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance ratios.

2.8 Quantitation of the individual congeners, total PCDDs, and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homologue, during which each calibration solution is analyzed once.

### 3.0 DEFINITIONS

#### 3.1 Method calibration limits (MCL)

3.1.1 Lower method calibration limit (LCML) is the low standard on the calibration curve.

3.1.2 Upper method calibration limit (UMCL) is the high standard on the calibration curve.

**NOTE:** The LCML and UCML values in Table 1 were those used during the method development process. Other calibration ranges may be used based on project data requirements.

3.2 Refer to Chapter One and the manufacturer's instructions for other definitions that may be relevant to this procedure.

### 4.0 INTERFERENCES

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data (see Refs. 1 and 2). All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific

selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. In addition, analysts should avoid using PVC gloves. Also refer to Method 8000 for a discussion of interferences.

4.2 The use of high-purity reagents and pesticide-grade solvents helps to minimize interference problems. Purification of solvents by distillation, in all glass systems, may be required.

4.3 Interferants coextracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated alkyldibenzofurans, that may be found at concentrations several orders of magnitude higher than that of the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established in Sec. 9.3.1.3. While cleanup techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve the sensitivity described in this method.

4.4 A high-resolution capillary column (60-m DB-5, J&W Scientific, or equivalent) is used in this method. However, no single column is known to resolve all 210 isomers. The 60-m DB-5 GC column is capable of 2,3,7,8-TCDD isomer specificity (Sec. 6.2.1). In order to determine the concentration of the 2,3,7,8-TCDF (if detected on the DB-5 column), the sample extract must be reanalyzed on a column capable of 2,3,7,8-TCDF isomer specificity (e.g., DB-225, SP-2330, SP-2331, or equivalent).

## 5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in the chemical analysis of samples suspected to contain PCDDs/PCDFs. Additional information on laboratory safety is given in Refs. 3, 4 and 5.

5.2 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCDDs or PCDFs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed.

5.3 The following safety practices are excerpts from EPA Method 1613, Sec. 5 (October 1994 version), amended as necessary for use in conjunction with this method. The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The analyst should note that finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. It is recommended that such samples be processed in a confined environment, such as a hood or a glove box. Laboratory personnel handling these types of samples should wear masks fitted with charcoal filters to prevent inhalation of dust.

5.4 The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.5 Each laboratory must develop a strict safety program for the handling of PCDDs and PCDFs. The laboratory practices listed below are recommended.

5.5.1 Contamination of the laboratory will be minimized by conducting most of the manipulations in a hood, or in a separate containment facility away from the main laboratory.

5.5.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high boiling alcohols.

5.5.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light at a wavelength less than 290 nm for several days (use F 40 BL lamps, or equivalent). Using this analytical method, analyze the irradiated liquid wastes and dispose of the solutions when 2,3,7,8-TCDD and 2,3,7,8-TCDF congeners can no longer be detected.

5.6 The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory were issued by Dow Chemical U.S.A. (revised 11/78) and were amended for use in conjunction with this method. The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. The 2,3,7,8-TCDD isomer is extremely toxic to certain kinds of laboratory animals. However, it has been handled for years without injury in analytical and biological laboratories. Many techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

5.6.1 Protective equipment -- Disposable plastic gloves, apron or lab coat, safety glasses and laboratory hood adequate for radioactive work. However, PVC gloves should not be used.

5.6.2 Training -- Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.6.3 Personal hygiene -- Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

5.6.4 Confinement -- Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

5.6.5 Waste -- Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.6.6 Disposal of hazardous wastes -- Refer to the November 7, 1986 issue of the Federal Register on Land Ban Rulings for details concerning the handling of dioxin-containing wastes.

5.6.7 Decontamination of personnel -- Apply a mild soap with plenty of scrubbing action.

5.6.8 Glassware, tools and surfaces -- Chlorothene NU Solvent™ (Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished by rinsing with chlorothene, then washing with a detergent and water. Dishwater may be disposed to the sewer after percolation through a charcoal bed filter. It is prudent to minimize solvent wastes because they require costly special disposal through commercial services.

5.6.9 Laundry -- Clothing known to be contaminated should be disposed according to the precautions of the source described under Sec. 5.6.6, "Disposal of hazardous wastes." Laboratory coats or other clothing worn in 2,3,7,8-TCDD work area may be laundered. Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through one full cycle before being used again for other clothing.

5.6.10 Wipe tests -- A useful method for determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract.

NOTE: A procedure for the collection, handling, analysis, and reporting requirements of wipe tests performed within the laboratory is described in Appendix A of this method. The results and decision-making processes are based on the presence of 2,3,7,8-substituted PCDDs/PCDFs.

5.6.11 Inhalation -- Any procedure that may generate airborne contamination must be carried out with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no significant inhalation hazards except in case of an accident.

5.6.12 Accidents -- Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.7 It is recommended that personnel working in laboratories where PCDDs/PCDFs are handled be given periodic physical examinations (at least annually). Such examinations should include specialized tests, such as those for urinary porphyrins and for certain blood parameters which, based upon published clinical observations, are appropriate for persons who may be exposed to PCDDs/PCDFs. Periodic facial photographs to document the onset of dermatologic problems are also advisable.

## 6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 High-resolution gas chromatograph/high-resolution mass spectrometer/data system (HRGC/HRMS/DS) -- The GC must be equipped for temperature programming, and all required accessories must be available, such as syringes, gases, and capillary columns.

6.1.1 GC injection port -- The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On-column 1- $\mu$ L injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2- $\mu$ L injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2  $\mu$ L). Also, 1- $\mu$ L injections may be used; however, laboratories must remain consistent throughout the analyses by using the same injection volume at all times.

6.1.2 GC/MS interface -- The GC/MS interface components should withstand 350 °C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

6.1.3 Mass spectrometer -- The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

6.1.4 Data system -- A dedicated data system is employed to control the rapid multiple-ion monitoring process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitations may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data at a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass spectral peak profiles (Sec. 9.3.2.3 and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should permit the measurement of noise on the base line.

NOTE: The detector ADC zero setting must allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power. The effect of different zero settings on the measured resolving power is shown in Figure 2.

## 6.2 GC columns

Fused-silica capillary columns are needed. The columns must demonstrate the required separation of all 2,3,7,8-specific isomers whether a dual-column or a single-column analysis is chosen. Chromatographic performance must be demonstrated and documented (Sec. 9.3.1) at the beginning of each 12-hr period (after mass resolution and GC resolution are demonstrated) during which sample extracts or concentration calibration solutions will be analyzed. Recommended operating conditions for the recommended columns are shown in Sec. 11.6.

The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that are available or that may be developed. Laboratories may use these columns or other columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.2.1 60-m DB-5 (J&W Scientific) or equivalent fused-silica capillary column

In order to have an isomer-specific determination of 2,3,7,8-TCDD and to allow the quantitation of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, use of this column is recommended. Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60-m DB-5 column. Problems have been associated with the separation of 2,3,7,8-TCDD from 1,2,3,7-TCDD and 1,2,6,8-TCDD, and with the separation of 2,3,7,8-TCDF from 1,2,4,9-, 1,2,7,9-, 2,3,4,6-, 2,3,4,7-, and 2,3,4,8-TCDF. Because of the toxicologic concern associated with 2,3,7,8-TCDD and 2,3,7,8-TCDF, additional analyses may be necessary for some samples.

6.2.2 30-m DB-225 (J&W Scientific) or equivalent fused-silica capillary column

For the DB-225 column, problems are associated with the separation of 2,3,7,8-TCDF from 2,3,4,7-TCDF and a combination of 1,2,3,9- and 2,3,4,8-TCDF.

6.3 Miscellaneous equipment and materials -- The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.3.1 Nitrogen evaporation apparatus with variable flow rate.

6.3.2 Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3.3 Centrifuge.

6.3.4 Water bath -- Equipped with concentric ring covers and capable of being temperature controlled within  $\pm 2$  °C.

6.3.5 Stainless steel or glass container large enough to hold contents of one-pint sample containers.

6.3.6 Glove box.

6.3.7 Drying oven.

6.3.8 Stainless steel spoons and spatulas.

6.3.9 Laboratory hoods.

6.3.10 Pipets -- Disposable, Pasteur, 150 mm long x 5 mm ID.

6.3.11 Pipets -- Disposable, serological, 10-mL, for the preparation of the carbon columns described in Sec. 11.5.3.

6.3.12 Reaction vial -- 2-mL, silanized amber glass (Reacti-vial, or equivalent).

6.3.13 Stainless steel meat grinder with a 3 to 5 mm hole size inner plate.

6.3.14 Separatory funnels -- 125-mL and 2000-mL.

6.3.15 Kuderna-Danish concentrator -- 500-mL, fitted with 10-mL concentrator tube and three-ball Snyder column. Other evaporation devices such as a Turbovap and Nevap may be used as appropriate for a given application. Users should consult the manufacturers guidelines for operational requirements and instructions.

**NOTE:** The use of a solvent vapor recovery system (Kontes K-545000-1006 or K-547300-0000, Ace Glass 6614-30, or equivalent) is recommended for use in methods that use Kuderna-Danish or other evaporative concentrators. Incorporation of this apparatus may be required by Federal, State, or local municipality regulations that govern air emissions of volatile organics. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program. Solvent recovery is a means to conform with waste minimization and pollution prevention initiatives.

6.3.16 PTFE or Carborundum™ (silicon carbide) boiling chips (or equivalent), washed with hexane before use.

**CAUTION:** PTFE boiling chips may float in methylene chloride, may not work in the presence of any water phase, and may be penetrated by nonpolar organic compounds.

6.3.17 Chromatographic columns -- Glass, 300 mm x 10.5 mm, fitted with PTFE stopcock.

6.3.18 Adapters for concentrator tubes.

6.3.19 Glass fiber filters -- 0.70- $\mu$ m, Whatman GFF, or equivalent.

6.3.20 Dean-Stark trap -- 5- or 10-mL, with T-joints, condenser and 125-mL flask.

6.3.21 Continuous liquid-liquid extractor -- 1-L sample capacity, suitable for use with heavier than water solvents.

6.3.22 All glass Soxhlet apparatus with 500-mL flask.

6.3.23 Soxhlet/Dean-Stark extractor (optional) -- All glass, 500-mL flask.

6.3.24 Glass funnels -- Sized to hold 170 mL of liquid.

6.3.25 Desiccator.

6.3.26 Solvent reservoir (125-mL) -- Compatible with gravity carbon column.

6.3.27 Rotary evaporator with a temperature-controlled water bath.

6.3.28 High speed tissue homogenizer -- Equipped with an EN-8 probe, or equivalent.

6.3.29 Glass wool -- Extract with methylene chloride, dry, and store in a glass jar.

6.3.30 Extraction jars -- Glass, 250-mL, equipped with PTFE-lined screw caps.

6.3.31 Volumetric flasks -- Class A, 10-mL to 1000-mL.

6.3.32 Glass vials -- 1-dram (or metric equivalent).

6.3.33 See Methods 3535, 3545, and 3546 for the supplies specific to their particular preparation procedures.

6.4 Laboratory glassware cleaning procedures -- Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure.

6.4.1 Rinse glassware with the last solvent used in it.

6.4.2 Wash with hot water containing detergent.

6.4.3 Rinse with copious amounts of tap water and several portions of organic-free reagent water. Drain dry.

6.4.4 Rinse with pesticide-grade acetone and hexane.

6.4.5 After glassware is dry, store inverted or capped with aluminum foil in a clean environment.

6.4.6 Do not bake reusable glassware as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but it should be minimized, because repeated baking may cause active sites on the glass surface that will irreversibly adsorb PCDDs/PCDFs.

**CAUTION:** The analysis for PCDDs/PCDFs in water samples is for much lower concentrations than in soil/sediment, fly ash, or chemical waste samples. Extreme care must be taken to prevent cross-contamination between soil/sediment, fly ash, chemical waste and water samples. Therefore, it is strongly recommended that separate glassware be reserved for analyzing water samples.

6.5 Pre-extraction of glassware -- All glassware should be rinsed or pre-extracted with solvent immediately before use. Soxhlet-Dean-Stark (SDS) apparatus and continuous liquid-liquid extractors should be pre-extracted for approximately 3 hr immediately prior to use, using the same solvent and extraction conditions that will be employed for sample extractions. The pooled waste solvent for a set of extractions may be concentrated and analyzed as a method of demonstrating that the glassware was free of contamination.

It is recommended that each piece of reusable glassware be numbered in such a fashion that the laboratory can associate all reusable glassware with the processing of a particular sample. This will assist the laboratory in:

- (1) Tracking down possible sources of contamination for individual samples,
- (2) Identifying glassware associated with highly contaminated samples that may require extra cleaning, and
- (3) Determining when glassware should be discarded.

## 7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

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

### 7.3 Column chromatography reagents

7.3.1 Alumina -- neutral, 80/200 mesh (Super 1, Woelm®, or equivalent). Store in a sealed container at room temperature, in a desiccator, over self-indicating silica gel.

7.3.2 Alumina -- acidic AG4 (Bio Rad Laboratories catalog #132-1240, or equivalent). Soxhlet extract with methylene chloride for 24 hr if blanks show contamination, and activate by heating in a foil-covered glass container for 24 hr at 190 °C. Store in a glass bottle sealed equipped with a PTFE-lined screw cap.

7.3.3 Silica gel -- high-purity grade, type 60, 70-230 mesh. Soxhlet extract with methylene chloride for 24 hr if blanks show contamination, and activate by heating in a foil covered glass container for 24 hr at 190 °C. Store in a glass bottle sealed equipped with a PTFE-lined screw cap.

7.3.4 Silica gel impregnated with sodium hydroxide. Add one part (by weight) of 1 M NaOH solution to two parts (by weight) silica gel (extracted and activated) in a screw cap bottle and mix with a glass rod until free of lumps. Store in a glass bottle sealed with a PTFE-lined screw cap.

7.3.5 Silica gel impregnated with 40 percent (by weight) sulfuric acid. Add two parts (by weight) concentrated sulfuric acid to three parts (by weight) silica gel (extracted and activated), mix with a glass rod until free of lumps, and store in a screw capped glass bottle. Store in a glass bottle sealed with a PTFE-lined screw cap.

7.3.6 Celite 545® (Supelco), or equivalent.

7.3.7 Charcoal carbon -- Activated carbon, Carbopak C (Supelco) or equivalent, prewashed with methanol and dried *in vacuo* at 110 °C. Store in a glass bottle sealed with a PTFE-lined screw cap. (Note: AX-21 [Anderson Development Company] carbon is no longer available, but existing stocks may be utilized.)

### 7.4 Reagents

7.4.1 Sulfuric acid, H<sub>2</sub>SO<sub>4</sub>, concentrated, ACS grade, specific gravity 1.84.

7.4.2 Potassium hydroxide, KOH, ACS grade, 20 percent (w/v) in organic-free reagent water.

7.4.3 Sodium chloride, NaCl, analytical reagent, 5 percent (w/v) in organic-free reagent water.

7.4.4 Potassium carbonate,  $K_2CO_3$ , anhydrous, analytical reagent.

7.5 Sodium sulfate (powder, anhydrous),  $Na_2SO_4$  -- Purify by heating at 400 °C for 4 hr in a shallow tray. If, after heating, the sodium sulfate develops a noticeable grayish cast (due to the presence of carbon in the crystal matrix), that batch of sodium sulfate is not suitable for use and should be discarded. Extraction with methylene chloride may produce sodium sulfate that is suitable for use in such instances, but following extraction, a reagent blank must be analyzed to demonstrate that there is no interference from the sodium sulfate.

7.6 Solvents -- All solvents should be (at a minimum) pesticide quality or equivalent, distilled-in-glass. Solvents may be degassed prior to use.

Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentrations of interest. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the desired project-specific concentration levels. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations.

7.6.1 Methylene chloride,  $CH_2Cl_2$ .

7.6.2 Hexane,  $C_6H_{14}$ .

7.6.3 Methanol,  $CH_3OH$ .

7.6.4 Nonane,  $C_9H_{20}$ .

7.6.5 Toluene,  $C_6H_5CH_3$ .

7.6.6 Cyclohexane,  $C_6H_{12}$ .

7.6.7 Acetone,  $CH_3COCH_3$ .

7.7 High-resolution concentration calibration (HRCC) solutions (Table 5) -- Five nonane solutions containing 17 unlabeled and 11 carbon-labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homologue-dependent, with the lowest values for the tetrachlorinated dioxin and furan (1.0 pg/ $\mu$ L) and the highest values for the octachlorinated congeners (1000 pg/ $\mu$ L). Standards containing more carbon-labeled PCDDs and PCDFs may also be employed. All standards should be stored away from any light source at  $\leq 6$  °C when not in use (-10 °C is recommended), and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification (GC column performance check) standard should be prepared, as necessary, and stored at  $\leq 6$  °C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.8 GC column performance check (evaluation) solution -- This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The  $^{13}C_{12}$ -2,3,7,8-TCDD is also present. The laboratory is required to use nonane as the solvent and adjust the volume so that the final

concentration does not exceed 100 pg/μL per congener. Table 7 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution.

7.9 Sample fortification solution -- This nonane solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that  $^{13}\text{C}_{12}$ -OCDF is not present in the solution.) Standards containing more carbon-labeled PCDDs and PCDFs may also be employed, provided that the same labeled compounds are contained in the calibration standards in Sec. 7.7.

7.10 Recovery standard solution -- This nonane solution contains two recovery standards,  $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and  $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD, at a nominal concentration of 50 pg/μL per compound. 10 to 50 μL (depending on the matrix) of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

### 8.1 Sample collection

8.1.1 Sample collection personnel should, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly not homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.1.2 Grab and composite samples must be collected in glass containers. Conventional sampling practices must be followed. The bottle must not be prewashed with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.2 Grinding or blending of fish samples -- If not otherwise specified in a project plan, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested. If so requested, the above whole fish requirement is superseded.

8.3 Recommended sample storage and holding times -- All samples should be stored at  $\leq 6$  °C, or lower, in the dark, extracted within 30 days and completely analyzed within 45 days of extraction. Fish and adipose tissue samples should be stored at  $< -10$  °C in the dark, and should be extracted within 30 days and completely analyzed within 45 days of collection.

8.4 Unused portions of samples and sample extracts should be stored at  $\leq 6$  °C in the dark for six months after sample receipt to allow further analyses if necessary.

**NOTE:** The holding times listed in this method are recommendations. PCDDs and PCDFs are very stable in a variety of matrices, and holding times under the conditions listed in this section may be as long as a year for certain matrices.

## 8.5 Phase separation

This is a guideline for phase separation for very wet (>25 percent water) soil, sediment and paper pulp samples. Place a 50-g portion in a suitable centrifuge bottle and centrifuge for 30 min at 2,000 rpm. Remove the bottle and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipet, transfer the liquid layer into a clean bottle. Mix the solid with a stainless steel spatula and remove a portion to be weighed and analyzed (percent dry weight determination, extraction). Return the remaining solid portion to the original sample bottle (empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil, sediment and paper pulp method. Take note of, and report, the estimated volume of liquid before disposing of the liquid as a liquid waste.

## 8.6 Soil, sediment, or paper sludge (pulp) percent dry weight determination

When results are to be reported on a dry-weight basis, the percent dry weight of soil, sediment or paper pulp samples may be determined according to the following procedure. Weigh a 10-g portion of the soil or sediment sample ( $\pm 0.5$  g) to three significant figures. Dry it to constant weight at 110 °C in an adequately ventilated oven. Allow the sample to cool in a desiccator. Weigh the dried solid to three significant figures. Calculate and report the percent dry weight. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

**WARNING:** Finely divided soils and sediments contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing PCDDs/PCDFs (including 2,3,7,8-TCDD). Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

## 8.7 Lipid content determination

8.7.1 Fish tissue -- To determine the lipid content of fish tissue, concentrate 125 mL of the fish tissue extract (Sec. 11.2.2), in a tared 200-mL round-bottom flask, on a rotary evaporator until a constant weight is achieved.

$$\% \text{ lipid} = \frac{\text{weight of residue} \times 2}{\text{weight of sample}} \times 100$$

The factor of 2 accounts for the use of half of the extract (e.g., 125 mL of 250 mL total volume) for the lipid determination.

Dispose of the lipid residue as a hazardous waste if the results of the analysis indicate the presence of PCDDs or PCDFs.

Other procedures and other extract volumes may be employed for the lipid determination, provided that they are clearly described and documented. Adjustments to the amount of internal standards spiked in Sec. 11.1 will be required if different volumes are employed.

8.7.2 Adipose tissue -- Details for the determination of the adipose tissue lipid content are provided in Sec. 11.3.3.

8.8 Also see the introductory material to Chapter Four, "Organic Analytes," for general information related to sample collection, preservation, and storage.

## 9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 for QC procedures to validate sample extraction and ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 8000, 3500, or 3600.

9.3 System performance criteria -- System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 6.2. The laboratory must document that all applicable system performance criteria (described in Sec. 9.3) were met before it analyses any sample. Sec. 11.6.1 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 3 provides a typical 12-hr analysis sequence, whereby the response factors and mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is only required at the beginning of each 12-hr period during which samples are analyzed. An HRGC/HRMS method blank run is required between a calibration run and the first sample run. The same method blank extract may thus be analyzed more than once if the number of samples within a batch requires more than 12 hr of analyses.

### 9.3.1 GC column performance

9.3.1.1 Inject 2  $\mu\text{L}$  of an aliquot (Sec. 6.1.1) of the column performance check solution (Sec. 7.8) and acquire selected ion monitoring (SIM) data as described in Sec. 11.6.2 within a total cycle time of  $\leq 1$  sec (Sec. 11.6.3.1).

9.3.1.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of  $\leq 25$  percent (Figure 4), where:

$$\text{Valley percent} = (x/y) \times (100)$$

x = measured as in Figure 4 from the 2,3,7,8-closest TCDD eluting isomer

y = the peak height of 2,3,7,8-TCDD

It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions described in this protocol. Their retention times are used to determine the five homologue retention time windows that are used for qualitative (Sec. 11.8.4.1) and quantitative purposes. All peaks (that includes  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD) should be labeled and identified on the chromatograms. Furthermore, all first eluters of a homologous series should be labeled with the letter "F," and all last eluters of a homologous series should be labeled with the letter "L" (Figure 4 shows an example of peak labeling for TCDD isomers). Any individual selected ion current profile (SICP) (for the tetras, this would be the SICP for m/z 322 and m/z 304) or the reconstructed homologue ion current (for the tetras, this would correspond to m/z 320 + m/z 322 + m/z 304 + m/z 306) constitutes an acceptable form of data presentation. An SICP for the labeled compounds (e.g., m/z 334 for labeled TCDD) is also required.

9.3.1.3 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds. Allowable tolerance on the daily verification with the GC performance check solution should be better than 10 sec for the absolute retention times of all the components of the mixture. Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (i.e., 1,2,8,9-TCDD) and the first pentachlorinated congener (i.e., 1,3,4,6,8-PeCDF), as these two compounds elute within 15 sec of each other on the 60-m DB-5 column. A laboratory with a GC/MS system that is not capable of detecting both congeners (1,2,8,9-TCDD and 1,3,4,6,8-PeCDF) within one analysis must take corrective action. If the recommended column is not used, then the first- and last-eluting isomer of each homologue must be determined experimentally on the column which is used, and the appropriate isomers must then be used for window definition and switching times.

### 9.3.2 Mass spectrometer performance

9.3.2.1 The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed (Sec. 11.8). Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. However, it is recommended that a check of the static resolution be made and documented before and after each analysis. Corrective action must be implemented whenever the resolving power does not meet the requirement.

9.3.2.2 Chromatography time for PCDDs and PCDFs exceeds the long term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a

mass drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

**NOTE:** Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

9.3.2.3 Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal ( $m/z$  380.9760) obtained during the above peak matching experiment by using the low-mass PFK ion at  $m/z$  304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 5) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at  $m/z$  380.9760 (or 0.038 amu at that particular mass).

#### 9.4 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis.

#### 9.6 Sample quality control for preparation and analysis

9.6.1 Performance evaluation samples -- When available, performance evaluation (PE) samples containing known amounts of unlabeled 2,3,7,8-substituted

PCDDs/PCDFs or other PCDD/PCDF congeners should be analyzed alongside routine field samples.

#### 9.6.2 Performance check solutions

9.6.2.1 At the beginning of each 12-hr period during which samples are to be analyzed, an aliquot of the (1) GC column performance check solution and (2) high-resolution concentration calibration solution No. 3 (HRCC-3; see Table 5) must be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. A mass resolution check must also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. Alternatively, these check sample analyses may be combined into one run using a commercially prepared standard.

9.6.2.2 To validate positive sample data, the routine or continuing calibration (HRCC-3; Table 5) and the mass resolution check must be performed also at the end of each 12-hr period during which samples are analyzed. Furthermore, an HRGC/HRMS method blank run should be recorded following a calibration run and prior to the first sample run.

9.6.2.2.1 If the laboratory operates only during one period (shift) each day of 12 hr or less, the GC performance check solution must be analyzed only once (at the beginning of the period) to validate the data acquired during the period. However, the mass resolution and continuing calibration checks must be performed at the beginning as well as at the end of the period.

9.6.2.2.2 If the laboratory operates during consecutive 12-hr periods (shifts), analysis of the GC performance check solution must be performed at the beginning of each 12-hr period. The mass resolution and continuing calibration checks from the previous period can be used for the beginning of the next period.

9.6.2.3 Results of at least one analysis of the GC column performance check solution and of two mass resolution and continuing calibration checks must be reported with the sample data collected during a 12-hr period.

9.6.2.4 Deviations from criteria specified for the GC performance check or for the mass resolution check invalidate all positive sample data collected between analyses of the performance check solution, and the extracts from those positive samples must be reanalyzed.

If the routine calibration run fails at the beginning of a 12-hr shift, the instructions in Sec. 11.7.4.4 must be followed. If the continuing calibration check performed at the end of a 12 hr period fails by no more than 25 percent RPD for the 17 unlabeled compounds and 35 percent RPD for the 9 labeled reference compounds, use the mean to the two "daily" RF values from the two daily routine calibration runs to compute the analyte concentrations, instead of the  $\bar{R}\bar{F}$  values obtained from the initial calibration. A new initial calibration (new  $\bar{R}\bar{F}$ s) is required immediately (within 2 hr) following the analysis of the samples, whenever the RPD from the end-of-shift routine calibration exceeds 25 percent or 35 percent,

respectively. Failure to perform a new initial calibration immediately following the analysis of the samples will automatically require reanalysis of all positive sample extracts analyzed before the failed end-of-shift continuing calibration check.

9.6.3 The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from commercial sources.

9.6.4 Field blanks -- Batches of field samples may contain a field blank sample of uncontaminated soil, sediment or water that is to be fortified before analysis according to Sec. 9.6.4.1. In addition to this field blank, a batch of samples may include a rinsate, which is a portion of the solvent that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

#### 9.6.4.1 Fortified field blank (OPTIONAL)

9.6.4.1.1 Weigh a 10-g portion or use 1-L (for aqueous samples) of the designated field blank sample and add 100  $\mu$ L of the solution containing the nine internal standards (Table 2) diluted with 1.0 mL acetone (Sec. 11.1).

9.6.4.1.2 Extract by using the procedures beginning in Secs. 11.4.5 or 11.4.6, as applicable, add 10  $\mu$ L of the recovery standard solution (Sec. 7.10) and analyze a 2- $\mu$ L aliquot of the concentrated extract.

9.6.4.1.3 Calculate the concentration (Sec. 11.9.1) of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards (Sec. 11.9.2).

#### 9.6.4.2 Rinsate sample (OPTIONAL)

9.6.4.2.1 Take a 100-mL ( $\pm$  0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add 100  $\mu$ L of the solution containing the nine internal standards (Table 2).

9.6.4.2.2 Using a K-D apparatus, concentrate to about 5 mL.

**NOTE:** As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the rinsate.

9.6.4.2.3 Transfer the 5 mL concentrate from the K-D concentrator tube in 1-mL portions to a 1-mL minivial, reducing the volume in the minivial as necessary with a gentle stream of dry nitrogen.

9.6.4.2.4 Rinse the K-D concentrator tube with two 0.5 mL portions of hexane and transfer the rinses to the 1 mL minivial. Concentrate with dry nitrogen, as necessary.

9.6.4.2.5 Just before analysis, add 10  $\mu$ L of the recovery standard solution (Table 2) and reduce the volume to its final volume, as necessary (Sec. 11.8.1). No column chromatography is required.

9.6.4.2.6 Analyze an aliquot of the solution following the same procedures used to analyze samples.

9.6.4.2.7 Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in µg/L of rinsate solvent.

#### 9.6.5 Duplicate analyses (OPTIONAL)

In each batch of samples, locate the sample designated for duplicate analysis (the sample may be labeled "double volume"), and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration.

9.6.5.1 The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference (difference expressed as percentage of the mean). Report all results.

#### 9.6.5.2 Recommended actions to help locate problems

- (1) Verify satisfactory instrument performance (Secs. 9.3 and 9.6).
- (2) If possible, verify that no error was made while weighing the sample portions.
- (3) Review the analytical procedures with the performing laboratory personnel.

9.7 Percent recovery of the internal standards -- For each sample, method blank and rinsate, calculate the percent recovery (Sec. 11.9.2). The percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards.

NOTE: A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data.

#### 9.8 Identification criteria

9.8.1 If either one of the identification criteria appearing in Secs. 11.8.4.1.1 through 11.8.4.1.4 is not met for an homologous series, it is reported that the sample does not contain unlabeled 2,3,7,8-substituted PCDD/PCDF isomers for that homologous series at the calculated quantitation limit (Sec. 11.9.5)

9.8.2 If the first initial identification criteria (Secs. 11.8.4.1.1 through 11.8.4.1.4) are met, but the criteria appearing in Secs. 11.8.4.1.5 and 11.8.4.2 are not met, that sample is presumed to contain interfering contaminants. This must be noted on the analytical report form, and the sample should be rerun or the extract reanalyzed.

9.9 The analysis of method blanks is critical to the provision of meaningful sample results.

9.9.1 Method blanks should be prepared at a frequency of at least 5%, that is, one method blank for each group of up to 20 samples prepared at the same time, by the same procedures.

9.9.2 When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

9.9.3 If the method blank results do not meet the project-specific acceptance criteria, then the laboratory should take corrective action to locate and reduce the source of the contamination and to re-extract and reanalyze any samples associated with the contaminated method blank.

9.9.4 The laboratory should not subtract the results of the method blank from those of any associated samples. Such "blank subtraction" is inappropriate and often leads to negative sample results. If the method blank results do not meet the acceptance criteria in 9.9.3 and reanalysis is not practical, then the data user should be provided with the sample results, the method blank results, and a discussion of the corrective actions undertaken by the laboratory.

9.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 10.0 CALIBRATION AND STANDARDIZATION

See Sec 11.7 for information on calibration and standardization.

## 11.0 PROCEDURE

### 11.1 Internal standard addition

The sample fortification solution (Sec. 7.9) containing the carbon-labeled internal standards is added to each sample prior to extraction.

11.1.1 Select an appropriately sized sample aliquot. Typical sample size requirements for different matrices are given in Sec. 11.4 and in Table 1. Transfer the sample portion to a tared flask and determine its weight.

11.1.2 Except for adipose tissue, add an appropriate quantity of the sample fortification mixture (Sec. 7.9) to the sample. All samples should be spiked with 100  $\mu\text{L}$  of the sample fortification mixture to give internal standard concentrations as indicated in Table 1. As an example, for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 1000 pg of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD to give the required 100 ppt fortification level. The fish tissue sample (20 g) must be spiked with 200  $\mu\text{L}$  of the internal standard solution, because half of the extract will be used to determine the lipid content (Sec. 8.7.1).

11.1.2.1 For the fortification of soil, sediment, fly ash, water, fish tissue, paper pulp and wet sludge samples, mix the sample fortification solution with 1.0 mL acetone.

11.1.2.2 Do not dilute the nonane solution for the other matrices.

11.1.2.3 The fortification of adipose tissue is carried out at the time of homogenization (Sec. 11.3.2.3).

## 11.2 Extraction and purification of fish and paper pulp samples

11.2.1 Add 60 g of anhydrous sodium sulfate to a 20-g portion of a homogeneous fish sample (Sec. 8.2) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the fish/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug. Add 250 mL of methylene chloride or hexane/methylene chloride (1:1) to the Soxhlet apparatus and reflux for 16 hr. The solvent must cycle completely through the system five to ten times per hour. Follow the same procedure for the partially dewatered paper pulp sample (using a 10-g sample, 30 g of anhydrous sodium sulfate and 200 mL of toluene).

**NOTE:** As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.2.2 Transfer the fish extract from Sec. 11.2.1 to a 250-mL volumetric flask and fill to the mark with methylene chloride. Mix well, then remove 125 mL for the determination of the lipid content (Sec. 8.7.1). Transfer the remaining 125 mL of the extract, plus two 15-mL hexane/methylene chloride rinses of the volumetric flask, to a K-D apparatus equipped with a Snyder column. Quantitatively transfer all of the paper pulp extract to a K-D apparatus equipped with a Snyder column.

**NOTE:** As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the extracts.

11.2.3 Add a PTFE (or equivalent) boiling chip. Concentrate the extract in a water bath to an apparent volume of 10 mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

11.2.4 Add 50 mL of hexane and a new boiling chip to the K-D flask. Concentrate in a water bath to an apparent volume of 5 mL. Remove the apparatus from the water bath and allow to cool for 5 minutes.

**NOTE:** The methylene chloride must have been completely removed before proceeding with the next step.

11.2.5 Remove and invert the Snyder column and rinse it into the K-D apparatus with two 1-mL portions of hexane. Decant the contents of the K-D apparatus and concentrator tube into a 125-mL separatory funnel. Rinse the K-D apparatus with two additional 5-mL portions of hexane and add the rinses to the funnel. Proceed with the appropriate cleanup according to the instructions in Sec. 11.5.

## 11.3 Extraction and purification of human adipose tissue

11.3.1 Human adipose tissue samples must be stored at a temperature of  $-20^{\circ}\text{C}$  or lower from the time of collection until the time of analysis. The use of chlorinated materials during the collection of the samples must be avoided. Samples are handled with stainless steel forceps, spatulas, or scissors. All sample bottles (glass) are cleaned as indicated in Sec. 6.4. PTFE-lined caps should be used.

**NOTE:** The storage temperature of  $-20^{\circ}\text{C}$  in Sec. 11.3.1 is the maximum storage temperature permissible for adipose tissue samples. Lower storage temperatures are recommended.

### 11.3.2 Adipose tissue extraction

11.3.2.1 Weigh a 10-g portion of a frozen adipose tissue sample to the nearest 0.01 g, into a culture tube (2.2 x 15 cm).

**NOTE:** The sample size may be smaller, depending on availability. In such situations, the analyst is required to adjust the volume of the internal standard solution added to the sample to meet the fortification level listed in Table 2.

11.3.2.2 Allow the adipose tissue specimen to reach room temperature (up to 2 hr).

11.3.2.3 Add 10 mL of methylene chloride and 100  $\mu$ L of the sample fortification solution. (Sec. 7.9) Homogenize the mixture for approximately 1 minute with a tissue homogenizer.

11.3.2.4 Allow the mixture to separate, then remove the methylene chloride extract from the residual solid material with a disposable pipet. Percolate the methylene chloride through a filter funnel containing a clean glass wool plug and 10 g of anhydrous sodium sulfate. Collect the dried extract in a graduated 100-mL volumetric flask.

11.3.2.5 Add a second 10 mL portion of methylene chloride to the sample and homogenize for 1 min. Decant the solvent, dry it, and transfer it to the 100-mL volumetric flask (Sec. 11.3.2.4).

11.3.2.6 Rinse the culture tube with at least two additional portions of methylene chloride (10-mL each), and transfer the entire contents to the filter funnel containing the anhydrous sodium sulfate. Rinse the filter funnel and the anhydrous sodium sulfate contents with additional methylene chloride (20 to 40 mL) into the 100-mL flask. Discard the sodium sulfate.

11.3.2.7 Adjust the volume to the 100-mL mark with methylene chloride.

### 11.3.3 Adipose tissue lipid content determination

11.3.3.1 Preweigh a clean 1-dram (or metric equivalent) glass vial to the nearest 0.0001 g on an analytical balance tared to zero.

11.3.3.2 Accurately transfer 1.0 mL of the final extract (100 mL) from Sec. 11.3.2.7 to the vial. Reduce the volume of the extract on a water bath (50-60  $^{\circ}$ C) by a gentle stream of purified nitrogen until an oily residue remains. Nitrogen evaporation is continued until a constant weight is achieved.

**NOTE:** When the sample size of the adipose tissue is smaller than 10 g, then the analyst may use a larger portion (up to 10 percent) of the extract defined in Sec. 11.3.2.7 for the lipid determination.

11.3.3.3 Accurately weigh the vial with the residue to the nearest 0.0001 g and calculate the weight of the lipid present in the vial based on the difference of the weights.

11.3.3.4 Calculate the percent lipid content of the original sample to the nearest 0.1 percent as shown below:

$$\% \text{ Lipid} = \frac{W_{\text{lr}} \times V_{\text{ext}}}{W_{\text{at}} \times V_{\text{al}}} \times 100$$

where:

$W_{\text{lr}}$  = weight of the lipid residue to the nearest 0.0001 g calculated from Sec. 11.3.3.3,

$V_{\text{ext}}$  = total volume (100 mL) of the extract in mL from Sec. 11.3.2.7,

$W_{\text{at}}$  = weight of the original adipose tissue sample to the nearest 0.01 g from Sec. 11.3.2.1, and

$V_{\text{al}}$  = volume of the aliquot of the final extract in mL used for the quantitative measure of the lipid residue (1.0 mL) from Sec. 11.3.3.2.

11.3.3.5 Record the weight of the lipid residue measured in Sec. 11.3.3.3 and the percent lipid content from Sec. 11.3.3.4.

#### 11.3.4 Adipose tissue extract concentration

11.3.4.1 Quantitatively transfer the remaining extract from Sec. 11.3.3.2 (99.0 mL) to a 500-mL Erlenmeyer flask. Rinse the volumetric flask with 20 to 30 mL of additional methylene chloride to ensure quantitative transfer.

11.3.4.2 Concentrate the extract on a rotary evaporator and a water bath at 40 °C until an oily residue remains.

#### 11.3.5 Adipose tissue extract cleanup

11.3.5.1 Add 200 mL of hexane to the lipid residue in the 500-mL Erlenmeyer flask and swirl the flask to dissolve the residue.

11.3.5.2 Slowly add, with stirring, 100 g of 40 percent (w/w) sulfuric acid-impregnated silica gel. Stir with a magnetic stirrer for 2 hr at room temperature.

11.3.5.3 Allow the solid phase to settle, and decant the liquid through a filter funnel containing 10 g of anhydrous sodium sulfate on a glass wool plug, into another 500-mL Erlenmeyer flask.

11.3.5.4 Rinse the solid phase with two 50-mL portions of hexane. Stir each rinse for 15 minutes, decant, and dry as described under Sec. 11.3.5.3. Combine the hexane extracts from Sec. 11.3.5.3 with the rinses.

11.3.5.5 Rinse the sodium sulfate in the filter funnel with an additional 25 mL of hexane and combine this rinse with the hexane extracts from Sec. 11.3.5.4.

11.3.5.6 Prepare an acidic silica column as follows: Pack a 2 cm x 10 cm chromatographic column with a glass wool plug, add approximately 20 mL of hexane, add 1 g of silica gel and allow to settle, then add 4 g of 40 percent (w/w) sulfuric acid-impregnated silica gel and allow to settle. Elute the excess hexane from the column until the solvent level reaches the top of the chromatographic packing. Verify that the column does not have any air bubbles and channels.

11.3.5.7 Quantitatively transfer the hexane extract from the Erlenmeyer flask (Secs. 11.3.5.3 through 11.3.5.5) to the silica gel column reservoir. Allow the hexane extract to percolate through the column and collect the eluate in a 500-mL K-D apparatus.

11.3.5.8 Complete the elution by percolating 50 mL of hexane through the column into the K-D apparatus. Concentrate the eluate on a steam bath to about 5 mL. Use nitrogen evaporation to bring the final volume to about 100  $\mu$ L.

**CAUTION:** If the silica gel impregnated with 40 percent sulfuric acid is highly discolored throughout the length of the adsorbent bed, the cleaning procedure must be repeated beginning with Sec. 11.3.5.1.

11.3.5.9 The extract is ready for the column cleanups described in Secs. 11.5.2 and 11.5.3.

## 11.4 Extraction and purification of environmental and waste samples

### 11.4.1 Sludge/wet fuel oil

11.4.1.1 Extract aqueous sludge or wet fuel oil samples by refluxing a sample (e.g., 2 g) with 50 mL of toluene in a 125-mL flask fitted with a Dean-Stark water separator. Continue refluxing the sample until all the water is removed.

**NOTE:** If the sludge or fuel oil sample dissolves in toluene, treat it according to the instructions in Sec. 11.4.2 below. If the sludge sample originates from pulp (paper mills), treat it according to the instructions starting in Sec. 11.2, but without the addition of sodium sulfate.

11.4.1.2 Cool the sample, filter the toluene extract through a glass fiber filter, or equivalent, into a 100-mL round-bottom flask.

11.4.1.3 Rinse the filter with 10 mL of toluene and combine the extract with the rinse.

11.4.1.4 Concentrate the combined solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

### 11.4.2 Still bottom/oil

11.4.2.1 Extract still bottom or oil samples by mixing a sample portion (e.g., 1.0 g) with 10 mL of toluene in a small beaker and filtering the solution

through a glass fiber filter (or equivalent) into a 50-mL round-bottom flask. Rinse the beaker and filter with 10 mL of toluene.

11.4.2.2 Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

### 11.4.3 Fly ash

**CAUTION:** Because of the tendency of fly ash to "fly," all handling steps should be performed in a hood in order to minimize contamination.

11.4.3.1 Weigh about 10 g of fly ash to two decimal places and transfer to an extraction jar. Add 100 µL of the sample fortification solution (Sec. 7.9), diluted to 1 mL with acetone, to the sample. Add 150 mL of 1 M HCl to the fly ash sample. Seal the jar with the PTFE-lined screw cap and shake for 3 hr at room temperature.

11.4.3.2 Rinse a glass fiber filter with toluene, and filter the sample through the filter paper, placed in a Buchner funnel, into a 1-L flask. Wash the fly ash cake with approximately 500 mL of organic-free reagent water and dry the filter cake overnight at room temperature in a desiccator.

11.4.3.3 Add 10 g of anhydrous powdered sodium sulfate, mix thoroughly, let sit in a closed container for 1 hr, mix again, let sit for another hr, and mix again.

11.4.3.4 Place the sample and the filter paper into an extraction thimble, and extract in a Soxhlet extraction apparatus charged with 200 mL of toluene for 16 hr using a five cycle/hour schedule.

**NOTE:** As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4.3.5 Cool and filter the toluene extract through a glass fiber filter into a 500-mL round-bottom flask. Rinse the filter with 10 mL of toluene. Add the rinse to the extract and concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50 °C or using nitrogen evaporation. Proceed with Sec. 11.4.4.

11.4.3.6 Alternatively, fly ash samples may be extracted with a toluene/acetic acid mixture using pressurized fluid extraction (PFE), as described in Method 3545. When using PFE, the HCl pretreatment in Sec. 11.4.3.1 may be omitted.

11.4.4 Transfer the concentrate to a 125-mL separatory funnel using 15 mL of hexane. Rinse the flask with two 5-mL portions of hexane and add the rinses to the funnel. Shake the combined solutions in the separatory funnel for two minutes with 50 mL of 5 percent sodium chloride solution, discard the aqueous layer, and proceed with Sec. 11.5.

#### 11.4.5 Aqueous samples

11.4.5.1 Allow the sample to come to ambient temperature, then mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Add the required acetone diluted sample fortification solution (Sec. 7.9).

11.4.5.2 When the sample is judged to contain 1 percent or more solids, the sample must be filtered through a glass fiber filter that has been rinsed with toluene. If the suspended solids content is too great to filter through the 0.45- $\mu\text{m}$  filter, centrifuge the sample, decant, and then filter the aqueous phase.

NOTE: Paper mill effluent samples normally contain 0.02%-0.2% solids, and would not require filtration. However, for optimum analytical results, all paper mill effluent samples should be filtered, the isolated solids and filtrate extracted separately, and the extracts recombined.

11.4.5.3 Combine the solids from the centrifuge bottle(s) with the particulates on the filter and with the filter itself and proceed with the Soxhlet extraction as indicated in Secs. 11.4.6.1 through 11.4.6.4.

NOTE: Pressurized fluid extraction has *not* been evaluated for the extraction of the particulate fraction.

Remove and invert the Snyder column and rinse it down into the K-D apparatus with two 1-mL portions of hexane.

11.4.5.4 Pour the aqueous filtrate into a 2-L separatory funnel. Add 60 mL of methylene chloride to the sample bottle, seal and shake for 30 sec to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting.

11.4.5.5 Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation (e.g., glass stirring rod).

11.4.5.6 Collect the methylene chloride in a K-D apparatus (mounted with a 10-mL concentrator tube) by passing the sample extracts through a filter funnel packed with a glass wool plug and 5 g of anhydrous sodium sulfate.

NOTE: As an option, a rotary evaporator may be used in place of the K-D apparatus for the concentration of the extracts.

11.4.5.7 Repeat the extraction twice with fresh 60-mL portions of methylene chloride. After the third extraction, rinse the sodium sulfate with an additional 30 mL of methylene chloride to ensure quantitative transfer. Combine all extracts and the rinse in the K-D apparatus.

NOTE: A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel. Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 sec to rinse the inner surface.

Transfer the solvent to the extractor. Repeat the rinse of the sample bottle with an additional 50- to 100-mL portion of methylene chloride and add the rinse to the extractor. Add 200 to 500 mL of methylene chloride to the distilling flask, add sufficient organic-free reagent water (Sec. 7.2) to ensure proper operation, and extract for 24 hr. Allow to cool, then detach the distilling flask. Dry and concentrate the extract as described in Secs. 11.4.5.6 and 11.4.5.8 through 11.4.5.10. Proceed with Sec. 11.4.5.11.

11.4.5.8 Attach a Snyder column and concentrate the extract on a water bath until the apparent volume of the liquid is 5 mL. Remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.4.5.9 Remove the Snyder column, add 50 mL of hexane, add the concentrate obtained from the Soxhlet extraction of the suspended solids (Sec. 11.4.5.3), if applicable, re-attach the Snyder column, and concentrate to approximately 5 mL. Add a new boiling chip to the K-D apparatus before proceeding with the second concentration step.

11.4.5.10 Rinse the flask and the lower joint with two 5-mL portions of hexane and combine the rinses with the extract to give a final volume of about 15 mL.

11.4.5.11 Determine the original sample volume by filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL. Proceed with Sec. 11.5.

#### 11.4.6 Soil/sediment

11.4.6.1 Add 10 g of anhydrous powdered sodium sulfate to the sample aliquot (10 g or less) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass wool plug (the use of an extraction thimble is optional).

NOTE: As an option, a Soxhlet/Dean-Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4.6.2 Add 200 to 250 mL of toluene to the Soxhlet apparatus and reflux for 16 hr. The solvent must cycle completely through the system 5 times per hr.

NOTE: If the dried sample is not of free flowing consistency, more sodium sulfate must be added.

11.4.6.3 Cool and filter the extract through a glass fiber filter into a 500-mL round-bottom flask for evaporation of the toluene. Rinse the filter with 10 mL of toluene, and concentrate the combined fractions to near dryness on a rotary evaporator at 50 °C. Remove the flask from the water bath and allow to cool for 5 min.

11.4.6.4 Transfer the residue to a 125-mL separatory funnel, using 15 mL of hexane. Rinse the flask with two additional portions of hexane, and add the rinses to the funnel. Proceed with Sec. 11.5.

11.4.6.5 Alternatively, soil/sediment samples may be extracted with toluene using pressurized fluid extraction (PFE), as described in Method 3545 or by microwave extraction, as described in Method 3546.

## 11.5 Cleanup

### 11.5.1 Acid-base washing

11.5.1.1 Partition the hexane extract against 40 mL of concentrated sulfuric acid. Shake for 2 min. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

11.5.1.2 Omit this step for the fish sample extract. Partition the extract against 40 mL of 5 percent (w/v) aqueous sodium chloride. Shake for 2 min. Remove and discard the aqueous layer (bottom).

11.5.1.3 Omit this step for the fish sample extract. Partition the extract against 40 mL of 20 percent (w/v) aqueous potassium hydroxide (KOH). Shake for 2 min. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base (KOH) is known to degrade certain PCDDs/PCDFs, so contact time must be minimized.

11.5.1.4 Partition the extract against 40 mL of 5 percent (w/v) aqueous sodium chloride. Shake for 2 min. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a 50-mL round-bottom flask. Rinse the funnel with the sodium sulfate with two 15-mL portions of hexane, add the rinses to the 50-mL flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35 °C water bath) or nitrogen evaporation, making sure all traces of toluene (when applicable) are removed.

### 11.5.2 Silica/alumina column cleanup

11.5.2.1 Pack a gravity column (glass, 30 cm x 10.5 mm), fitted with a PTFE stopcock, with silica gel as follows: Insert a glass wool plug into the bottom of the column. Place 1 g of silica gel in the column and tap the column gently to settle the silica gel. Add 2 g of sodium hydroxide-impregnated silica gel, 4 g of sulfuric acid-impregnated silica gel, and 2 g of silica gel. (Secs. 7.3.4, 7.3.5, and 7.3.3) Tap the column gently after each addition. A small positive pressure (5 psi) of clean nitrogen may be used if needed. Elute with 10 mL of hexane and close the stopcock just before exposure of the top layer of silica gel to air. Discard the eluate. Check the column for channeling. If channeling is observed, discard the column. Do not tap the wetted column.

11.5.2.2 Pack a gravity column (glass, 300 mm x 10.5 mm), fitted with a PTFE stopcock, with alumina as follows: Insert a glass wool plug into the bottom of the column. Add a 4 g layer of sodium sulfate. (Sec 7.5) Add a 4 g layer of Woelm® Super 1 neutral alumina. (Sec. 7.3.1) Tap the top of the column gently. Woelm® Super 1 neutral alumina need not be activated or cleaned before use, but it should be stored in a sealed desiccator. Add a 4 g layer of anhydrous sodium sulfate to cover the alumina. Elute with 10 mL hexane and close the stopcock just before exposure of the sodium sulfate layer to air. Discard the eluate. Check the

column for channeling. If channeling is observed, discard the column. Do not tap a wetted column.

**NOTE:** Alternatively, acidic alumina (Sec. 7.3.2) may be used in place of neutral alumina.

11.5.2.3 Dissolve the residue from Sec. 11.5.1.4 in 2 mL of hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4 mL) to quantitatively transfer the sample to the surface of the silica gel.

11.5.2.4 Elute the silica gel column with 90 mL of hexane, concentrate the eluate on a rotary evaporator (35 °C water bath) to approximately 1 mL, and apply the concentrate to the top of the alumina column (Sec. 11.5.2.2). Rinse the rotary evaporator flask twice with 2 mL of hexane, and add the rinses to the top of the alumina column.

11.5.2.5 Add 20 mL of hexane to the alumina column and elute until the hexane level is just below the top of the sodium sulfate. Do not discard the eluted hexane, but collect it in a separate flask and store it for later use, as it may be useful in determining where the labeled analytes are being lost if recoveries are not satisfactory.

11.5.2.6 Add 15 mL of 60 percent methylene chloride in hexane (v/v) to the alumina column and collect the eluate in a conical-shaped (15-mL) concentration tube. With a carefully regulated stream of nitrogen, concentrate the 60 percent methylene chloride/hexane fraction to about 2 mL.

### 11.5.3 Carbon column cleanup

11.5.3.1 Thoroughly mix 9.0 g of activated carbon (Sec. 7.3.7) and 41.0 g of Celite 545<sup>®</sup> to produce an 18% w/w mixture. Activate the mixture at 130 °C for 6 hr, and store in a desiccator.

**NOTE:** Check each new batch of the carbon/Celite mixture by adding 50 µL of the calibration verification solution to 950 µL of hexane. Take this solution through the carbon column cleanup step, concentrate to 50 µL and analyze. If the recovery of any of the analytes is less than 80%, this batch of carbon/Celite mixture may not be used.

11.5.3.2 Prepare a 4-inch long glass column by cutting off each end of a 10-mL disposable serological pipet. Fire polish both ends and flare if desired. Insert a glass wool plug at one end of the column, and pack it with 1 g of the carbon/Celite mixture. Insert an additional glass wool plug in the other end.

**CAUTION:** It is very important that the column be packed properly to ensure that carbon fines are not carried into the eluate. PCDDs/PCDFs will adhere to the carbon fines and greatly reduce recovery. If carbon fines are carried into the eluate, filter the eluate, using a 0.7-µm filter (pre-rinsed with toluene), then proceed to Sec. 11.5.3.6.

#### 11.5.3.3 Rinse the column with:

- 4 mL of toluene
- 2 mL of methylene chloride/methanol/toluene (75:20:5 v/v)
- 4 mL of cyclohexane/methylene chloride (50:50 v/v)

The flow rate should be less than 0.5 mL/min. Discard all the column rinsates.

11.5.3.4 While the column is still wet, transfer the concentrated eluate from Sec. 11.5.2.6 to the prepared carbon column. Rinse the eluate container with two 0.5-mL portions of hexane and transfer the rinses to the carbon column. Elute the column with the following sequence of solvents:

- 10 mL of cyclohexane/methylene chloride (50:50 v/v).
- 5 mL of methylene chloride/methanol/toluene (75:20:5 v/v).

**NOTE:** The above two eluates may be collected and combined, and used as a check on column efficiency.

11.5.3.5 Once the solvents have eluted through the column, turn the column over, and elute the PCDD/PCDF fraction with 20 mL of toluene, and collect the eluate.

11.5.3.6 Concentrate the toluene fraction to about 1 mL on a rotary evaporator by using a water bath at 50 °C or with nitrogen evaporation. Carefully transfer the concentrate into a 1-mL minivial and, again at elevated temperature (50 °C), reduce the volume to about 100 µL using a stream of nitrogen and a sand bath. Rinse the rotary evaporator flask three times with 300 µL of a solution of 1 percent toluene in methylene chloride, and add the rinses to the concentrate. Add 10 µL of the nonane recovery standard solution (Sec. 7.10) for soil, sediment, water, fish, paper pulp and adipose tissue samples, or 50 µL of the recovery standard solution for sludge, still bottom and fly ash samples. Store the sample at room temperature in the dark.

### 11.6 Chromatographic/mass spectrometric conditions and data acquisition parameters

#### 11.6.1 Gas chromatograph operating conditions

Column coating:	DB-5
Film thickness:	0.25 µm
Column dimension:	60-m x 0.32 mm
Injector temperature:	270 °C
Splitless valve time:	45 s
Interface temperature:	Function of the final temperature
Temperature program	
Initial temperature:	200 °C
Initial hold time:	2 min
1st temp. ramp:	5 °C/min to 220 °C, hold for 16 min
2nd temp. ramp:	5 °C/min to 235 °C, hold for 7 min
3rd temp. ramp:	5 °C/min to 330 °C, hold for 5 min
Total time:	60 min

## 11.6.2 Mass spectrometer

11.6.2.1 The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less (Sec. 11.6.3.1). At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that with the exception of the last descriptor (OCDD/OCDF), all descriptors contain 10 ions. The selection (Table 6) of the molecular ions M and M+2 for <sup>13</sup>C-HxCDF and <sup>13</sup>C-HpCDF rather than M+2 and M+4 (for consistency) was made to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The selection of the lock-mass ion is left to the performing laboratory.

**NOTE:** At the option of the analyst, the tetra- and pentachlorinated dioxins and furans may be combined into a single descriptor.

11.6.2.2 The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6. By using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

## 11.6.3 Data acquisition

11.6.3.1 The total cycle time for data acquisition must be  $\leq 1$  sec. The total cycle time includes the sum of all the dwell times and voltage reset times.

11.6.3.2 Acquire SIM data for all of the ions in the descriptors in Table 6.

## 11.7 Calibration

### 11.7.1 Initial calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria in Sec. 11.7.2. Initial calibration is also required if any routine calibration (Sec. 11.7.3) does not meet the required criteria listed in Sec. 11.7.4.

11.7.1.1 At a minimum, all five high-resolution concentration calibration solutions listed in Table 5 must be used for the initial calibration.

11.7.1.2 Tune the instrument with PFK, as described in Sec. 11.6.2.2.

11.7.1.3 Inject 2  $\mu$ L of the GC column performance check solution (Sec. 7.8) and acquire SIM mass spectral data as described earlier in Sec. 11.6.2. The total cycle time must be  $\leq 1$  sec. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 9.6.1 were met.

11.7.1.4 By using the same GC (Sec. 11.6.1) and MS (Sec. 11.6.2) conditions that produced acceptable results with the column performance check solution, analyze a 2- $\mu$ L portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameters.

11.7.1.4.1 The ratio of integrated ion current for the ions appearing in Table 8 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) for all unlabeled calibration standards in Table 5.

11.7.1.4.2 The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards (Table 5) must be within the control limits stipulated in Table 8.

NOTE: Secs. 11.7.1.4.1 and 11.7.1.4.2 require that 17 ion ratios from Sec. 11.7.1.4.1 and 11 ion ratios from Sec. 11.7.1.4.2 be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

11.7.1.4.3 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. Measurement of S/N is required for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

11.7.1.4.4 Referring to Table 9, calculate the 17 relative response factors (RF) for unlabeled target analytes [RF(n); n = 1 to 17] relative to their appropriate internal standards (Table 5) and the nine RFs for the  $^{13}\text{C}_{12}$ -labeled internal standards [RF(is); is = 18 to 26]) relative to the two recovery standards (Table 5) according to the following formulae:

$$\text{RF}_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

$$\text{RF}_{is} = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

where:

$A_n^1$  and  $A_n^2$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for unlabeled PCDDs/PCDFs,

$A_{is}^1$  and  $A_{is}^2$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the labeled internal standards,

$A_{rs}^1$  and  $A_{rs}^2$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the labeled recovery standards,

$Q_{is}$  = quantity of the internal standard injected (pg),

$Q_{rs}$  = quantity of the recovery standard injected (pg), and

$Q_n$  = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The  $RF_n$  and  $RF_{is}$  are dimensionless quantities; the units used to express  $Q_{is}$ ,  $Q_{rs}$  and  $Q_n$  must be the same.

11.7.1.4.5 Calculate the  $\overline{RF}$  values and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RF}_n = \frac{\sum_{j=1}^5 RF_{n(j)}}{5}$$

where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 9), and j is the injection number (or calibration solution number; j = 1 to 5).

11.7.1.4.6 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 9) are calculated as follows:

For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the RF used will be the same as the RF determined in Sec. 11.7.1.4.5.

**NOTE:** The calibration solutions do not contain  $^{13}C_{12}$ -OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the  $[M+6]^+$  ion of  $^{13}C_{12}$ -OCDF from the  $[M+2]^+$  ion of OCDD (and  $[M+4]^+$  from  $^{13}C_{12}$ -OCDF with  $[M]^+$  of OCDD). Therefore, the  $\overline{RF}$  or OCDF is calculated relative to  $^{13}C_{12}$ -OCDD.

For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the  $\overline{RF}$  used for those homologous series will be the mean of the RFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RF}_k = \frac{\sum_{n=1}^t RF_n}{t}$$

where:

k = 27 to 30 (Table 9), with 27 = PeCDF; 28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

**NOTE:** Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

11.7.1.4.7 Relative response factors ( $RF_m$ ) to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RF_m = \frac{A_{is}^m \times Q_{rs}}{Q_{is}^m \times A_{rs}}$$

$$\overline{RF}_m = \frac{\sum_{j=1}^5 RF_{m(j)}}{5}$$

where:

m = 18 to 26 (congener type) and j = 1 to 5 (injection number),

$A_{is}^m$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for a given internal standard (m = 18 to 26),

$A_{rs}$  = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 9) for the appropriate recovery standard (see Table 5, footnotes),

- $Q_{rs}, Q_{is}^m$  = quantities of, respectively, the recovery standard (rs) and a particular internal standard (is = m) injected (pg),
- $RF_m$  = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and
- $\overline{RF}_m$  = calculated mean relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from the five initial calibration injections (j).

11.7.2 Criteria for acceptable calibration -- The criteria listed below for acceptable calibration must be met before sample analyses are performed.

11.7.2.1 The percent relative standard deviations for the mean response factors ( $\overline{RF}_n$  and  $\overline{RF}_m$ ) from the 17 unlabeled standards must not exceed  $\pm 20$  percent, and those for the nine labeled reference compounds must not exceed  $\pm 20$  percent.

11.7.2.2 The S/N for the GC signals present in every SICP (including the ones for the labeled standards) must be  $\geq 10$ .

11.7.2.3 The ion abundance ratios (Table 8) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Sec. 11.7.2.1 is met, the analyte-specific RF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RFs will be used for all calculations until the routine calibration criteria (Sec. 11.7.4) are no longer met. At such time, new  $\overline{RF}$  values will be calculated from a new set of injections of the calibration solutions.

11.7.3 Routine calibration (continuing calibration check) -- Routine calibrations must be performed at the beginning of a 12-hr period, after successful mass resolution and GC resolution performance checks. A routine calibration is also required at the end of a 12-hr shift. Inject 2  $\mu$ L of the concentration calibration solution HRCC-3 standard (Table 5). By using the same HRGC/HRMS conditions as used in Secs. 11.6.1 and 11.6.2, determine and document an acceptable calibration as provided in Sec. 11.7.4.

11.7.4 Criteria for acceptable routine calibration -- The following criteria must be met before further analysis is performed.

11.7.4.1 The measured RFs [ $RF_n$  for the unlabeled standards] obtained during the routine calibration runs must be within  $\pm 20$  percent of the mean values established during the initial calibration (Sec. 11.7.1.4.5).

11.7.4.2 The measured RFs [ $RF_m$  for the labeled standards] obtained during the routine calibration runs must be within  $\pm 30$  percent of the mean values established during the initial calibration (Sec. 11.7.1.4.7).

11.7.4.3 The ion abundance ratios (Table 8) must be within the allowed control limits.

11.7.4.4 If either one of the criteria in Secs. 11.7.4.1 and 11.7.4.2 is not satisfied, repeat one more time. If these criteria are still not satisfied, the entire routine calibration process (Sec. 11.7.3) must be reviewed. If the ion abundance ratio criterion (Sec. 11.7.4.3) is not satisfied, refer to the note in Sec. 11.7.1.4.2 for resolution.

**NOTE:** An initial calibration must be carried out whenever the HRCC-3, the sample fortification, or the recovery standard solution is replaced by a new solution from a different lot.

## 11.8 Analysis

11.8.1 Remove the sample or blank extract (from Sec. 11.5.3.6) from storage. With a stream of dry, purified nitrogen, reduce the extract volume to 10  $\mu$ L to 50  $\mu$ L.

**NOTE:** A final volume of 20  $\mu$ L or more should be used whenever possible. A 10- $\mu$ L final volume is difficult to handle, and injection of 2  $\mu$ L out of 10  $\mu$ L leaves little sample for confirmations and repeat injections, and for archiving.

11.8.2 Inject a 2- $\mu$ L aliquot of the extract into the GC, operated under the conditions that have been established to produce acceptable results with the performance check solution (Secs. 9.1.6.1 and 9.6.2).

11.8.3 Acquire SIM data according to Secs. 11.6.2 and 11.6.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Secs. 11.7.1.4.4 through 11.7.1.4.7). Ions characteristic of polychlorinated diphenyl ethers are included in the descriptors listed in Table 6.

**NOTE:** The acquisition period must at least encompass the PCDD/PCDF overall retention time window previously determined (Sec. 9.3.1.3). Selected ion current profiles (SICP) for the lock-mass ions (one per mass descriptor) must also be recorded and included in the data package. These SICPs must be true representations of the evolution of the lock-mass ions amplitudes during the HRGC/HRMS run (see Sec. 9.3.2.2 for the proper level of reference compound to be metered into the ion chamber). The analyst may be required to monitor a PFK ion, not as a lock-mass, but as a regular ion, in order to meet this requirement. It is recommended to examine the lock-mass ion SICP for obvious basic sensitivity and stability changes of the instrument during the GC/MS run that could affect the measurements.

11.8.4 Identification criteria -- For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

### 11.8.4.1 Retention times

11.8.4.1.1 For 2,3,7,8-substituted congeners, which have an isotopically-labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Tables 2 and 3), the retention time (RRT; at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 to +3 sec of the isotopically-labeled standard.

11.8.4.1.2 For 2,3,7,8-substituted compounds that do not have an isotopically-labeled internal standard present in the sample

extract (this represents a total of six congeners; Table 3), the retention time must fall within 0.005 retention time units of the relative retention times measured in the routine calibration. Identification of OCDF is based on its retention time relative to  $^{13}\text{C}_{12}$ -OCDD as determined from the daily routine calibration results.

11.8.4.1.3 For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution (Sec. 9.6.2).

11.8.4.1.4 The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach maximum simultaneously ( $\pm 2$  sec).

11.8.4.1.5 The ion current responses for both ions used for the labeled standards (e.g., for  $^{13}\text{C}_{12}$ -TCDD: m/z 331.9368 and m/z 333.9339) must reach maximum simultaneously ( $\pm 2$  sec).

**NOTE:** The analyst is required to verify the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF (Sec. 9.3.1.3) in the SICPs of the daily performance checks. Should either compound be missing, the analyst must take corrective action as it may indicate a potential problem with the ability to detect all of the PCDDs/PCDFs.

#### 11.8.4.2 Ion abundance ratios

The integrated ion currents for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Secs. 11.7.1.4.1 and 11.7.1.4.2 and Table 8 for details.

#### 11.8.4.3 Signal-to-noise ratio

All ion current intensities must be  $\leq 2.5$  times noise level for positive identification of an unlabeled PCDD/PCDF compound or a group of coeluting isomers. Figure 6 describes the procedure to be followed for the determination of the S/N. Labeled analytes must have an S/N  $\geq 10$ .

#### 11.8.4.4 Polychlorinated diphenyl ether interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N  $\leq 2.5$  is detected at the same retention time ( $\pm 2$  sec) in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

### 11.9 Calculations

11.9.1 For gas chromatographic peaks that have met the criteria outlined in Sec. 11.8.4, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RF}_n}$$

where:

$C_x$  = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,

$A_x$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for unlabeled PCDDs/PCDFs,

$A_{is}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

$Q_{is}$  = quantity, in pg, of the internal standard added to the sample before extraction,

$W$  = weight, in g, of the sample (solid or organic liquid), or volume in mL of an aqueous sample, and

$\overline{RF}_n$  = calculated mean relative response factor for the analyte ( $\overline{RF}_n$  with  $n = 1$  to 17; Sec. 11.7.1.4.5).

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs,  $\overline{RF}_n$  is the value calculated using the equation in Sec. 11.7.1.4.5. However, if it is a non-2,3,7,8-substituted congener, the  $RF(k)$  value is the one calculated using the equation in Sec. 11.7.1.4.6 ( $RF_k$ , for  $k = 27$  to 30).

11.9.2 Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{percent recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RF}_m} \times 100$$

where:

$A_{is}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

$A_{rs}$  = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

$Q_{is}$  = quantity, in pg, of the internal standard added to the sample before extraction,

$Q_{rs}$  = quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RF}_m$  = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Sec. 11.7.1.4.7 ( $\overline{RF}_m$  with  $m = 18$  to 26).

**NOTE:** For human adipose tissue, adjust the percent recoveries by adding 1 percent to the calculated value to compensate for the 1 percent of the extract diverted for the lipid determination.

11.9.3 If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limits (MCL) listed in Table 1 (e.g., 200 pg/ $\mu$ L for TCDD in soil), the linear range of response versus concentration may have been exceeded, and a second analysis of the sample (using a one-tenth aliquot) should be undertaken. The volumes of the internal and recovery standard solutions should remain the same as described for the sample preparation (Secs. 11.0).

If a smaller sample size would not be representative of the entire sample, one of the following options is recommended:

- (1) Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to GC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then, analyze the diluted extract.
- (2) Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Immediately following extraction, transfer the sample to a volumetric flask and dilute to known volume. Remove an appropriate aliquot and proceed with cleanup and analysis.
- (3) Use the original analysis data to quantitate the internal standard recoveries. Respike the original extract (note that no additional cleanup is necessary) with 100 times the usual quantity of internal standards. Dilute the re-spiked extract by a factor of 100. Reanalyze the diluted sample using the internal standard recoveries calculated from the initial analysis to correct the results for losses during isolation and cleanup.

11.9.4 The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value must be specified in the report. If an isomer is not detected, use zero (0) in this calculation.

11.9.5 Sample specific estimated quantitation limit -- The sample specific estimated quantitation limit (EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

11.9.5.1 Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression below to calculate an EDL for each 2,3,7,8-substituted PCDD/PCDF that does not have a response with  $S/N \geq 2.5$ . The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a  $^{13}\text{C}$ -labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that peak height. Use the formula:

$$\text{EQL} = \frac{2.5 \times H_x \times Q_{\text{is}}}{H_{\text{is}} \times W \times \overline{\text{RF}}_n}$$

where:

EQL = estimated quantitation limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

$H_x$  = sum of the height of the noise level for each quantitation ion (Table 6) for the unlabeled PCDDs/PCDFs, measured as shown in Figure 6.

$H_{\text{is}}$  = sum of the height of the noise level for each quantitation ion (Table 6) for the labeled internal standard, measured as shown in Figure 6.

$W$ ,  $\overline{\text{RF}}_n$ , and  $Q_{\text{is}}$  retain the same meanings as defined in Sec. 11.9.1.

11.9.5.2 Estimated maximum possible concentration -- An estimated maximum possible concentration (EMPC) is calculated for 2,3,7,8-substituted isomers that are characterized by a response with an S/N of at least 2.5 for both the quantitation ions, and meet all of the identification criteria in Sec. 11.8.4 except the ion abundance ratio criteria or when a peak representing a PCDPE has been detected. An EMPC is a worst-case estimate of the concentration. Calculate the EMPC according to the expression shown in Sec. 11.9.1.

11.9.6 The relative percent difference (RPD) of any duplicate sample results is calculated as follows:

$$\text{RPD} = \frac{|S_1 - S_2|}{\frac{S_1 + S_2}{2}} \times 100$$

where  $S_1$  and  $S_2$  represent sample and duplicate sample results.

11.9.7 The 2,3,7,8-TCDD toxicity equivalents (TE) of PCDDs and PCDFs present in the sample are calculated, if requested by the data user, according to the method recommended by the Chlorinated Dioxins Workgroup (CDWG) of the EPA and the Center for Disease Control (CDC). This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the fifteen 2,3,7,8-substituted PCDDs and PCDFs (Table 3) and to OCDD and OCDF, as shown in Table 10. The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10. The exclusion of other homologous series such as mono-, di-, and tri- chlorinated dibenzodioxins and dibenzofurans does not mean that they are non-toxic. However, their toxicity, as known at this time, is much lower than the toxicity of the compounds listed in Table 10. The above procedure for calculating the 2,3,7,8-TCDD toxicity equivalents is not claimed by the CDWG to be based on a thoroughly established scientific foundation. The procedure, rather, represents a "consensus recommendation on science policy." Since the procedure may be changed in the future, reporting requirements for PCDD and PCDF data would still include the reporting of the analyte concentrations of the PCDD/PCDF congener as calculated in Secs. 11.9.1 and 11.9.4.

#### 11.9.8 Two GC column TEF determination

11.9.8.1 The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60-m DB-5 (or equivalent) fused-silica capillary column. The experimental conditions remain the same as the conditions described previously in Sec. 11.8, and the calculations are performed as outlined in Sec. 11.9. The chromatographic separation between the 2,3,7,8-TCDD and its close eluters (1,2,3,7/1,2,3,8-TCDD and 1,2,3,9-TCDD) must be equal or less than 25 percent valley.

11.9.8.2 The concentration of the 2,3,7,8-TCDF is obtained from the analysis of the sample extract on the 30-m DB-225 (or equivalent) fused-silica capillary column. However, the GC/MS conditions must be altered so that: (1) only the first three descriptors (i.e., tetra-, penta-, and hexachlorinated congeners) of Table 6 are used; and (2) the switching time between descriptor 2 (pentachlorinated congeners) and descriptor 3 (hexachlorinated congeners) takes place following the elution of  $^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD. The concentration calculations are performed as outlined in Sec. 11.9. The chromatographic separation between the 2,3,7,8-TCDF and its close eluters (2,3,4,7-TCDF and 1,2,3,9-TCDF) must be equal or less than 25 percent valley.

NOTE: The confirmation and quantitation of 2,3,7,8-TCDD (Sec. 11.9.8.1) may be accomplished on the SP-2330 GC column instead of the DB-5 column, provided the criteria listed in Sec. 9.3.1 are met and the requirements described in Sec. 9.6.2 are followed.

11.9.8.3 For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance and signal-to-noise ratio criteria described in Secs. 11.8.4.2 and 11.8.4.3, respectively. In addition, the retention time identification criterion described in Sec. 11.8.4.1.1 applies here for congeners for which a carbon-labeled analogue is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogues are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally,

this is accomplished by using the attributions described in Table 11 and the results from the routine calibration run on the SP-2330 column.

## 12.0 DATA ANALYSIS AND CALCULATIONS

12.1 See Sec. 11.9 and Method 8000 for information on data analysis and calculations.

12.2 Results need to be reported in units commensurate with their intended use and all dilutions need to be taken into account when computing final results.

## 13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance goals should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 Table 12 provides data from a comparison of the Soxhlet extraction and pressurized fluid extraction of samples of ground chimney brick. The data are taken from Reference 8 and are provided for guidance purposes only.

13.3 Table 13 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of samples of urban dust. The data are taken from Reference 8 and are provided for guidance purposes only.

13.4 Table 14 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of samples of fly ash. PFE data are provided for samples that were pretreated with an HCl wash and for samples that were not pretreated, but were extracted with a mixture of toluene and acetic acid. The data are taken from Reference 8 and are provided for guidance purposes only.

13.5 Table 15 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of a soil sample (EC-2) from the National Water Research Institute (Burlington, Ontario, Canada) that contained high levels of PCDDs and PCDFs. The data are taken from Reference 8 and are provided for guidance purposes only.

13.6 Table 16 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction of a sediment sample (HS-2) from the National Research Council Institute for Marine Biosciences (Halifax, Nova Scotia, Canada) that contained low levels of PCDDs and PCDFs. The data are taken from Reference 8 and are provided for guidance purposes only.

13.7 Table 17 provides data from a comparison of Soxhlet extraction and pressurized fluid extraction for two field-contaminated sediment samples. The data are taken from Reference 8 and are provided for guidance purposes only.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution

prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

## 16.0 REFERENCES

1. D. G. Patterson, J. S. Holler, D. F. Grote, L. R. Alexander, C. R. Lapeza, R. C. O'Connor and J. A. Liddle, "Control of Interferences in the Analysis of Human Adipose Tissue for 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin," *Environ. Toxicol. Chem.* 5, 355-360 (1986).
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3. "Carcinogens -- Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.
4. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).
5. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)
6. Y. Tondeur, W. J. Niederhut, S. R. Missler, and J. E. Campana, "Hybrid HRGC/MS/MS Method for the Characterization of Tetrachlorinated Dibenzo-*p*-dioxins in Environmental Samples," *Mass Spectrom*, 14, 449-456 (1987).
7. USEPA National Dioxin Study -- Phase II, "Analytical Procedures and Quality Assurance Plan for the Determination of PCDD/PCDF in Fish," EPA-Duluth, October 26, 1987.

8. B. E. Richter, J. L. Ezzell, D. E. Knowles, and F. Hoefler, "Extraction of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Environmental Samples Using Accelerated Solvent Extraction (ASE)," *Chemosphere*, 34 (5-7), 975-987, 1997.

#### 17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

TYPES OF MATRICES, SAMPLE SIZES, AND 2,3,7,8-TCDD-BASED  
METHOD CALIBRATION LIMITS (PARTS PER TRILLION)

	Water	Soil Sediment Paper Pulp <sup>b</sup>	Fly Ash	Fish Tissue <sup>c</sup>	Human Adipose Tissue	Sludge Fuel Oil	Still Bottom
Lower MCL <sup>a</sup>	0.01	1.0	1.0	1.0	1.0	5.0	10
Upper MCL <sup>a</sup>	2	200	200	200	200	1000	2000
Sample Weight (g)	1000	10	10	20	10	2	1
IS Spiking Level (ppt)	1	100	100	100	100	500	1000
Final Ext. Vol. (μL) <sup>d</sup>	10-50	10-50	50	10-50	10-50	50	50

<sup>a</sup> For other congeners multiply the values by 1 for TCDF/PeCDD/PeCDF, by 2.5 for HxCDD/HxCDF/HpCDD/HpCDF, and by 5 for OCDD/OCDF.

<sup>b</sup> Sample dewatered according to Sec. 8.5.

<sup>c</sup> One half of the extract from the 20 g sample is used for determination of lipid content (Sec. 11.2.2).

<sup>d</sup> See Sec. 11.8.1.

**NOTE:** Chemical reactor residues are treated as still bottoms, if their appearances so suggest.

TABLE 2  
COMPOSITION OF THE SAMPLE FORTIFICATION  
AND RECOVERY STANDARD SOLUTIONS<sup>a</sup>

Analyte	Sample Fortification Solution Concentration (pg/ $\mu$ L)	Recovery Standard Solution Concentration (pg/ $\mu$ L)
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	10	--
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	10	--
<sup>13</sup> C <sub>12</sub> -1,2,3,4-TCDD	--	50
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	10	--
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	10	--
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	25	--
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	25	--
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	--	50
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	25	--
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	25	--
<sup>13</sup> C <sub>12</sub> -OCDD	50	--

<sup>a</sup>These solutions should be made freshly every day in nonane or other appropriate solvent because of the possibility of adsorptive losses to glassware. If these solutions are to be kept for more than one day, then the sample fortification solution concentrations should be increased ten fold, and the recovery standard solution concentrations should be doubled. Corresponding adjustments of the spiking volumes must then be made.

TABLE 3

## THE FIFTEEN 2,3,7,8-SUBSTITUTED PCDD AND PCDF CONGENERS

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDF(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF

\* The <sup>13</sup>C-labeled analogue is used as an internal standard.

+ The <sup>13</sup>C-labeled analogue is used as a recovery standard.

TABLE 4

ISOMERS OF CHLORINATED DIOXINS AND FURANS AS A FUNCTION  
OF THE NUMBER OF CHLORINE ATOMS

Number of Chlorine Atoms	Number of Dioxin Isomers	Number of 2,3,7,8-Dioxins	Number of Furan Isomers	Number of 2,3,7,8-Furans
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	75	7	135	10

TABLE 5  
HIGH-RESOLUTION CONCENTRATION CALIBRATION SOLUTIONS

Analyte	Concentration (pg/ $\mu$ L)				
	5	4	3	2	1
<u>Unlabeled Analytes</u>					
2,3,7,8-TCDD	200	50	10	2.5	1
2,3,7,8-TCDF	200	50	10	2.5	1
1,2,3,7,8-PeCDD	500	125	25	6.25	2.5
1,2,3,7,8-PeCDF	500	125	25	6.25	2.5
2,3,4,7,8-PeCDF	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDD	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDD	500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDF	500	125	25	6.25	2.5
2,3,4,6,7,8-HxCDF	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDD	500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDF	500	125	25	6.25	2.5
1,2,3,4,7,8,9-HpCDF	500	125	25	6.25	2.5
OCDD	1,000	250	50	12.5	5
OCDF	1,000	250	50	12.5	5
<u>Internal Standards</u>					
$^{13}\text{C}_{12}$ -2,3,7,8-TCDD	50	50	50	50	50
$^{13}\text{C}_{12}$ -2,3,7,8-TCDF	50	50	50	50	50
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDD	50	50	50	50	50
$^{13}\text{C}_{12}$ -1,2,3,7,8-PeCDF	50	50	50	50	50
$^{13}\text{C}_{12}$ -1,2,3,6,7,8-HxCDD	125	125	125	125	125
$^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDF	125	125	125	125	125
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDD	125	125	125	125	125
$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-HpCDF	125	125	125	125	125
$^{13}\text{C}_{12}$ -OCDD	250	250	250	250	250
<u>Recovery Standards</u>					
$^{13}\text{C}_{12}$ -1,2,3,4-TCDD <sup>(a)</sup>	50	50	50	50	50
$^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD <sup>(b)</sup>	125	125	125	125	125

(a) Used for recovery determinations of TCDD, TCDF, PeCDD and PeCDF internal standards.

(b) Used for recovery determinations of HxCDD, HxCDF, HpCDD, HpCDF and OCDD internal standards.

TABLE 6

## IONS MONITORED FOR HRGC/HRMS ANALYSIS OF PCDDs/PCDFs

Descriptor	Accurate Mass <sup>a</sup>	Ion ID	Elemental Composition	Analyte
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK
2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDF
	341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF
	351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDF (S)
	353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)
	355.8546	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD
	357.8516	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD
	367.8949	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)
	369.8919	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)
	409.7974	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDFE
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK
3	373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF
	375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF
	383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> O	HxCDF (S)
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)

TABLE 6 (Continued)

Descriptor	Accurate Mass <sup>a</sup>	Ion ID	Elemental Composition	Analyte
4	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDPE
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK
	407.7818	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF
	409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF
	417.8250	M	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> O	HpCDF (S)
	419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF
	423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD
	425.7737	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD
	435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)
	437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)
	479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDPE
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK
	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO
443.7399		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
457.7377		M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
459.7348		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
469.7780		M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)
471.7750		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)
513.6775		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE
[442.9728]		LOCK	C <sub>10</sub> F <sub>17</sub>	PFK

S = internal/recovery standard

<sup>a</sup> The following nuclidic masses were used:

H	=	1.007825	O	=	15.994915
C	=	12.000000	<sup>35</sup> Cl	=	34.968853
<sup>13</sup> C	=	13.003355	<sup>37</sup> Cl	=	36.965903
F	=	18.9984			

TABLE 7

PCDD AND PCDF CONGENERS PRESENT IN THE GC PERFORMANCE EVALUATION  
SOLUTION AND USED FOR DEFINING THE HOMOLOGUE GC RETENTION TIME  
WINDOWS ON A 60-M DB-5 COLUMN

# Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	First Eluter	Last Eluter	First Eluter	Last Eluter
4 <sup>a</sup>	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,4,6,7,9/1,2,4,6,8,9	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,8	1,2,3,4,7,8,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

<sup>a</sup>In addition to these two TCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, <sup>13</sup>C<sub>12</sub>-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present as a check of column resolution.

TABLE 8

THEORETICAL ION ABUNDANCE RATIOS AND THEIR CONTROL LIMITS  
FOR PCDDS AND PCDFS

# Chlorine Atoms	Ion Type	Theoretical Abundance Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6 <sup>(a)</sup>	M/M+2	0.51	0.43	0.59
7 <sup>(b)</sup>	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

<sup>a</sup>Used only for <sup>13</sup>C-HxCDF (IS).

<sup>b</sup>Used only for <sup>13</sup>C-HpCDF (IS).

TABLE 9

## RELATIVE RESPONSE FACTOR [RF (NUMBER)] ATTRIBUTIONS

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCDF
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD
19	<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF
20	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD
21	<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF
22	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD
23	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
24	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD
25	<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF
26	<sup>13</sup> C <sub>12</sub> -OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

TABLE 10

2,3,7,8-TCDD TOXICITY EQUIVALENCY FACTORS (TEFs) FOR THE POLYCHLORINATED  
DIBENZODIOXINS AND DIBENZOFURANS

Analyte	TEF <sup>a</sup>
2,3,7,8-TCDD	1.00
1,2,3,7,8-PeCDD	0.50
1,2,3,6,7,8-HxCDD	0.10
1,2,3,7,8,9-HxCDD	0.10
1,2,3,4,7,8-HxCDD	0.10
1,2,3,4,6,7,8-HpCDD	0.01
1,2,3,4,6,7,8,9-OCDD	0.001
2,3,7,8-TCDF	0.1
1,2,3,7,8-PeCDF	0.05
2,3,4,7,8-PeCDF	0.5
1,2,3,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDF	0.1
1,2,3,4,7,8-HxCDF	0.1
2,3,4,6,7,8-HxCDF	0.1
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,7,8,9-HpCDF	0.01
1,2,3,4,6,7,8,9-OCDF	0.001

<sup>a</sup>Taken from "Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-*p*-Dioxin and -Dibenzofurans (CDDs and CDFs) and 1989 Update", (EPA/625/3-89/016, March 1989).

TABLE 11

## ANALYTE RELATIVE RETENTION TIME REFERENCE ATTRIBUTIONS

Analyte	Analyte RRT Reference <sup>a</sup>
1,2,3,4,7,8-HxCDD	<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF

<sup>a</sup>The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to <sup>13</sup>C<sub>12</sub>-1,2,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to <sup>13</sup>C<sub>12</sub>-1,2,3,4,6,7,8-HpCDF.

TABLE 12

COMPARISON OF SOXHLET AND PRESSURIZED FLUID EXTRACTION (PFE)  
FOR EXTRACTION OF GROUND CHIMNEY BRICK

Analyte	Soxhlet (n=1) (ng/kg)	PFE (n=2)* (ng/kg)
2,3,7,8-TCDD	6	6
1,2,3,7,8-PeCDD	52	57
1,2,3,4,7,8-HxCDD	46	52
1,2,3,6,7,8-HxCDD	120	130
1,2,3,7,9,9-HxCDD	97	1000
1,2,3,4,6,7,8-HpCDD	1000	820
OCDD	2900	2600
2,3,7,8-TCDF	160	180
1,2,3,7,8 ( + 1,2,3,4,8)-PeCDF	430	470
2,3,4,7,9-PeCDF	390	390
1,2,3,4,7,8 ( + 1,2,3,4,7,9)-HxCDF	1100	1100
1,2,3,6,7,8-HxCDF	540	570
2,3,4,6,7,8-HxCDF	400	360
1,2,3,7,8,9-HxCDF	42	42
1,2,3,4,6,7,8-HpCDF	2100	2000
1,2,3,4,7,8,9-HpCDF	140	120
OCDF	2000	2000
Total TCDD	440	530
Total PeCDD	900	940
Total HxCDD	1800	2000
Total HpCDD	2000	2100
Total TCDF	2300	2600
Total PeCDF	4100	4300
Total HxCDF	4700	4700
Total HpCDF	2800	2600

\* Sum of two extractions of each sample  
Data from Reference 8

TABLE 13

## COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF URBAN DUST

Analyte	Soxhlet (n=1) (ng/kg)	PFE (n=2)* (ng/kg)
2,3,7,8-TCDD	3.3	3.2
1,2,3,7,8-PeCDD	11.8	13.1
1,2,3,4,7,8-HxCDD	9.8	8.0
1,2,3,6,7,8-HxCDD	11.5	9.5
1,2,3,7,9,9-HxCDD	ND (8)	ND (8)
1,2,3,4,6,7,8-HpCDD	113	107
OCDD	445	314
2,3,7,8-TCDF	12.5	18.6
1,2,3,7,8 ( + 1,2,3,4,8)-PeCDF	9.9	12.0
2,3,4,7,9-PeCDF	13.9	18.1
1,2,3,4,7,8 ( + 1,2,3,4,7,9)-HxCDF	18.7	23.7
1,2,3,6,7,8-HxCDF	10.7	15.8
2,3,4,6,7,8-HxCDF	3.3	8.7
1,2,3,7,8,9-HxCDF	ND (2)	ND (2)
1,2,3,4,6,7,8-HpCDF	13.2	29.4
1,2,3,4,7,8,9-HpCDF	ND (3)	ND (3)
OCDF	ND (10)	ND (10)
Total TCDD	182	325
Total PeCDD	175	281
Total HxCDD	86.7	81.7
Total HpCDD	221	217
Total TCDF	333	419
Total PeCDF	146	179
Total HxCDF	65.9	122
Total HpCDF	13.2	29.4

ND = Not detected, with quantitation limit given in parentheses

\* Sum of two extractions of each sample

Data from Reference 8

TABLE 14

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF FLY ASH  
(with and without HCl pretreatment for PFE)

Analyte	Soxhlet (n=1) with HCl (µg/kg)†	PFE (n=2)* with HCl (µg/kg)†	PFE (n=2)* w/o HCl (µg/kg)‡
2,3,7,8-TCDD	0.32	0.36	0.28
1,2,3,7,8-PeCDD	1.6	2.1	1.7
1,2,3,4,7,8-HxCDD	1.2	1.4	1.2
1,2,3,6,7,8-HxCDD	2.4	2.7	2.4
1,2,3,7,9,9-HxCDD	2.4	2.3	2.2
1,2,3,4,6,7,8-HpCDD	8.2	9.6	8.1
OCDD	11.4	12.8	10.6
2,3,7,8-TCDF	3.7	4.3	3.4
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	4.2	4.6	3.9
2,3,4,7,9-PeCDF	5.6	6.6	5.8
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	7.8	8.7	5.4
1,2,3,6,7,8-HxCDF	7.2	8.5	5.3
2,3,4,6,7,8-HxCDF	6.6	7.2	4.5
1,2,3,7,8,9-HxCDF	0.43	0.56	0.30
1,2,3,4,6,7,8-HpCDF	18.0	17.6	16.8
1,2,3,4,7,8,9-HpCDF	2.3	2.4	2.0
OCDF	13.5	15.8	13.9
Total TCDD	12.0	12.4	10.5
Total PeCDD	16.6	20.5	16.2
Total HxCDD	38.2	42.4	36.7
Total HpCDD	15.0	19.8	16.0
Total TCDF	60.5	67.5	56.1
Total PeCDF	83.5	87.3	77.4
Total HxCDF	65.2	73.5	46.1
Total HpCDF	28.1	32.2	26.5

† Fly ash was pretreated with HCl, followed by a water rinse, and extracted with toluene.

‡ These samples received no HCl pretreatment, and were extracted with a mixture of toluene and acetic acid.

\* Sum of two extractions of each sample  
Data from Reference 8

TABLE 15

## COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF SOIL (EC-2)

Analyte	Soxhlet Results (n=10)		PFE Results (n=2)	
	ng/kg	% RSD	ng/kg	% RSD
2,3,7,8-TCDD	270	9.1	270	0.0
1,2,3,7,8-PeCDD	24	12	22	3.3
1,2,3,4,7,8-HxCDD	23	8.3	24	3.0
1,2,3,6,7,8-HxCDD	83	3.6	87	0.8
1,2,3,7,9,9-HxCDD	60	6.2	57	7.4
1,2,3,4,6,7,8-HpCDD	720	6.7	720	1.0
OCDD	4000	6.2	4200	0.0
2,3,7,8-TCDF *	100	7.3	82	2.6
1,2,3,7,8 ( + 1,2,3,4,8)-PeCDF	39	14	36	3.9
2,3,4,7,9-PeCDF	62	5.5	60	0.0
1,2,3,4,7,8 ( + 1,2,3,4,7,9)-HxCDF	740	5.3	690	0.0
1,2,3,6,7,8-HxCDF	120	6.2	120	0.0
2,3,4,6,7,8-HxCDF	45	9.0	60	1.2
1,2,3,7,8,9-HxCDF	4.9	31	5.3	15
1,2,3,4,6,7,8-HpCDF	2600	6.7	2500	0.0
1,2,3,4,7,8,9-HpCDF	160	5.5	160	0.0
OCDF	7800	8.3	7000	3.1
Total TCDD	430	9.7	370	1.9
Total PeCDD	300	3.7	280	7.7
Total HxCDD	720	5.8	690	2.0
Total HpCDD	1300	7.0	1300	0.0
Total TCDF	620	12	380	19
Total PeCDF	820	9.4	710	7.0
Total HxCDF	1900	5.7	1900	0.0
Total HpCDF	3800	8.2	3900	3.6

\* Single-column analysis only, may include contributions from other isomers that may co-elute.  
Data from Reference 8

TABLE 16

## COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF SEDIMENT (HS-2)

Analyte	Soxhlet Results (n=10)		PFE Results (n=2)	
	ng/kg	% RSD	ng/kg	% RSD
2,3,7,8-TCDD	ND (1)	--	ND (1)	--
1,2,3,7,8-PeCDD	1.6	4.6	ND (1)	--
1,2,3,4,7,8-HxCDD	4.5	4.8	5.2	11
1,2,3,6,7,8-HxCDD	19	4.3	21	0.0
1,2,3,7,9,9-HxCDD	24	4.3	28	2.6
1,2,3,4,6,7,8-HpCDD	1200	8.1	1300	0.0
OCDD	6500	4.2	7100	0.0
2,3,7,8-TCDF *	8.5	11	6.6	5.4
1,2,3,7,8 (+ 1,2,3,4,8)-PeCDF	1.9	17	2.0	0.0
2,3,4,7,9-PeCDF	3.7	7.9	3.7	3.8
1,2,3,4,7,8 (+ 1,2,3,4,7,9)-HxCDF	17	7.3	17	4.3
1,2,3,6,7,8-HxCDF	3.7	5.6	4.0	5.4
2,3,4,6,7,8-HxCDF	3.7	18	4.4	3.2
1,2,3,7,8,9-HxCDF	ND (1)	--	ND (1)	--
1,2,3,4,6,7,8-HpCDF	91	1.6	96	3.7
1,2,3,4,7,8,9-HpCDF	5.2	6.7	5.3	6.7
OCDF	300	3.8	280	2.6
Total TCDD	3.9	14	2.5	34
Total PeCDD	17	7.8	10	10
Total HxCDD	510	5.6	570	1.3
Total HpCDD	4700	8.3	5100	11
Total TCDF	39	11	24	3.0
Total PeCDF	33	13	28	0.0
Total HxCDF	89	3.2	87	12
Total HpCDF	293	3.3	310	0.0

\* Single-column analysis only, may include contributions from other isomers that may co-elute.

. ND = Not detected, with quantitation limit given in parentheses

. Data from Reference 8

TABLE 17

COMPARISON OF SOXHLET AND PFE FOR EXTRACTION OF  
CONTAMINATED SEDIMENTS

Analyte	Hamilton Harbor		Parrots Bay	
	Soxhlet	PFE	Soxhlet	PFE
2,3,7,8-TCDD	3.7	3.1	19	19
1,2,3,7,8-PeCDD	5.1	5.4	8.3	6.0
1,2,3,4,7,8-HxCDD	6.4	7.2	8.6	6.7
1,2,3,6,7,8-HxCDD	27	26	26	17
1,2,3,7,9,9-HxCDD	20	28	24	18
1,2,3,4,6,7,8-HpCDD	460	430	280	250
OCDD	3100	3100	1900	1600
2,3,7,8-TCDF *	61	44	80	48
1,2,3,7,8 ( + 1,2,3,4,8)-PeCDF	14	14	ND (20)	9.8
2,3,4,7,9-PeCDF	26	25	22	14
1,2,3,4,7,8 ( + 1,2,3,4,7,9)-HxCDF	27	37	79	59
1,2,3,6,7,8-HxCDF	17	16	ND (20)	15
2,3,4,6,7,8-HxCDF	14	14	21	11
1,2,3,7,8,9-HxCDF	ND (2)	1.6	4.9	ND (1)
1,2,3,4,6,7,8-HpCDF	130	130	270	220
1,2,3,4,7,8,9-HpCDF	14	13	17	12
OCDF	270	210	510	370
Total TCDD	50	14	39	48
Total PeCDD	63	15	87	66
Total HxCDD	220	180	230	200
Total HpCDD	850	810	580	530
Total TCDF	370	130	400	270
Total PeCDF	290	110	180	170
Total HxCDF	240	160	230	230
Total HpCDF	350	290	400	360

\* Single-column analysis only, may include contributions from other isomers that may co-elute.  
. ND = Not detected, with quantitation limit given in parentheses  
. Data from Reference 8

FIGURE 1

GENERAL STRUCTURES OF DIBENZO-*p*-DIOXIN (TOP) AND DIBENZOFURAN (BOTTOM)

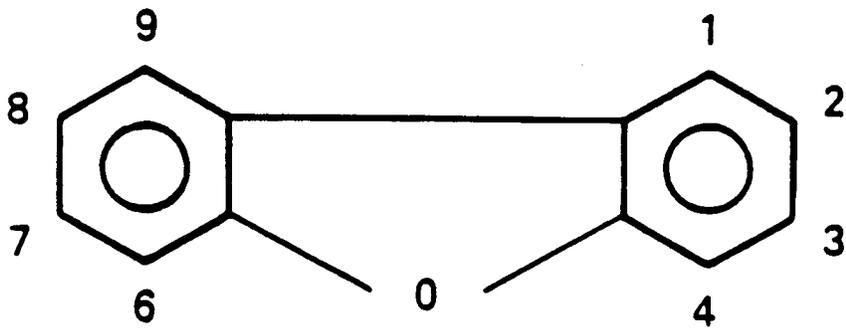
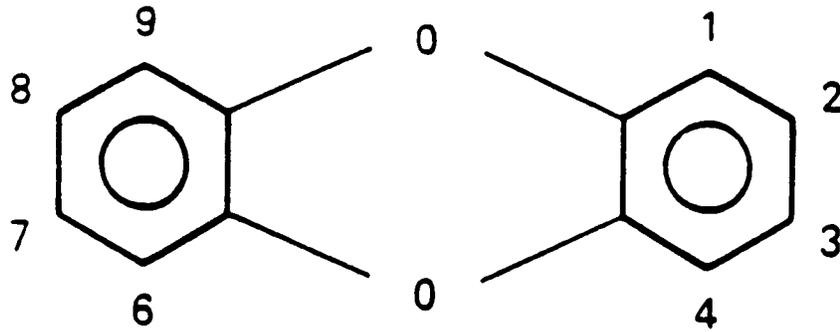
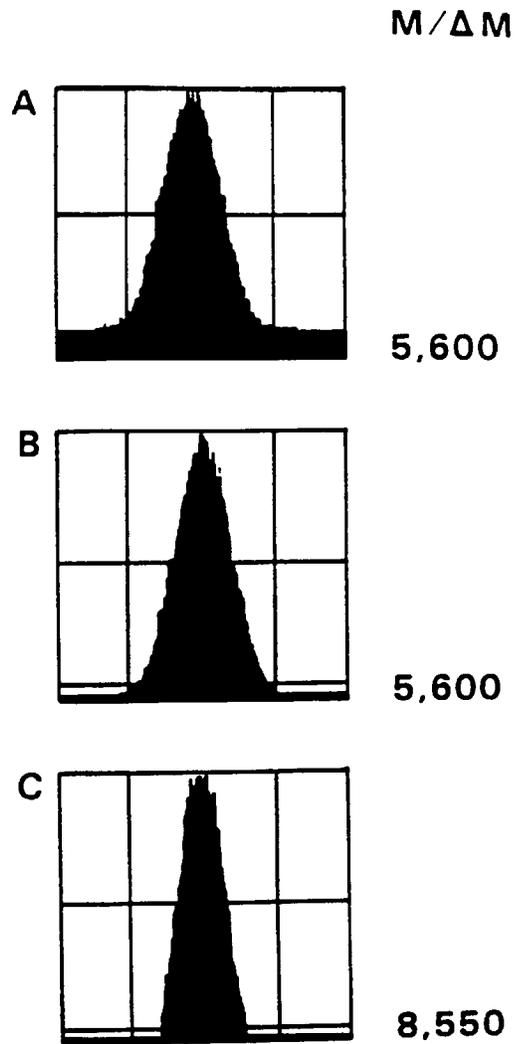


FIGURE 2

PEAK PROFILE DISPLAYS DEMONSTRATING THE EFFECT OF THE DETECTOR ZERO ON THE MEASURED RESOLVING POWER



In this example, the true resolving power is 5,600.

- A) The zero was set too high; no effect is observed upon the measurement of the resolving power.
- B) The zero was adjusted properly.
- C) The zero was set too low; this results in overestimating the actual resolving power because the peak-to-peak noise cannot be measured accurately.

FIGURE 3

TYPICAL 12-Hr ANALYSIS SEQUENCE OF EVENTS.

## Analytical Procedure

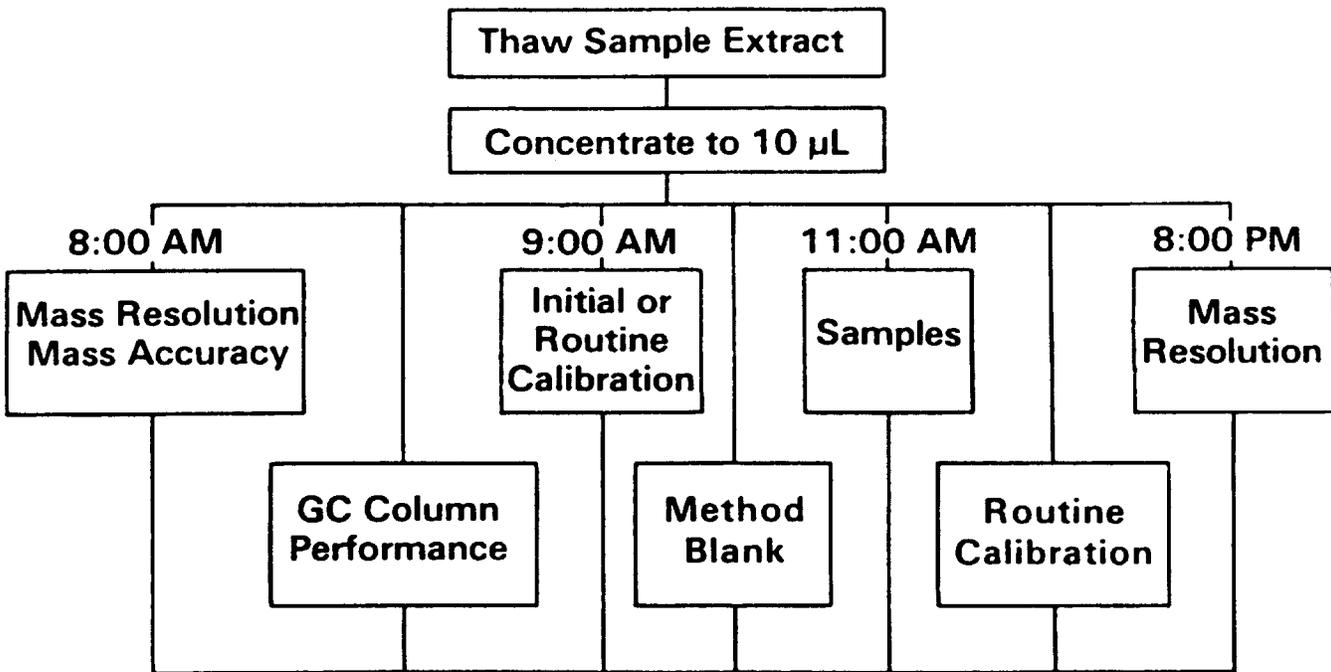


FIGURE 4

SELECTED ION CURRENT PROFILE FOR M/Z 322 (TCDDS) PRODUCED BY MS ANALYSIS OF THE GC PERFORMANCE CHECK SOLUTION ON A 60 M DB-5 FUSED-SILICA CAPILLARY COLUMN UNDER THE CONDITIONS LISTED IN SEC. 11.6.

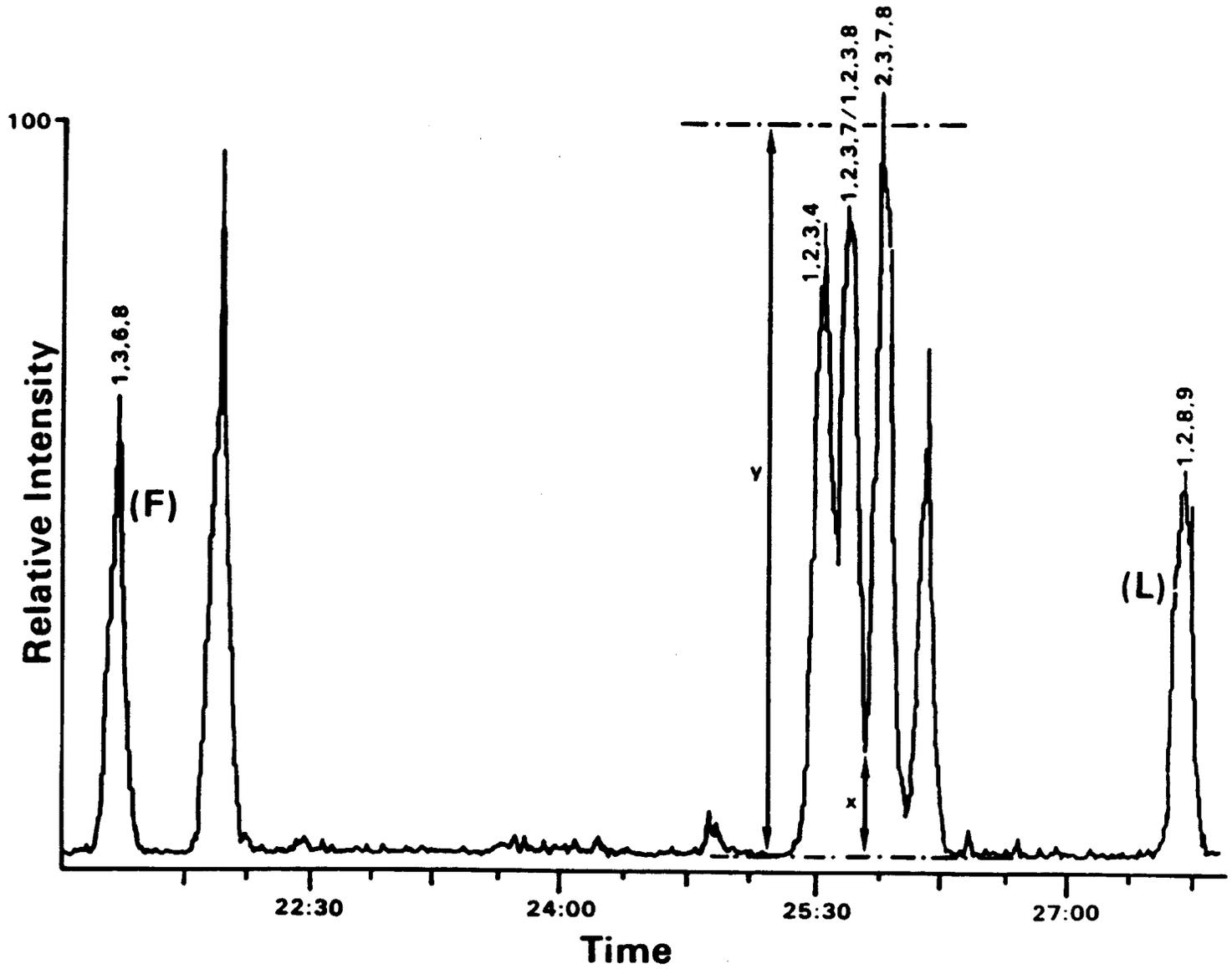
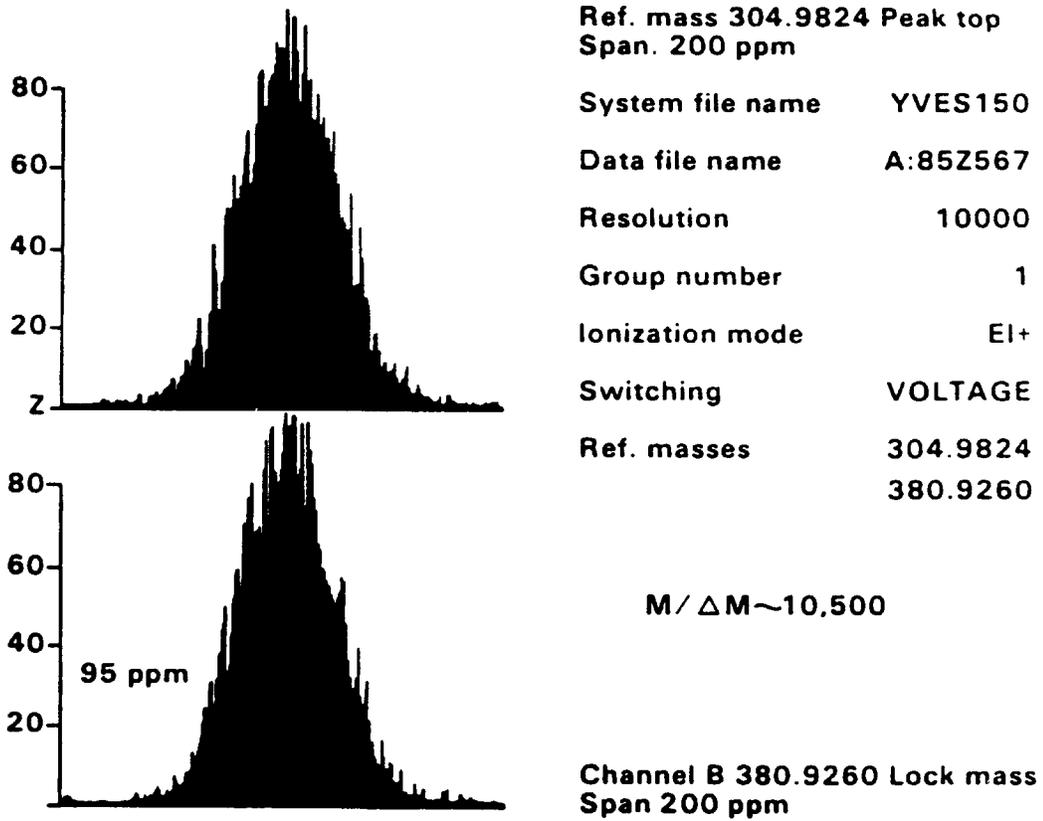


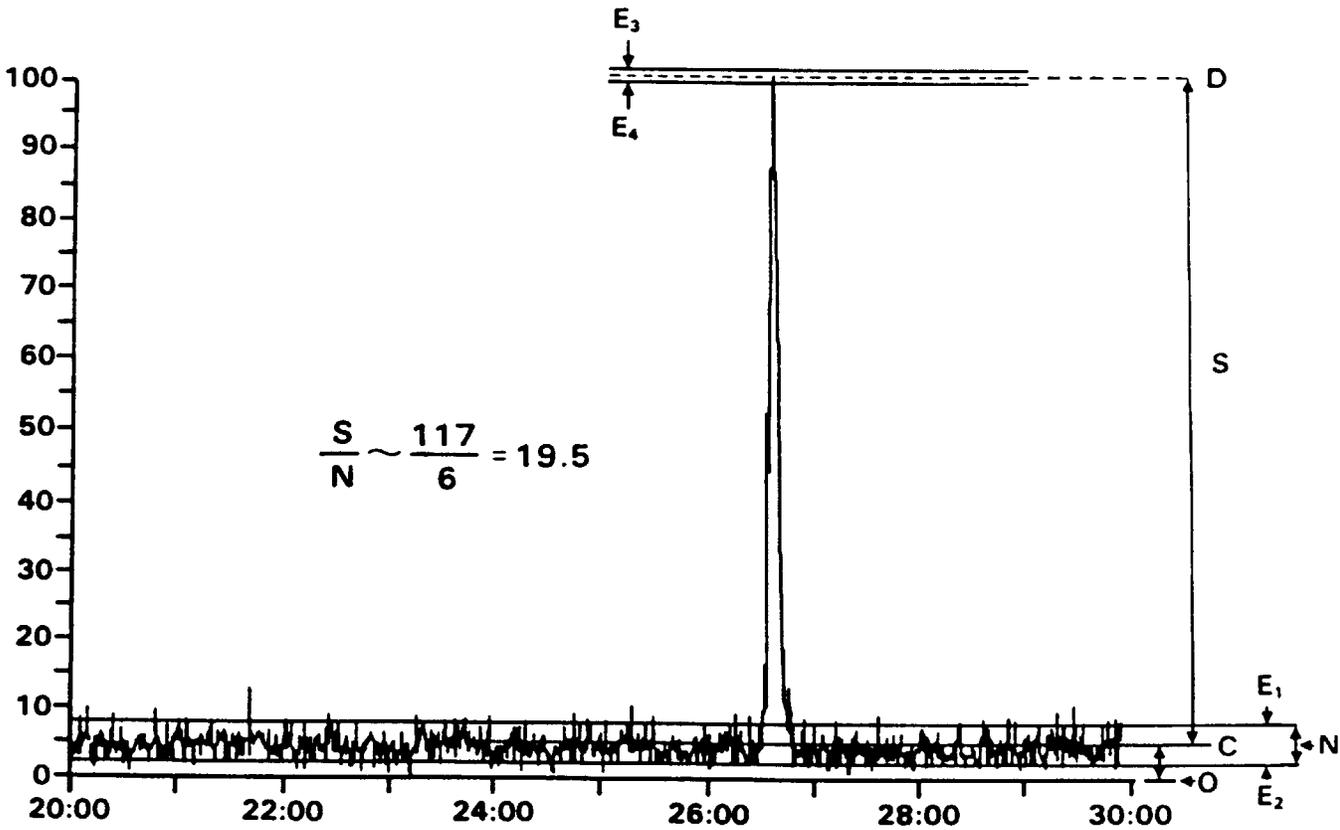
FIGURE 5

PEAK PROFILES REPRESENTING TWO PFK REFERENCE IONS AT M/Z 305 AND 381



The resolution of the high-mass signal is 95 ppm at 5 percent of the peak height; this corresponds to a resolving power  $M/\Delta M$  of 10,500 (10 percent valley definition).

FIGURE 6  
 MANUAL DETERMINATION OF S/N.



The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

## APPENDIX A

### PROCEDURES FOR THE COLLECTION, HANDLING, ANALYSIS, AND REPORTING OF WIPE TESTS PERFORMED WITHIN THE LABORATORY

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

A.1 Perform the wipe tests on surface areas of two inches by one foot with glass fiber paper saturated with distilled-in-glass acetone using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL of distilled-in-glass acetone. Place an equal number of unused wipers in 200 mL acetone and use this as a control. Add 100  $\mu$ L of the sample fortification solution (Sec. 7.8) to each jar containing used or unused wipers.

A.1.1 Close the jar containing the wipers and the acetone and extract for 20 min using a wrist action shaker. Transfer the extract into a K-D apparatus fitted with a concentration tube and a three-ball Snyder column. Add two PTFE or Carborundum™ boiling chips and concentrate the extract to an apparent volume of 1.0 mL on a steam bath. Rinse the Snyder column and the K-D assembly with two 1-mL portions of hexane into the concentrator tube, and concentrate its contents to near dryness with a gentle stream of nitrogen. Add 1.0 mL of hexane to the concentrator tube and swirl the solvent on the walls.

A.1.2 Prepare a neutral alumina column as described in Sec. 11.5.2.2 and follow the steps outlined in Secs. 11.5.2.3 through 11.5.2.5.

A.1.3 Add 10  $\mu$ L of the recovery standard solution as described in Sec. 11.5.3.6.

A.2 Concentrate the contents of the vial to a final volume of 10  $\mu$ L (either in a minivial or in a capillary tube). Inject 2  $\mu$ L of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as described in the analytical method in Sec. 11.8. Perform calculations according to Sec. 11.9.

A.3 Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 10 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 3 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is  $10 \times 5 = 50$  pg/WTE and the positive response for the blank would be  $3 \times 5 = 15$  pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

A.4 At a minimum, wipe tests should be performed when there is evidence of contamination in the method blanks.

A.5 An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed (use multiplication factors listed in footnote (a) from Table 1 for other congeners). This value corresponds to 2½ times the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of

wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory after corrective action has been taken.



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Environment  
Programme**

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**Conference of the Parties of the Stockholm  
Convention on Persistent Organic Pollutants  
Third meeting**

Dakar, 30 April–4 May 2007

Item 5 (b) (ii) of the provisional agenda\*

**Matters for consideration or action by the Conference of the Parties:  
measures to reduce or eliminate releases from unintentional production:  
identification and quantification of releases**

**World Health Organization re-evaluation of dioxin toxic  
equivalency factors**

**Note by the Secretariat**

The annex to the present note contains information on the World Health Organization re-evaluation of dioxin toxic equivalency factors. The information is being circulated as submitted by the secretariat of the World Health Organization and had not been formally edited.

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\* UNEP/POPS/COP.3/1.



## WHO Re-evaluation of Dioxin Toxic Equivalency Factors

### *Background*

In the Stockholm Convention, Annex C, Part IV: Definitions, it is stated that:

*'The toxic equivalency factor values to be used for the purposes of this Convention shall be consistent with accepted international standards, commencing with the World Health Organization 1998 mammalian toxic equivalency factor values for polychlorinated dibenzo-p-dioxins and dibenzofurans and coplanar polychlorinated biphenyls.'*

Over the last 15 years, WHO through the International Programme on Chemical Safety (IPCS) has established and regularly re-evaluated Toxic Equivalency Factors (TEFs) for dioxins and related compounds through expert consultations. WHO-TEF values have been established for humans and mammals, birds and fish. These international-consensus TEFs have been developed to allow assessment and management of complex mixtures of these related and co-occurring compounds. The WHO-TEF scheme has been adopted formally by a number of countries and supranational bodies for monitoring and regulatory purposes.

### **WHO re-evaluation of TEFs**

At the last assessment in 1997 by a WHO expert consultation in Stockholm, it was agreed to re-evaluate TEF values on a regular basis. To follow up on this recommendation, an expert workshop was held in June 2005 at WHO Headquarters in Geneva. A Public Session was held immediately preceding the workshop to give interested parties an opportunity to express their views.

At the workshop itself, an extensive review was performed and each TEF value considered in the light of new data, which led to the revision of a number of values. The table attached lists the TEF values for all 29 congeners currently included in the TEF scheme. The new 2005 WHO-TEF values are given as well as the previous 1998 WHO-TEF values for comparative purposes.

### *Implication of changes*

The expert group examined the implication of the changes in the TEF values on a few case examples and, in general, application of the 2005 TEF values will lead to a slight decrease in total Toxic Equivalents (TEQs) compared to application of the 1998 TEF values.

### *WHO recommendation*

The 2005 WHO dioxin TEFs are based on the latest scientific information available at the time of the review and supersede the 1998 values. WHO recommends that countries note and, where applicable, implement usage of the new WHO 2005 TEF values to replace the previous TEF scheme, consistent with Annex C of the Stockholm Convention referred to above.

For more details please refer to the WHO website at: [http://www.who.int/ipcs/assessment/tef\\_update/en/index.html](http://www.who.int/ipcs/assessment/tef_update/en/index.html).

The outcome of the expert workshop has been published as a scientific review paper:  
Van den Berg et al., TOXICOLOGICAL SCIENCES 93(2), 223–241 (2006).



Compound	WHO 1998 TEF	WHO 2005 TEF*
<i>chlorinated dibenzo-p-dioxins</i>		
2,3,7,8-TCDD	1	1
1,2,3,7,8-PeCDD	1	1
1,2,3,4,7,8-HxCDD	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01
OCDD	0.0001	<b>0.0003</b>
<i>chlorinated dibenzofurans</i>		
2,3,7,8-TCDF	0.1	0.1
1,2,3,7,8-PeCDF	0.05	<b>0.03</b>
2,3,4,7,8-PeCDF	0.5	<b>0.3</b>
1,2,3,4,7,8-HxCDF	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01
1,2,3,6,7,8,9-HpCDF	0.01	0.01
OCDF	0.0001	<b>0.0003</b>
<i>non-ortho substituted PCBs</i>		
PCB 77	0.0001	0.0001
PCB 81	0.0001	<b>0.0003</b>
PCB 126	0.1	0.1
PCB 169	0.01	<b>0.03</b>
<i>mono-ortho substituted PCBs</i>		
105	0.0001	<b>0.00003</b>
114	0.0005	<b>0.00003</b>
118	0.0001	<b>0.00003</b>
123	0.0001	<b>0.00003</b>
156	0.0005	<b>0.00003</b>
157	0.0005	<b>0.00003</b>
167	0.00001	<b>0.00003</b>
189	0.0001	<b>0.00003</b>

\* Numbers in bold indicate a change in TEF value

Reference - *Van den Berg et al.*

The 2005 World Health Organization Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxins and Dioxin-like Compounds

TOXICOLOGICAL SCIENCES 93(2), 223-241 (2006)

**Attachment B**  
**DoD ELAP Accreditation Letters**

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SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL LLC  
 2425 New Holland Pike  
 Lancaster, PA 17601  
 Dorothy M. Love Phone: 717-556-7327

ENVIRONMENTAL

Valid To: November 30, 2016

Certificate Number: 0001.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2009 NELAC Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.0 of the DoD Quality Systems Manual for Environmental Laboratories, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F<sup>-</sup>, O<sub>2</sub>), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
<b>Demands</b>					
BOD	-----	-----	SM 5210B-2001	-----	-----
CBOD	-----	-----	SM 5210B-2001	-----	-----
COD	-----	-----	EPA 410.4	-----	-----
Total Carbon	-----	-----	-----	SM 5310C-2000	SM 5310B-2000 MOD
Total Inorganic Carbon	-----	-----	-----	SM 5310C-2000	SM 5310B-2000 MOD

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Total Organic Carbon	-----	-----	EPA 415.1 EPA 9060 EPA 9060A SM 5310C-2000	EPA 9060 EPA 9060A SM 5310C-2000	EPA 9060 EPA 9060A SM 5310B MOD
<b>Nutrients</b>					
Ammonia	-----	-----	EPA 350.1 SM 4500 NH3 B & D-1997	-----	EPA 350.1
Fluoride	-----	-----	SM 4500 FC-1997 EPA 300.0 EPA 340.2 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	EPA 300.0
Nitrate (as N)	-----	-----	EPA 300.0 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	EPA 300.0
Nitrite (as N)	-----	-----	EPA 300.0 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	EPA 300.0
Nitrate/Nitrite	-----	-----	EPA 353.2	-----	-----
Orthophosphate (as P)	-----	-----	EPA 365.3	-----	-----
Total Kjeldahl Nitrogen	-----	-----	EPA 351.2	-----	EPA 351.2
Total Phosphorus	-----	-----	EPA 365.1	-----	EPA 365.1
<b>Wet Chemistry</b>					
Acid Volatile Sulfide	-----	-----	-----	-----	EPA-821-R-91-100
Acidity	-----	-----	SM 2310B-1997	-----	-----
Alkalinity	-----	-----	SM 2320B-1997	-----	-----
Bromide	-----	-----	EPA 300.0 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	-----
Bulk Density	-----	-----	-----	ASTM E868-82	ASTM E868-82
Chloride	-----	-----	EPA 300.0 EPA 325.3 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	EPA 300.0
Color	-----	-----	SM 2120B-2001	-----	-----
Corrosivity	-----	-----	-----	SW-846 Chapter 7	SW-846 Chapter 7
Cyanide	EPA 9012A EPA 9012B	-----	EPA 335.2 EPA 335.4 MOD EPA 9012A EPA 9012B ASTM D7511 OIA-1677-09	EPA 9012A EPA 9012B ASTM D7511 OIA-1677-09	EPA 9012A EPA 9012B

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Dissolved Oxygen	-----	-----	SM 4500 OG-2001	-----	-----
Dissolved Silica	-----	-----	EPA 370.1 SM 4500 SiC-1997	-----	-----
Ferrous Iron	-----	-----	SM 3500Fe B-MOD 1997	-----	-----
Filterable Residue	-----	-----	SM 2540C-1997	-----	-----
Flashpoint	-----	-----	-----	EPA1010A	EPA 1010A
Grain Size	-----	-----	-----	-----	ASTM D422
Hardness	-----	-----	SM 2340C-1997	-----	-----
HEM-SGT	-----	-----	EPA 1664A EPA 1664B	-----	EPA 9071B
Hexavalent Chromium Digestion	EPA 3060A	-----	-----	-----	EPA 3060A
Hexavalent Chromium	EPA 7196A	-----	SM 3500 CrB-2009 EPA 218.6 EPA 7196A EPA 7199	EPA 218.6 EPA 7196A EPA 7199	EPA 7196A EPA 7199
Ignitability	-----	-----	-----	40 CFR 261.21	40 CFR 261.21
Non-filterable Residue	-----	-----	EPA 160.2 SM 2540D-1997	-----	-----
Oxidation Reduction Potential	-----	-----	ASTM D1498	-----	-----
Paint Filter Test	-----	-----	EPA 9095A	EPA 9095A	EPA 9095A
pH	-----	-----	SM 4500 H+B-2000 EPA 150.1 EPA 9040B EPA 9040C	EPA 9040B EPA 9040C EPA 9045C EPA 9045D	EPA 9040B EPA 9040C EPA 9045C EPA 9045D
Phenol	-----	-----	EPA 420.4 EPA 9066	EPA 9066	-----
Reactivity	-----	-----	-----	SW-846 Chapter 7.3	SW-846 Chapter 7.3
Settleable Residue	-----	-----	SM 2540F-1997	-----	-----
Specific Conductance	-----	-----	EPA 120.1 SM 2510B-1997 EPA 9050A	EPA 9050A	-----
Sulfate	-----	-----	EPA 300.0 EPA 375.4 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A	EPA 300.0

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Sulfide	-----	-----	EPA 376.1 EPA 376.2 SM 4500 S2D- 2000 SM 4500 S2F- 2000	-----	-----
Surfactants	-----	-----	SM 5540C-2000	-----	-----
Total Filterable Residue	-----	-----	SM 2540C-1997	-----	-----
Total Residue	-----	-----	EPA 160.3 SM 2540B-1997	-----	-----
Turbidity	-----	-----	EPA 180.1 SM 2130 B-2001	-----	-----
Volatile Residue	-----	-----	EPA 160.4	-----	-----
<b>Metals</b>					
Metals Digestion	EPA 3050B	EPA 3050B	EPA 200.2 EPA 3050B EPA 3005A EPA 3010A EPA 3010A MOD	EPA 3050B EPA 3010A EPA 3010A MOD	EPA 3050B
Aluminum	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Antimony	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Arsenic	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Barium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Beryllium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Boron	EPA 6010B EPA 6010C	-----	EPA 200.7 EPA 6010B EPA 6010C	EPA 6010B EPA 6010C	EPA 6010B EPA 6010C
Cadmium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Calcium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Chromium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Cobalt	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Copper	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Iron	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Lead	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Molybdenum	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Magnesium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Manganese	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Mercury	EPA 7471A EPA 7471B	-----	EPA 245.1 EPA 7470A	EPA 7470A	EPA 7471A EPA 7471B
Nickel	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Potassium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Selenium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Silicon	-----	-----	EPA 6010C	EPA 6010C	EPA 6010C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Silver	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Sodium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Strontium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Thallium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	-----	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Tin	EPA 6010B EPA 6010C	-----	EPA 200.7 EPA 6010B EPA 6010C	EPA 6010B EPA 6010C	EPA 6010B EPA 6010C
Vanadium	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Zinc	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 200.8 MOD EPA 6020 EPA 6020A	EPA 200.7 EPA 200.8 EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A	EPA 6010B EPA 6010C EPA 6020 EPA 6020A
Zirconium	-----	-----	EPA 200.7 EPA 6010B EPA 6010C	EPA 200.7 EPA 6010B EPA 6010C	EPA 200.7 EPA 6010B EPA 6010C
<b>Purgeable Organics (Volatiles)</b>					
Volatile Preparation	-----	-----	EPA 5030A EPA 5030B	EPA 5030A EPA 5030B	EPA 5035 EPA 5035A

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Acetone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Acetonitrile	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Acrolein	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Acrylonitrile	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Alpha Methyl Styrene	-----	EPA TO-15	-----	-----	-----
Allyl Chloride	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
tert-Amyl Alcohol	-----	-----	-----	-----	EPA 8260B EPA 8260C
tert-Amyl Methyl Ether	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
tert-Butyl Alcohol	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
tert-Butyl formate	-----	-----	-----	-----	EPA 8260B EPA 8260C
Benzene	-----	EPA TO-15 EPA TO- 15 SIM EPA 18 mod EPA 25 mod	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
Benzyl Chloride	-----	EPA TO-15	-----	-----	-----

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Bromobenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Bromochloromethane	-----	-----	EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Bromodichloromethane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Bromoethene	-----	EPA TO-15	-----	-----	-----
Bromoform	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Bromomethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Butane	-----	EPA 18 mod EPA 25 mod	-----	-----	-----
1,3-Butadiene	-----	EPA TO-15 EPA TO- 15 SIM	-----	-----	-----
2-Butanone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
n-Butylbenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
sec-Butylbenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
tert-Butylbenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Carbon Disulfide	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Carbon Tetrachloride	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
2-Chloro-1,3-Butadiene	-----	-----	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Chloroacetonitrile	-----	-----	EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Chlorobenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1-Chlorobutane	-----	-----	EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Chlorodifluoromethane	-----	EPA TO-15	-----	-----	-----
Chloroethane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
2-Chloroethyl Vinyl Ether	-----	-----	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Carbon Range Organics C1-C10 (including subsets of this range i.e. hydrocarbons as propane, hydrocarbons as methane, hydrocarbons as hexane)	-----	EPA 18 mod EPA 25 mod	-----	-----	-----

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Chloroform	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Chloromethane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
3-Chloroprene	-----	EPA TO-15	-----	-----	-----
2-Chlorotoluene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
4-Chlorotoluene	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Cyclohexane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Di-Isopropyl Ether	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Dibromochloromethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,2-Dibromo-3- chloropropane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8011 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8011 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Dibromomethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
1,2-Dibromoethane (EDB)	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8011 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8011 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,2-Dichlorobenzene	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,3-Dichlorobenzene	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,4-Dichlorobenzene	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
trans-1,4-Dichloro-2-Butene	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Dichlorodifluoromethane	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1-Dichloroethane	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,2-Dichloroethane	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1-Dichloroethene	-----	EPA TO-15 EPA TO-15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
cis-1,2-Dichloroethene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
trans-1,2-Dichloroethene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Dichlorofluoromethane	-----	EPA TO-15	EPA 524.2 (DW)	-----	-----
1,2-Dichloropropane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,3-Dichloropropane	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
2,2-Dichloropropane	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1-Dichloropropanone	-----	-----	EPA 524.2 (DW)	-----	-----
1,1-Dichloropropene	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
cis-1,3-Dichloropropene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
trans-1,3-Dichloropropene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,4-Dioxane	-----	EPA TO-15	EPA 8260B EPA 8260C EPA 8260 SIM	EPA 8260B EPA 8260C EPA 8260 SIM	EPA 8260B EPA 8260C EPA 8260 SIM

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Ethanol	-----	EPA TO-15	EPA 8260B EPA 8260C EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Ethane	-----	EPA 18 mod EPA 25 mod	-----	-----	-----
Ethyl Acetate	-----	EPA TO-15	-----	-----	-----
Ethyl Acrylate	-----	EPA TO-15	-----	-----	-----
Ethylbenzene	-----	EPA TO-15 EPA TO- 15 SIM EPA 18 mod EPA 25 mod	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
Ethyl Ether	-----	-----	EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Ethyl Methacrylate	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
4-Ethyltoluene	-----	EPA TO-15	-----	-----	-----
Ethyl tert-Butyl Ether	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Freon-113	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Freon-114	-----	EPA TO-15	-----	-----	-----
Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)]	-----	-----	EPA 8015B EPA 8015C EPA 8015D EPA 8260B EPA 8260C NW TPH-Gx MA VPH WA DOE VPH OA-1	EPA 8015B EPA 8015C EPA 8015D EPA 8260B EPA 8260C NW TPH-Gx MA VPH WA DOE VPH OA-1	EPA 8015B EPA 8015C EPA 8015D EPA 8260B EPA 8260C NW TPH-Gx MA VPH WA DOE VPH OA-1

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Heptane	-----	EPA TO-15	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Hexane	-----	EPA TO-15 EPA 18 mod EPA 25 mod	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
2-Hexanone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Hexachlorobutadiene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Hexachloroethane	-----	EPA TO-15	EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Isooctane	-----	EPA TO-15	-----	-----	-----
Isopropyl Alcohol	-----	EPA TO-15	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Isopropylbenzene	-----	EPA TO-15	EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260COA-1
1,4-Isopropyltoluene	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methane	-----	EPA 18 mod EPA 25 mod	-----	-----	-----
Methylacrylonitrile	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl Acetate	-----	-----	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl Acrylate	-----	EPA TO-15	EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Methyl Iodide	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl Ethyl Ketone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methylene Chloride	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl Isobutyl Ketone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl Methacrylate	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methyl tert-Butyl Ether	-----	EPA TO-15 EPA TO- 15 SIM EPA 18 mod EPA 25 mod	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
4-Methyl-2-pentanone	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Methylcyclohexane	-----	-----	EPA 624 EPA 8260B EPA 8260C	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
2-Nitropropane	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Naphthalene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
Nitrobenzene	-----	-----	EPA 524.2 (DW)	-----	-----

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Octane	-----	EPA TO-15	-----	-----	-----
Pentachloroethane	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Pentane	-----	EPA TO-15 EPA 18 mod EPA 25 mod	-----	-----	-----
Propionitrile	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Propane	-----	EPA 18 mod EPA 25 mod	-----	-----	-----
Propene	-----	EPA TO-15	-----	-----	-----
n-Propylbenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Styrene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 602 EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
tert-Amyl Ethyl Ether	-----	-----	EPA 8260B EPA 8260C EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1,1,2- Tetrachloroethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1,2,2- Tetrachloroethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Tetrachloroethene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Tetrahydrofuran	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW)	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Toluene	-----	EPA TO-15 EPA TO- 15 SIM EPA 18 mod EPA 25 mod	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
1,2,3-Trichlorobenzene	-----	-----	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,2,4-Trichlorobenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1,1-Trichloroethane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,1,2-Trichloroethane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Trichloroethene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Trichlorofluoromethane	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
1,2,3-Trichloropropane	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,2,4-Trimethylbenzene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
1,3,5-Trimethylbenzene	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Vinyl Acetate	-----	EPA TO-15	EPA 624 EPA 8260B EPA 8260C EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Vinyl Chloride	-----	EPA TO-15 EPA TO- 15 SIM	EPA 624 EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B	EPA 8260B EPA 8260C	EPA 8260B EPA 8260C
Xylenes, total	-----	EPA TO-15 EPA TO- 15 SIM EPA 18 mod EPA 25 mod	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
1,2-Xylene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
1,3-Xylene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
1,4-Xylene	-----	EPA TO-15 EPA TO- 15 SIM	EPA 602 EPA 624 EPA 8021B EPA 8260B EPA 8260C EPA 524.2 (DW) EPA 6200B OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1	EPA 8021B EPA 8260B EPA 8260C OA-1
<b>Extractable Organics (Semivolatiles)</b>					
Organic Extraction	EPA 3540C EPA 3546 EPA 3550B EPA 3550C	-----	EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550B EPA 3550C
Acenaphthene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM
Acenaphthylene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270C SIM EPA 8270D EPA 8270D SIM
Acetic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Acetophenone	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Acetylaminofluorene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Alkylated PAHs	EPA 8270C SIM EPA 8270D SIM	-----	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM
4-Aminobiphenyl	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Amino-4,6-dinitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
4-Amino-2,6-dinitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
Aniline	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Anthracene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Atrazine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Benzaldehyde	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Benzidine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Benzoic Acid	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Benzo (a) Anthracene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Benzo (b) Fluoranthene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Benzo (k) Fluoranthene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Benzo (ghi) Perylene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Benzo (a) Pyrene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Benzo (e) Pyrene	EPA 8270C SIM EPA 8270D SIM	-----	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM
Benzyl Alcohol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Biphenyl	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Bis (2-chloroethoxy) Methane	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Bis (2-chloroethoxy) Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Bis (2-chloroethyl) Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Bis (2-chloroisopropyl) Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Bis (2-ethylhexyl) Phthalate	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
4-Bromophenylphenyl Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Butyl benzyl Phthalate	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Butyric Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Caprolactam	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Carbazole	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO)	-----	-----	EPA 8015B EPA 8015C EPA 8015D TN EPH	EPA 8015B EPA 8015C EPA 8015D TN EPH	EPA 8015B EPA 8015C EPA 8015D TN EPH
4-Chloroaniline	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
4-Chloro-3-methylphenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Chlorobenzilate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1-Chloronaphthalene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Chloronaphthalene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Chlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
4-Chlorophenyl Phenyl Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Chrysene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Citric Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Cresols (Methyl Phenols)	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
cis-/trans-Diallate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,4-Diamino-6-nitrotoluene	-----	-----	EPA 8330B	EPA 8330B	EPA 8330B
2,6-Diamino-4-nitrotoluene	-----	-----	EPA 8330B	EPA 8330B	EPA 8330B
Dibenzo (a,h) Acridine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Dibenzo (a,h) Anthracene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Dibenzofuran	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270D SIM
Dibenzothiophene	EPA 8270C SIM EPA 8270D SIM	-----	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
1,2-Dichlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,3-Dichlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,4-Dichlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
3,3-Dichlorobenzidine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)]	-----	-----	EPA 8015B EPA 8015C EPA 8015D CT ETPH MA EPH NWTPH DX NJ EPH TX1005/ TX1006 WADOE EPH OA-2	EPA 8015B EPA 8015C EPA 8015D CT ETPH  MA EPH NWTPH DX NJ EPH TX1005/ TX1006 WADOE EPH OA-2	EPA 8015B EPA 8015C EPA 8015D CT ETPH  MA EPH NWTPH DX NJ EPH TX1005/ TX1006 WADOE EPH OA-2
2,4-Dichlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,6-Dichlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Diethyl Phthalate	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Dimethoate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
p- Dimethylaminoazobenze ne	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
7,12-Dimethylbenz (a) Anthracene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
alpha-,alpha- Dimethylphenethylamine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,4-Dimethylphenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Dimethyl Phthalate	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
3,3-Dimethylbenzidine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Di-n-butyl Phthalate	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Di-n-octyl Phthalate	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
3,5-Dinitroaniline	-----	-----	EPA 8330B	EPA 8330B	EPA 8330B
1,3-Dinitrobenzene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B
2,4-Dinitrophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,4-Dinitrotoluene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B
2,6-Dinitrotoluene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B
1,4-Dioxane	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Diphenylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Diphenyl Ether	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,2-Diphenylhydrazine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Ethyl Methane Sulfonate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Fluoroanthene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Fluorene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Formic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Hexachlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Hexachlorobutadiene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Hexachlorocyclo- pentadiene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Hexachloroethane	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Hexachloropropene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Hexahydro-1,3,5- trinitro-1,3,5-triazine (RDX)	-----	-----	EPA 8330 EPA 8330A <u>EPA 8330B</u>	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
Indeno (1,2,3-cd) Pyrene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Isodrin	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Isophorone	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Isosafrole	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Isobutyric Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Lactic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Methapyriline	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
3-Methycholanthrene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Methyl-4,6-Dinitrophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Methyl Methane Sulfonate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1-Methylnaphthalene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
2-Methylnaphthalene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270C SIM EPA 8270D SIMEPA 8270D	EPA 8270C EPA 8270C SIM EPA 8270D SIM EPA 8270D
2-Methylphenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
4-Methylphenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Naphthalene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
1,4-Naphthoquinone	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1-Naphthylamine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Naphthylamine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
4-Nitroquinoline-1-oxide	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Nitroaniline	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
3-Nitroaniline	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
4-Nitroaniline	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Nitrobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B	EPA 8270C EPA 8270D EPA 8330 EPA 8330A EPA 8330B
Nitroglycerin	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
2-Nitrophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
4-Nitrophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Nitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
3-Nitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
4-Nitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
5-Nitro-o-toluidine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitroso-di-n-butylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosodiethylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosodimethylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosodimethylethylamine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosomorpholine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosodi-n-propylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
n-Nitrosodiphenylamine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosopiperidine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
n-Nitrosopyrrolidine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
Oxalic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
2,2-Oxybis (1-chloropropane)	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pentachlorobenzene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pentachloronitrobenzene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pentachlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pentaerythritol Tetranitrate (PETN)	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
Perylene	EPA 8270C SIM EPA 8270D SIM	-----	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM	EPA 8270C SIM EPA 8270D SIM
Petroleum Range Organics	-----	-----	FLPRO	FLPRO	FLPRO
Phenacetin	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Phenanthrene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Phenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,4-Phenylenediamine	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2-Picoline	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pronamide	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Propionic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Pyrene	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	-----	EPA 625 EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM	EPA 8270C EPA 8270D EPA 8270C SIM EPA 8270D SIM
Pyridine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Pyruvic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Quinic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Succinic Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Tartaric Acid	-----	-----	EPA 8015B EPA 8015D	EPA 8015B EPA 8015D	-----
Safrole	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,2,4,5- Tetrachlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,3,4,6- Tetrachlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Tetraethyl Dithiopyrophosphate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
Tetryl	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
Thionazin	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
o-Toluidine	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,3,5-Trinitrobenzene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
2,4,5-Trichlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,4,6-Trichlorophenol	EPA 8270C EPA 8270D	-----	EPA 625 EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
O,O,O-Tri-ethylphosphorothioate	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
1,3,5-Trinitrobenzene	EPA 8270C EPA 8270D	-----	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D	EPA 8270C EPA 8270D
2,4,6-Trinitrotoluene	-----	-----	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B	EPA 8330 EPA 8330A EPA 8330B
<b>Pesticides/Herbicides/ PCBs</b>					
Organic Extraction	EPA 3540C EPA 3546 EPA 3550B EPA 3550C	-----	EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550B EPA 3550C
Acifluorfen	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Aldrin	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Azinphos Methyl (Guthion)	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
alpha-BHC	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
beta-BHC	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
delta-BHC	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
gamma-BHC (Lindane)	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Bentazon	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Bolstar	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
alpha-Chlordane	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Chloramben	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Chlordane (technical)	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Chlorobenzilate	-----	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Chlorpyrifos	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Coumaphos	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
2,4-D	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
2,4'-DDD	EPA 8081A EPA 8081B	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
2,4'-DDE	EPA 8081A EPA 8081B	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
2,4'-DDT	EPA 8081A EPA 8081B	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Dalapon	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
4,4'-DDD	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
4,4'-DDE	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
4,4'-DDT	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Demeton-O	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Demeton-S	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Diallate	-----	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Diazinon	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
1,2-Dibromo-3- chloropropane (DBCP)	-----	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Dicamba	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
3,5-Dichlorobenzoic Acid	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Dichlorvos	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Dichloroprop	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Dieldrin	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Dinoseb	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Disulfoton	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Diuron	-----	-----	EPA 8321A	EPA 8321A	EPA 8321A
Endosulfan I (alpha)	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Endosulfan II (beta)	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Endosulfan Sulfate	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Endrin	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Endrin Aldehyde	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Endrin Ketone	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Ethion	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Ethoprop	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Fensulfothion	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Fenthion	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Fenuron	-----	-----	EPA 8321A	EPA 8321A	EPA 8321A
Gamma-chlordane	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Heptachlor	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Heptachlor Epoxide	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Hexachlorobenzene	EPA 8081A EPA 8081B	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Hexachlorocyclopentadiene	-----	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Isodrin	-----	-----	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Malathion	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
MCPA	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Merphos	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Methoxychlor	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Mevinphos	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Mirex	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Parathion Ethyl	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Parathion Methyl	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
PCB-1016 (Arochlor)	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1221	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1232	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1242	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1248	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1254	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1260	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1262	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB-1268	EPA 8082 EPA 8082A	-----	EPA 608 EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
Aroclor 5432	-----	-----	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
Aroclor 5442	-----	-----	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
Aroclor 5460	-----	-----	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A	EPA 8082 EPA 8082A
PCB Congeners (209)	EPA 1668	-----	EPA 1668	EPA 1668	EPA 1668
Pentachlorophenol (PCP)	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Phorate	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Picloram	-----	-----	EPA 8151A	EPA 8151A	EPA 8151A
Simazine	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
Stirophos (Tetrachlorvinphos)	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
2,4,5-T	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Tokuthion (Prothiofos)	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
2,4,5-TP (Silvex)	EPA 8151A	-----	EPA 8151A	EPA 8151A	EPA 8151A
Toxaphene	EPA 8081A EPA 8081B	-----	EPA 608 EPA 8081A EPA 8081B	EPA 8081A EPA 8081B	EPA 8081A EPA 8081B
Trichloronate	-----	-----	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B	EPA 8141A EPA 8141B
<b>Dioxins/Furans</b>					
2,3,7,8-TCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,7,8-TCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8-PeCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,4,7,8-PeCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8-PeCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8-HxCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,6,7,8-HxCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
2,3,4,6,7,8-HxCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8,9-HxCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8,-HxCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,6,7,8-HxCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,7,8,9-HxCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,6,7,8-HpCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,7,8,9-HpCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
1,2,3,4,6,7,8-HpCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
OCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
OCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HpCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HpCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total HxCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Air</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	
				<u>Aqueous</u>	<u>Solid</u>
Total HxCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total PeCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total PeCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total TCDD	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
Total TCDF	EPA 1613B EPA 8290A	-----	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A	EPA 1613B EPA 8290A
<b>Misc. Headspace Analysis</b>					
Carbon Dioxide	-----	-----	RSK-175	RSK-175	-----
Ethane	-----	-----	RSK-175	RSK-175	-----
Ethene	-----	-----	RSK-175	RSK-175	-----
Methane	-----	-----	RSK-175	RSK-175	-----
<b>Hazardous Waste Characteristics</b>					
Toxicity Characteristic Leaching Procedure	-----	-----	-----	EPA 1311	EPA 1311
Synthetic Precipitation Leaching Procedure	-----	-----	-----	EPA 1312	EPA 1312
<b>Other</b>					
Perchlorate	-----	-----	EPA 6850	EPA 6850	EPA 6850
Hydrazine	-----	-----	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Formaldehyde	-----	-----	-----	EPA 8315A	EPA 8315A
Methylhydrazine	-----	-----	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	-----	-----	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	-----	-----	EPA 5030A EPA 5030B	EPA 5030A EPA 5030B	EPA 5035 EPA 5035A
Organic Extraction	EPA 3540C EPA 3546 EPA 3550B EPA 3550C	-----	EPA 3510C EPA 3511	EPA 3510C EPA 3511	EPA 3540C EPA 3546 EPA 3550B EPA 3550C

<u>Parameter/Analyte</u>	<u>Tissue</u>	<u>Drinking Water</u>	<u>Nonpotable Water (*DW)</u>	<u>Solid Hazardous Waste</u>	<u>Parameter/Analyte</u>
<b>Perfluorinated Alkyl Acids (PFAAs)</b>					
Perfluorohexanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluoroheptanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorooctanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorononanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorodecanoic acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluoroundecanic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorododecanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorotridecanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorotetradecanoic Acid	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorobutanesulfonate	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorohexanesulfonate	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
Perfluorooctanesulfonate	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----
8:2 Fluorotelomersulfonate	-----	EPA 537 MOD	EPA 537 MOD	EPA 537 MOD	-----

\* **DW noted in parenthesis for drinking water method**