

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

ANALYSIS OF
CHLORINATED DIBENZO-P-DIOXINS (CDDs) AND
CHLORINATED DIBENZO FURANS (CDFs)

Multi-Media, Multi-Concentration

DLM01.1
August 2000

THIS PAGE INTENTIONALLY LEFT BLANK

STATEMENT OF WORK

TABLE OF CONTENTS

EXHIBIT A:	SUMMARY OF REQUIREMENTS
EXHIBIT B:	REPORTING AND DELIVERABLES REQUIREMENTS
EXHIBIT C:	TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS
EXHIBIT D:	ANALYTICAL METHODS
EXHIBIT E:	QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS
EXHIBIT F:	CHAIN-OF-CUSTODY, DOCUMENT CONTROL, AND WRITTEN STANDARD OPERATING PROCEDURES
EXHIBIT G:	GLOSSARY OF TERMS
EXHIBIT H:	DATA DICTIONARY AND FORMAT FOR DATA DELIVERABLES IN COMPUTER- READABLE FORMAT

THIS PAGE INTENTIONALLY LEFT BLANK

EXHIBIT A
SUMMARY OF REQUIREMENTS

THIS PAGE INTENTIONALLY LEFT BLANK

Exhibit A - Summary of Requirements

Table of Contents

<u>Section</u>	<u>Page</u>
1.0 PURPOSE	5
2.0 DESCRIPTION OF SERVICE	5
3.0 DATA USES	5
4.0 SUMMARY OF REQUIREMENTS	5
4.1 Introduction to the Dioxin/Furan Statement of Work	5
4.2 Overview of Major Task Areas	6
4.3 Technical and Management Capability	11

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 PURPOSE

The purpose of the multi-media, multi-concentration dioxin/furan analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

The dioxin/furan analytical service provides a contractual framework for laboratories to apply USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 17 2,3,7,8-substituted tetra- through octa-chlorinated dibenzo-p-dioxins (CDDs) and dibenzofurans (CDFs) in water, soil, sediment, sludge, tissue (no human tissue), ash, oil, and oily matrices. The analytical service provides the methods to be used and the specific contractual requirements by which the USEPA will evaluate the data. This service uses a High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS) method to analyze the target compounds.

3.0 DATA USES

This analytical service provides data which USEPA uses for a variety of purposes such as: determining the nature and extent of contamination at a hazardous waste site; assessing priorities for response based on risks to human health and the environment; determining appropriate clean-up actions; and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections; Hazard Ranking System (HRS) scoring; remedial investigation/feasibility studies; remedial design; treatability studies; and removal actions. In addition, this service provides data that are available for use in Superfund enforcement/litigation activities.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Dioxin/Furan Statement of Work

This Statement of Work (SOW) is designed as part of the documentation for a contract between USEPA and a commercial laboratory performing analyses in support of USEPA Superfund programs. The SOW is comprised of eight exhibits. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and instructions. Exhibit C specifies the CDD/CDF target compound list for this SOW with the contract required quantitation limits for the sample matrices. Exhibit D details the required analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), QA/QC performance, and the reporting of data. Exhibit F contains Chain-of-Custody and sample documentation requirements which the Contractor shall follow. To ensure proper understanding of the terms utilized in this SOW, a glossary can be found in Exhibit G. When a term is used in the text without explanation, the glossary meaning shall be applicable. Specifications for reporting data in computer-readable format appear in Exhibit H.

Exhibit A -- Section 4
Summary of Requirements (Con't)

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced.

4.2.1 Task I: Sample Receiving, Storage, and Disposal

4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper Chain-of-Custody. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete Sample Delivery Group File (CSF) (See Exhibit B). The Contractor shall establish and use appropriate procedures to handle confidential information received from USEPA.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor's facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). The Contractor shall communicate with SMO personnel by telephone, as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including weekends.

4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation and paperwork (e.g., Traffic Reports not with shipment, sample and Traffic Report do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional Office may include a sample shipping cooler temperature blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER TEMPERATURE INDICATOR.

4.2.1.2.3.1 When the USEPA Regional Office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.

- 4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler, and prior to unpacking the samples or removing the packing material.
- 4.2.1.2.3.3 To determine the temperature of the cooler, the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer ($\pm 1^{\circ}\text{C}$) shall have a measurable range of $0-50^{\circ}\text{C}$. Other devices which can measure temperature may be used if they can be calibrated to $\pm 1^{\circ}\text{C}$ and have a range of $0-50^{\circ}\text{C}$. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the Sample Delivery Group (SDG) Narrative.
- 4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C , the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the Region from which the samples were shipped for instruction on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA sample numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.
- 4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 - Cooler Temperature, and in the SDG Narrative.
- 4.2.1.2.4 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.
- 4.2.1.2.5 The Contractor is required to retain unused sample volume and used sample containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain all water/aqueous (preserved and unpreserved) and/or soil/sediment samples at 4°C ($\pm 2^{\circ}\text{C}$), and tissue samples at $< -10^{\circ}\text{C}$.
- 4.2.1.2.6 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling office within 14 calendar days following shipment receipt (see clause titled, "Government Furnished Supplies and Materials").

Exhibit A -- Section 4
Summary of Requirements (Con't)

4.2.1.3 The Contractor may be requested by USEPA to perform modified analyses. These modifications will be within the scope of the Exhibit D method for the analysis of the target compounds identified in Exhibit C of this SOW and may include, but are not limited to, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP Project Officer (CLP PO) and Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample analyses.

4.2.2 Task II: Sample Preparation and Analysis

4.2.2.1 Overview

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique USEPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

4.2.2.2.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- C Each Case of field samples received, or
- C Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- C Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- C In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time.

4.2.2.2.2 Samples may be assigned to SDGs by matrix (e.g., all soils in one SDG, all waters in another), at the discretion of the laboratory. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received, and shall not be made retroactively.

4.2.2.2.3 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report bearing the sample number and descriptive information regarding the sample. The Contractor shall complete and sign the Traffic

Report, recording the date of sample receipt and sample condition on receipt for each sample container.

- 4.2.2.2.4 The Contractor shall submit signed copies of Traffic Reports for all samples in an SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of Traffic Reports do not meet this requirement. Traffic Reports shall be submitted in SDG sets (e.g., all Traffic Reports for an SDG shall be clipped together) with an TR Cover Sheet containing information regarding the SDG, as specified in Exhibit B.
- 4.2.2.2.5 USEPA Case numbers, SDG numbers, and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract, both verbally and in reports/correspondence.
- 4.2.2.3 Preparation Techniques: The Contractor shall prepare samples, as described in Exhibit D.
- 4.2.2.3.1 If multiphase samples (e.g., a two-phase liquid sample) are received by the Contractor, the Contractor must contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the Region may require the Contractor to do the following:
- C Mix the sample and analyze an aliquot from the homogenized sample.
 - C Separate the phases of the sample and analyze each phase individually. SMO will provide EPA sample numbers for the additional phases.
 - C Separate the phases and analyze one or more of the phases, but not all of the phases. SMO will provide EPA sample numbers for the additional phases, if required.
 - C Do not analyze the sample.
- 4.2.2.3.2 If insufficient sample volume (less than the required amount) is received to perform the analysis, the Contractor shall contact SMO to apprise them of the problem. SMO will contact the Region for instructions. The Region will either approve that no sample analysis be performed, or require that a reduced volume be used for the sample analysis. No other changes in the analysis will be permitted. SMO will notify the Contractor of the Region's decision. The Contractor shall document the Region's decision in the SDG Narrative.
- 4.2.2.4 Analytical Techniques: The Target Compounds listed in Exhibit C shall be identified, as described in the methodologies given in Exhibit D. Automated computer programs may be used to facilitate the identification of compounds.
- 4.2.2.5 Qualitative Verification of Compounds: The dioxin and furan compounds identified by HRGC/HRMS techniques shall be verified by an analyst competent in the interpretation of mass spectra. The analyst will compare the HRGC retention time and ion-abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard and the theoretical ion-abundance ratio of the two exact m/z's.

Exhibit A -- Section 4
Summary of Requirements (Con't)

- 4.2.2.5.1 If a compound initially identified by HRGC/HRMS techniques cannot be verified, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, the Contractor shall report that identification as an "estimated maximum possible concentration" (EMPC) and proceed with quantitation.
- 4.2.2.6 Quantitation of Verified Compounds. The Contractor shall quantitate components identified by HRGC/HRMS techniques using Selected Ion Current Profile (SICP) areas in one of the ways described in Exhibit D, Section 2.3.
- 4.2.2.7 Quality Assurance/Quality Control Procedures
- 4.2.2.7.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibits B and H.
- 4.2.2.7.2 The Contractor shall maintain a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection, as well as the quality assessment measures performed by management to ensure acceptable data production.
- 4.2.2.7.3 Additional QC shall be conducted in the form of the analysis of laboratory evaluation samples submitted to the laboratory by USEPA. Unacceptable results of all such QC or laboratory evaluation samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values, as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D), QA/QC (Exhibit E), data reporting and other deliverables (Exhibits B and H), and sample custody, sample documentation, and SOP documentation (Exhibit F). As an alternative to data rejection, USEPA may require re-analysis of noncompliant samples. Re-analysis will be performed by the Contractor at no additional cost to USEPA, unless it is determined that the laboratory evaluation sample(s) was defective.
- 4.2.3 Task III: Sample Reporting and Resubmission of Data
- 4.2.3.1 USEPA has provided, to the Contractor, formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets and computer-readable data on diskette (or via an alternate means of electronic transmission approved in advance by USEPA) in a format specified in this SOW and within the time specified in Exhibit B, Section 1.1.
- 4.2.3.2 Use of formats other than those designated by USEPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format, at no additional cost to the Government, shall be required.

- 4.2.3.3 Computer-generated forms may be submitted in the hardcopy Sample Data Package(s), provided that the forms are in **exact USEPA format**. This means that the order of data elements is the same as on each USEPA-required form, including form numbers and titles, page numbers, and header information.
- 4.2.3.4 If the submitted data package does not conform to the specified contractual or technical criteria, the Contractor will be required to resubmit the data package with all deficiencies corrected at its own expense. The Contractor will respond within seven (7) days to requests for additional information or explanations that result from the Government's inspection activities. If the Contractor is required to submit or resubmit data as a result of a Regional request, the data shall be clearly marked as ADDITIONAL DATA. The Contractor shall include a cover letter which describes which data are being delivered, to which USEPA project the data pertain, and who requested the data. Any and all resubmissions must be in accordance with the documentation requirements of this SOW.
- 4.2.3.5 The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the hardcopy forms or the computer-readable data, or both sets of data, at no additional cost to USEPA.
- 4.2.3.6 In addition, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, since it is used to make major decisions regarding public health and environmental welfare. The data may also be used in litigation against Potentially Responsible Parties in the enforcement of Superfund legislation.

4.3 Technical and Management Capability

4.3.1 Personnel

The Contractor shall have adequate personnel at all times during the performance of the contract to ensure that USEPA receives data that meet the terms and conditions of the contract. This includes personnel available to process samples at any time delivery service is operating, including weekends, to ensure that sample analysis time requirements can be met.

4.3.2 Instrumentation

The Contractor shall have a sufficient High-Resolution Gas Chromatograph/Mass Spectrometer/Data System (HRGC/HRMS/DS), including magnetic tape storage devices, and a Gel Permeation Chromatography system (GPC) capability to meet all the terms and conditions of the contract.

4.3.3 Facilities

The Contractor shall maintain a facility suitable for the receipt, storage, analysis, and delivery of the product meeting the terms and conditions of the contract.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

THIS PAGE INTENTIONALLY LEFT BLANK

Exhibit B - REPORTING AND DELIVERABLES REQUIREMENTS

Table of Contents

<u>Section</u>	<u>Page</u>
1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION	5
1.1 Report Deliverable Schedule	5
1.2 Distribution	8
2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES	9
2.1 Introduction	9
2.2 Resubmission of Data	9
2.3 Quality Assurance (QA) Plan and Standard Operating Procedures (SOPs)	10
2.4 Sample Traffic Reports	10
2.5 Sample Data Package	10
2.6 Complete SDG File	15
2.7 Data in Computer-Readable Format	16
2.8 HRGC/HRMS Tapes	16
2.9 Extracts	17
3.0 FORM INSTRUCTIONS	19
3.1 Introduction	19
3.2 General Information	19
3.3 Header Information	19
3.4 CDD/CDF Data Reporting Forms	21
3.5 Sample Log-In Sheet [Form DC-1]	30
3.6 CDD/CDF Complete SDG File (CSF) Inventory Sheet [Form DC-2]	31
4.0 DATA REPORTING FORMS	32

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table reiterates the contract reporting and deliverables requirements and specifies the distribution that is required for each deliverable.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The USEPA Office of Emergency and Remedial Response (OERR) Analytical Operations/Data Quality Center (AOC) Dioxin Program Manager will notify the Contractor, in writing, of such changes when they occur.

TABLE 1

Item	No. of Copies ^A	Delivery Schedule	Distribution			
			SMC	Reg	AOC	CLF PO#
A.	Sample Traffic Reports	1	3 working days after receipt of last sample in SDG ² .	X		
B. ³	Sample Data Package ^C	2	35 days after VTSR ¹ of last sample in SDG.	X		
C. ³	Data in Computer-Readable Format	1	35 days after VTSR ¹ of last sample in SDG.	X	X	
D. ³	Results of Intercomparison Study/PE Sample Analysis Study	1	35 days after VTSR ¹ of last sample in SDG.	X		
E. ^{3,4}	Complete SDG File ³	1	35 days after VTSR ¹ of last sample in SDG.		X	
F. ⁵	Quality Assurance Plan	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.	As Directed		

TABLE 1 (Con't)

Item		No. of Copies ^A	Delivery Schedule	Distribution ^B			
				SNO	Region	AOC PM	CLP PO
G. ⁵	Updated Standard Operating Procedures	1	Revise within 60 days after contract award. Submit within 7 days of receipt of written request to recipients as directed.	As Directed			
H.	GC/MS Tapes	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request.	As Directed			
I.	Extracts	Lot	Retain for 365 days after data submission. Submit within 7 days after receipt of written request by the USEPA Regional CLP Project Officer.	As Directed			

Footnotes:

^AThe number of copies specified is the number of copies required to be delivered to each recipient.

^BContract Laboratory Program Project Officer (CLP PO).

^CContractor-concurrent delivery to the USEPA's designated recipient (e.g., QATS) may be required upon request by the USEPA AOC Dioxin Program Manager. Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the USEPA AOC Dioxin Program Manager.

¹Validated time of sample receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report.

²A sample delivery group (SDG) is a group of samples within a Case, received over a period of 7 calendar days or less and not exceeding 20 samples (excluding PE samples). Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

³DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or of any sample within the SDG, is the date all samples have been delivered. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time shall be considered late.

⁴Complete SDG file will contain the original Sample Data Package, plus all the original documents described in Exhibit B, Section 2.6 and Exhibit E.

⁵See Exhibit E and F for more description, time is cited in calendar days.

NOTE: As specified in the Contract Schedule (Government Furnished Supplies and Materials), unless otherwise instructed by SMO based on a Regional decision, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than 60 days following submission of reconciled analytical data. Sample disposal and disposal of unused sample bottles/containers is the responsibility of the Contractor, and should be done in accordance with all applicable laws and regulations governing disposal of such materials.

Exhibit B -- Section 1
Contract Reports/Deliverables Distribution (Con't)

1.2 Distribution

The following addresses correspond to the "Distribution" column in Table 1 of Section 1.1.

SMO:USEPA Contract Laboratory Program (CLP)
Sample Management Office (SMO)⁶
2000 Edmund Halley Drive
Reston, VA 20191-3436

Region: USEPA REGIONS: SMO will provide the Contractor with the list of addressees for the 10 USEPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

USEPA AOC Dioxin Program Manager:

Mailing Address: USEPA OERR Analytical Operations/
Data Quality Center
Ariel Rios Building (5204G)
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460
Attn: CLP Dioxin Program Manager

Fed-Ex/Overnight Delivery: USEPA OERR Analytical Operations/
Data Quality Center
1235 Jefferson Davis Highway
Crystal Gateway I, 12th Floor
Arlington, VA 22202
Attn: CLP Dioxin Program Manager

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the USEPA Regional CLP POs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

QATS: USEPA Contract Laboratory Program (CLP)
Quality Assurance Technical Support (QATS) Laboratory⁷
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

⁶The SMO is a Contractor-operated facility operating under the CLASS contract awarded and administered by USEPA.

⁷The QATS laboratory is a Contractor-operated facility operating under the QATS contract awarded and administered by USEPA.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor will provide reports and other deliverables as specified in the Contract Schedule. The required content and form of each deliverable are described in this exhibit. All reports and documentation must be:

- C Legible;
- C Clearly labeled and completed in accordance with instructions in this exhibit;
- C Arranged in the order specified in this section;
- C Paginated sequentially in ascending order starting from the SDG Narrative, and
- C Copies must be legible and double-sided.

NOTE: Complete SDG Files (CSFs) need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to the USEPA Contract Laboratory Program (CLP) Sample Management Office (SMO), and USEPA-designated recipients, [e.g., Quality Assurance Technical Support (QATS)], upon written request must be double-sided.

2.1.1 Requirements for each deliverable item cited in the Contract Schedule (Contract Performance/Delivery Schedule) are specified in Sections 2.3 through 2.9. Prior to submission, the Contractor shall arrange items and the components of each item in the order listed in these sections.

2.1.2 The Contractor shall use EPA Case numbers, Sample Delivery Group (SDG) numbers, EPA Sample numbers, and Client numbers (if applicable) to identify samples received under this contract, both verbally and electronically and in reports and correspondence. The Contract number shall be specified in all correspondence.

2.2 Resubmission of Data

2.2.1 If submitted documentation does not conform to the instructions in this exhibit, the Contractor will be required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the USEPA.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of an onsite laboratory evaluation, or through a USEPA Region CLP PO action, or through a Regional data reviewer's request, the data must be clearly marked as ADDITIONAL DATA and must be sent to both contractual data recipients (SMO, and the Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. A cover letter will be included, by the Contractor, describing what data are being delivered, to which EPA project the data pertain, and who requested the data.

2.2.3 Whenever the Contractor is required to submit or resubmit data as a result of contract compliance screening by SMO, the data must be sent to the two contractual data recipients (SMO, and the Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. In all instances, the data

must be accompanied by a color-coded Cover Sheet (Laboratory Response to Results of Contract Compliance Screening) provided by SMO. Electronic Deliverables shall be submitted or resubmitted to SMO and the Region. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the Region.

2.3 Quality Assurance (QA) Plan and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports

2.4.1 Each sample received by the Contractor will be labeled with an EPA Sample number and will be accompanied by a Sample Traffic Report (TR) bearing the sample number and descriptive information regarding the sample. The Contractor shall complete the TR (marked "Lab Copy for Return to SMO"), recording the date of sample receipt and sample condition upon receipt for each container, and shall sign the TR. Information shall be recorded for each sample in the SDG.

2.4.2 The contractor shall submit TRs in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an TR cover sheet attached. The TR cover sheet shall contain the following items:

C Laboratory name;

C Contract number;

C Sample analysis price (full sample price from the contract);

C Case number; and

C List of EPA sample numbers of all samples in the SDG, identifying the **first** and **last** samples received, and their Laboratory Receipt Dates (LRDs).

2.4.3 Each TR must be clearly marked with the SDG number. This information should be entered below the laboratory receipt date on the TR. In addition, the TR for the last sample received in the SDG must be clearly marked "SDG - FINAL SAMPLE". The EPA sample number of the first sample received in the SDG is the SDG number. When several samples are received together in the first SDG shipment, the SDG number will be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.

2.4.4 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the Contractor must make the appropriate number of photocopies of the TR and submit one copy with each TR Cover Sheet.

2.5 Sample Data Package

The Sample Data Package will include data for analyses of all samples in one SDG, including field samples, dilutions, re-analyses, blanks, laboratory control samples, and laboratory control sample duplicates. The Sample Data Package is divided into the three major units (SDG Narrative, Traffic Reports, and CDD/CDF data) described below. The Contractor will retain a copy of the Sample Data Package for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

2.5.1 SDG Narrative

2.5.1.1 This document will be clearly labeled "SDG Narrative" and will contain: laboratory name; Case number; EPA sample numbers, differentiating between initial analyses and re-analyses; SDG number; Contract number; Client number (if applicable); and detailed documentation of any quality control, samples, shipment and/or analytical problems encountered in processing the samples reported in the data package.

All GC columns used for analysis shall be documented in the SDG Narrative. List the GC Column identification - brand-name, internal diameter in mm, and length in meters, coating material, and film thickness.

NOTE: If a column is used that has different first and last eluting isomers than the DB-5 column, the Contractor will fully document, in the SDG Narrative, the order of elution of the isomers and identify the first and last eluting isomers for that particular column for the window defining mix and CS3 Solution.

2.5.1.2 Whenever data from sample re-analyses are submitted, the Contractor will state the reason in the SDG Narrative for each re-analysis. The Contractor must also include any problems encountered, both technical and administrative, the corrective actions taken and the resolutions, and an explanation for all flagged edits (i.e., manual edits) on quantitation lists. This includes documenting the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations or curves to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any requested SOW modifications. This includes attaching a copy of the approved modification form to the SDG Narrative.

2.5.1.3 The SDG Narrative will contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or his/her designee, as verified by the following signature." This statement will be directly followed by the original signature of the Laboratory Manager or his/her designee with a typed line below it containing the signer's name and title, and the date of signature. All copies of the SDG Narrative will be signed in an original signature.

2.5.2 Traffic Reports

2.5.2.1 The Contractor shall include a copy of the TRs submitted in Section 2.4 for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample numbering order, considering both letters and numbers in ordering samples. Copies of the TR Cover Sheet will be included with the copies of the TRs.

2.5.2.2 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the Contractor must make the appropriate number of photocopies of the TR so that a copy is submitted with each applicable data package.

Exhibit B -- Section 2

Reporting Requirements and Order of Data Deliverables (Con't)

2.5.2.3 In any instance where samples from more than one multi-sample TR are in the same data package, the Contractor must submit a copy of the TR Cover Sheet with copies of the TRs.

2.5.3 CDD/CDF Data

2.5.3.1 CDD/CDF Sample Data

Sample data shall be arranged in packets with the CDD/CDF Sample Data Summary (Form I-HR CDD-1 and Forms I-HR CDD-2, CDD-3, if applicable), followed by the raw data for the sample and Form II-HR CDD. These sample packets shall be placed in order of increasing EPA Sample Number, considering both letters and numbers.

2.5.3.1.1 Sample Data Summary (Form I-HR CDD-1)

Tabulated results (identification and quantification) of the specified target analytes and recoveries of the associated labeled compounds shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.5.1.3). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.5.3.1.2 Toxicity Equivalence Summary (Form I-HR CDD-2)

Tabulated adjusted concentrations for the target analytes based on toxicity equivalent factors. This form shall be included, even if no target analytes are positively identified.

2.5.3.1.3 Second Column Confirmation (Form I-HR CDD-3)

Tabulated results (identification and quantitation) of 2,3,7,8-TCDD, 2,3,7,8-TCDF and the recoveries of their corresponding labeled compounds on a second GC column if original analysis was performed on a DB-5 GC column, or equivalent.

2.5.3.1.4 Selected Ion Current Profile (SICP) for each sample or sample extract, including dilutions and re-analyses.

SICPs must be presented so the two quantitation ions and any relevant labeled compounds, internal standards, or diphenyl ether interferents are on one page. The SICP must show the full time window scanned for each ion. Enlarge any SICP peak for any 2,3,7,8-substituted congener present below the signal to noise ratio of 10 or below the Contract Required Quantitation Limit (CRQL). Each SICP must contain the following header information:

- C EPA sample number;
- C Date and time of analysis;
- C RT (and scan number if available) of identified compounds;
- C HRGC/HRMS instrument ID;

C Lab File ID, and

C Analyst ID.

2.5.3.1.5 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report, including but not limited to quantitation reports and area summaries, shall be provided in all sample data packages, in addition to the SICPs. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the sample data package, in addition to the SICP:

C EPA sample number;

C Date and time of analysis;

C RT (and scan number if available) of identified target compounds;

C Ions used for quantitation with measured areas;

C Copy of area table from data system;

C HRGC/HRMS instrument ID;

C Lab File ID; and

C Analyst ID.

2.5.3.1.6 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the HRGC/HRMS operator shall identify the changes made to the report, by initialing and dating all handwritten changes, and shall include the integration scan range. In addition, a hardcopy printout of the chromatogram displaying the manual integration shall be included in the raw data.

NOTE: Second column confirmation is required for all samples in which 2,3,7,8-TCDF/TCDD is positively identified at, or greater than, the CRQL by analysis on a DB-5 (or equivalent) HRGC column, or if 2,3,7,8-TCDF/TCDD is reported as an Estimated Maximum Possible Concentration (EMPC) at, or greater than, the CRQL.

2.5.3.1.7 Total Homologue Concentration Summary (Form II-HR CDD)

Tabulated total homologue concentrations shall be completed for each sample, blank, and QC sample analyzed. EMPC values will be flagged "*" and Estimated Detection Limit (EDL) will be qualified "U" on the form.

2.5.3.2 Quality Control Data

2.5.3.2.1 Lab Control Sample Summary (Form III-HR CDD-1) - in order by EPA Sample Number assigned to the LCS.

2.5.3.2.2 Lab Control Sample Duplicate Summary (Form III-HR CDD-2) - in order by EPA Sample Number.

Exhibit B -- Section 2

Reporting Requirements and Order of Data Deliverables (Con't)

- 2.5.3.2.3 Method Blank Summary (Form IV-HR CDD) - in order by EPA Sample Number assigned to the blanks.
- 2.5.3.2.4 Window Defining Mix Summary (Form V-HR CDD-1) - in order by EPA Sample Number assigned to the window defining mix.

Window Defining Mix Summary must be completed for each 12-hour period. The retention time for the first and last eluting CDD and CDF isomers are included on this form.
- 2.5.3.2.5 Chromatographic Resolution Summary (Form V-HR CDD-2) - in order by EPA sample number assigned to the standard used to evaluate the column resolution.

Chromatographic Resolution Summary must be completed for each 12-hour period.
- 2.5.3.2.6 Analytical Sequence Summary (Form V-HR CDD-3) - This form is used to report the analytical sequence for CDD/CDF analysis for all GC columns and instruments.
- 2.5.3.3 Calibration Data
 - 2.5.3.3.1 Initial Calibration Data (Form VI-HR CDD-1, CDD-2) - in order by instrument, if more than one instrument is used.
 - 2.5.3.3.1.1 Perfluorokerosene (PFK) mass resolution for initial calibration shall be provided and labeled with EPA sample number, date and time, HRGC/HRMS instrument ID, Lab File ID, and analyst ID.
 - 2.5.3.3.1.2 CDD/CDF standard(s) SICPs and complete data system reports including area summaries for the initial (five-point) calibration will be labeled as stated in Sections 2.5.3.1.4 and 2.5.3.1.5.
 - 2.5.3.3.1.3 When more than one initial calibration is performed, the data must be arranged in chronological order by instrument.
 - 2.5.3.3.2 Continuing Calibration Data (Form VII-HR CDD-1, CDD-2) - in order by instrument, if more than one instrument is used.
 - 2.5.3.3.2.1 PFK mass resolution for continuing calibration shall be provided for each 12-hour period and labeled with EPA sample number, date and time, HRGC/HRMS instrument ID, Lab File ID, and analyst ID.
 - 2.5.3.3.2.2 CDD/CDF standard(s) SICPs and complete data system reports including area summaries for all continuing calibrations will be labeled as specified in Sections 2.5.3.1.4 and 2.5.3.1.5.
 - 2.5.3.3.2.3 When more than one continuing calibration is performed, the data must be arranged in chronological order, by instrument.
 - 2.5.3.3.2.4 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the HRGC/HRMS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan page. In addition, a hardcopy printout of the chromatogram of the quantitation ion(s) displaying the manual integration

Exhibit B -- Section 2

Reporting Requirements and Order of Data Deliverables (Con't)

shall be included in the raw data. This applies to all compounds listed in Exhibit C, labeled compounds, and internal standards.

2.5.3.4 Raw Quality Control Data

2.5.3.4.1 Blank Data shall be included in order by EPA Sample Number assigned to the blank.

C FORM I-HR CDD-1, CDD-2, and CDD-3, if applicable.

C SICPs and a complete data system report including area summaries shall be submitted for each blank analyzed and labeled as specified in Sections 2.5.3.1.4 and 2.5.3.1.5.

2.5.3.4.2 Lab Control Sample Data

C Tabulated results (FORM I-HR CDD-1 and CDD-2).

C SICPs and a complete data system report including area summaries labeled as specified in Sections 2.5.3.1.4 and 2.5.3.1.5.

2.5.3.4.3 Lab Control Sample Duplicate Data

C Tabulated results (FORM I-HR CDD-1 and CDD-2).

C SICPs and a complete data system report including area summaries labeled as specified in Sections 2.5.3.1.4 and 2.5.3.1.5.

2.6 Complete SDG File

2.6.1 Complete SDG File (CSF), including the original Sample Data Package, will be delivered to the Region concurrently with delivery of the Sample Data Package to SMO. The contents of the CSF will be numbered according to the specifications described in Sections 3.6. The CSF will contain all original documents specified in Sections 3 and 4 and in Form DC-2. No copies will be placed in the CSF unless the originals were initially written in a bound notebook maintained by the laboratory, or the originals were previously submitted to the USEPA with another SDG in accordance with the requirements described in Exhibit F.

2.6.2 The CSF will consist of the following original documents, in addition to the documents in the Sample Data Package:

C Original Sample Data Package;

C A completed and signed CDD/CDF Complete SDG File (CSF) Inventory Sheet (Form DC-2);

C All original shipping documents including, but not limited to, the following:

- USEPA Chain-of-Custody Record;
- Airbills (if an airbill is not received, include a hardcopy receipt from the shipping company or a printout of the shipping company's electronic tracking information);
- USEPA (SMO) Traffic Reports, and
- Sample tags (if present) sealed in plastic bags.

- C All original receiving documents including, but not limited to, the following:
 - Form DC-1;
 - Other receiving forms or copies of receiving logbooks, and
 - TR Cover Sheet.

- C All original laboratory records not already submitted in the sample data package of sample transfer, preparation, and analysis including, but not limited to, the following documents:
 - Original preparation and analysis forms or copies of preparation and analysis logbook pages;
 - Internal sample and sample extract transfer chain-of-custody records;
 - Screening records, and
 - All instrument output, including strip charts from screening activities.

- C All other original SDG-specific documents in the possession of the Contractor, including, but not limited to, the following documents:
 - Telephone contact logs;
 - Copies of personal logbook pages;
 - All hand-written SDG-specific notes, and
 - Any other SDG-specific documents not covered by the above.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other SDG-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA (original to the Region and copies to SMO).

- 2.6.3 If the Contractor does submit SDG-specific documents to USEPA after submission of the CSF, the documents should be identified with unique accountable numbers, a revised Form DC-2 should be submitted, and the unique accountable numbers and locations of the documents in the CSF should be recorded in the "Other Records" section on the revised Form DC-2. Alternatively, the Contractor may number the newly submitted SDG-specific documents to USEPA as a new CSF and submit a new Form DC-2. The revised Form DC-2 or new Form DC-2 should be submitted to the USEPA Regions only.

2.7 Data in Computer-Readable Format

The Contractor shall provide a computer-readable copy of all sample data, as specified in Exhibit H, and delivered as specified in Section 1 of this exhibit. Computer-readable data deliverables shall be submitted on an IBM or IBM compatible formatted 3.5-inch high-density 1.44 M.byte diskette(s) (or via an alternative means of electronic transmission, if approved in advance by USEPA). When submitted, diskette(s) shall be packaged and shipped in such a way that the diskette(s) cannot be bent or folded and will not be exposed to extreme heat/cold or any type of electromagnetic radiation. The diskette(s) shall be included in the same shipment as the hardcopy data, and, at a minimum, shall be enclosed in a diskette mailer. The data shall adhere to the specifications listed in Exhibit H.

2.8 HRGC/HRMS Tapes

- 2.8.1 The Contractor must store all raw and processed HRGC/HRMS data on magnetic media in the appropriate instrument manufacturer's format. This media must include data for samples, LCS/LCSD blanks, initial and continuing calibrations, as well as all laboratory-generated

quantitation reports and SICPs required to generate the data package.

The Contractor will maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, and blanks. The logbook should include EPA sample numbers and standard and blank IDs, identified by Case, Client number, and SDG. The Contractor is required to retain the HRGC/HRMS tapes for three years after submission of the reconciled complete data package. During that time, the Contractor will submit tapes and associated logbook pages within seven days after receipt of a written request from the USEPA Regional CLP PO.

2.8.2 When submitting HRGC/HRMS tapes to USEPA, the following materials must be delivered in response to the request:

- C All associated raw data files for samples, blanks, QC samples, LCS, LCSD, initial and continuing calibration standards;
- C All processed data files and quantitation output files associated with the raw data files described above;
- C All associated identifications and calculation files used to generate the data submitted in the data package; and
- C A copy of the Contractor's written reference logbook relating tape files to EPA sample number, calibration data, standards, blanks, LCS, and LCSD. The logbook must include EPA sample numbers and lab file identifiers for all samples, blanks, and standards, identified by Case and SDG.

2.8.3 The laboratory must also provide a statement attesting to the completeness of the HRGC/HRMS data tape submission, signed and dated by the Laboratory Manager. This statement must be part of a cover sheet that includes the following information relevant to the magnetic media submission:

- C Laboratory name;
- C Date of submission;
- C Case number;
- C Client number (if applicable);
- C SDG number;
- C HRGC/HRMS make and model number;
- C Software version;
- C Disk drive type (e.g., CDC, PRIAM);
- C File transfer method (e.g., DSD, DTD, FTP, Aquarius); and
- C Names and telephone numbers of two laboratory contacts for further information regarding the submission.

2.9 Extracts

2.9.1 The Contractor will preserve sample extracts in the dark at room temperature in bottles/vials with Teflon-lined septa. Extract bottles/vials will be labeled with the EPA sample number, Case number, SDG number, and Client number (if applicable). A logbook of

Exhibit B -- Section 2

Reporting Requirements and Order of Data Deliverables (Con't)

stored extracts, listing EPA sample numbers and associated Case and SDG numbers, will be maintained.

- 2.9.2 The Contractor is required to retain extracts for 365 days following submission of reconciled complete data package. During that time, the Contractor will submit extracts and associated logbook pages within seven days following receipt of a written request from the USEPA Regional CLP PO.

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section contains specific instructions for completion of all required CDD/CDF Data Reporting Forms.

3.2 General Information

- 3.2.1 The data reporting forms presented in Exhibit B, Section 4.0 have been designed in conjunction with the computer-readable data formats specified in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format". The specific length of each variable for computer-readable data transmission purposes is given in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID".

NOTE: On the hardcopy forms, the space provided for entries is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (see Exhibit H). Greater space is provided on the hardcopy forms for visual clarity.

- 3.2.2 All characters which appear on the data reporting forms presented in Section 4 must be reproduced by the Contractor when submitting data, and the format of the forms submitted must be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the USEPA Regional CLP PO. The names of the various fields and compounds (e.g., "Lab Code", "2378-TCDD") must appear as they do on the forms in the contract, including the options specified in the form (i.e., "Matrix: (Soil, Water, Ash, Tissue, Oil)" must appear, not just "Matrix"). For items appearing on the uncompleted forms (Section 4), the use of uppercase and lowercase letters is optional.
- 3.2.3 Alphabetic entries made onto the forms by the Contractor will be in ALL UPPERCASE letters (e.g., "SOIL", not "Soil" or "soil"). If an entry does not fill the entire blank space provided on the form, null characters will be used to remove the remaining underscores that comprise the blank line.

3.3 Header Information

Six pieces of information are common to the header section of each data reporting form. They are Lab Name, Contract, Lab Code, Case No., Client No., and SDG No. Except as noted below for Client No., this information must be entered on every form and must match on every form.

- 3.3.1 "Lab Name" will be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.
- 3.3.2 "Lab Code" is an alpha-numeric abbreviation of up to six letters and numbers assigned by USEPA to identify the laboratory and aid in data processing. This lab code will be assigned by USEPA at the time a contract is awarded and will not be modified by the Contractor, except at the direction of USEPA. If a change of name or ownership occurs at the laboratory, the lab code will remain the same unless and until the Contractor is directed by USEPA to use another lab code assigned by USEPA.

Exhibit B -- Section 3
Form Instructions (Con't)

- 3.3.3 "Case No." is the USEPA-assigned Case number associated with the sample and reported on the Traffic Report or sample shipping paperwork.
- 3.3.4 "Contract" is the number of the USEPA contract under which the analyses were performed.
- 3.3.5 "SDG No." is the USEPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number will be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.
- 3.3.6 The "Client No." is a unique number identifying the client and the project. This number may be the USEPA-assigned number for analyses performed under Non-Routine Analytical Services (NRAS).
- 3.3.7 The "EPA Sample Number" is the other information common to most of the forms. This number appears either in the upper right-hand corner of the form, or as the left column of a table summarizing data from a number of samples. When the "EPA Sample No." is entered into the triple-spaced box in the upper right-hand corner of any form, it should be entered on the middle line of the three lines that comprise the box.
- 3.3.7.1 All samples, LCS, LCSD, blanks and standards will be identified with an EPA sample number. For field samples, the EPA sample number is based on the unique identifying number given in the Traffic Report or sample shipping records for that sample.
- 3.3.7.2 In order to facilitate data assessment, the following suffixes must be used:
- DXXXXX = EPA sample number
DXXXXXRE = Re-extracted and re-analyzed aliquot of sample "DXXXXX"
DXXXXXDL = Diluted analysis of sample "DXXXXX"
- 3.3.7.3 Form V-HR CDD-3 requires that all samples analyzed in a given 12-hour analytical sequence be listed, regardless of whether or not they are part of the SDG being reported, and regardless of whether or not they are EPA samples. Therefore, use "ZZZZZ" as the EPA sample number for any sample analysis that is not associated with the SDG being reported.
- 3.3.7.4 For blanks and standards, the following identification scheme must be used as the "EPA Sample No.":
- C Method blanks will be identified as DFBLK##;
- C Calibration standards will be identified as CS1##, CS2##, CS3##, CS4## and CS5## and will correspond to the calibration solutions identified in Exhibit D;
- C The window defining mixture will be identified as WDM##;
- C The isomer specificity check will be identified as ISC##;
- C If combined, the WDM and ISC will be identified as CPS##;
- C The LCS will be identified as DLCS##;
- C The LCSD will be identified as DLCSD##, and

C The PFK mass resolution check will be identified as PFK##.

3.3.7.5 "EPA Sample No." must be unique within an SDG. Therefore, the Contractor must replace the two-character "##" terminator of the identifier with one or two characters or numbers, or a combination of both, to create a unique EPA sample number for each blank and standard within the SDG. For example, possible identifiers for method blanks would be DFBLK1, DFBLK2, DFBLKA1, DFBLKB2, DFBLKAB, etc.

3.3.8 Other Common Fields

Other pieces of information are common to many of the data reporting forms. These include "Matrix", "Lab Sample ID", "Lab File ID", "Instrument", and "GC Column".

3.3.8.1 For "Matrix", enter "SOIL" for a soil/sediment/sludge sample, "WATER" for an aqueous sample, "TISSUE" for tissue (non-human), "OIL" for oil and oil matrix, and "ASH" for fly ash samples.

3.3.8.2 "Lab Sample ID" is an optional laboratory-generated internal identifier. Up to 12 alpha-numeric characters may be reported here. If the Contractor does not have a lab sample ID, this field may be left blank. However, if this identifier is used on any of the forms or accompanying hardcopy data deliverables, it must be reported on all the appropriate forms.

3.3.8.3 "Lab File ID" is the laboratory-generated name of the HRGC/HRMS data system file containing information pertaining to a particular analysis. Up to 14 alpha-numeric characters may be used here.

3.3.8.4 "Instrument ID" is common to many of the forms, particularly those containing calibration data. The identifier used by the laboratory must include some indication of the manufacturer and/or model of the instrument, and contain additional characters or numbers that differentiate between all instruments of the same type in the laboratory. The instrument identifier must be consistent on all forms within the SDG.

3.3.8.5 "GC Column" and "ID (mm)" are common to various other forms. These two fields are to be used to identify the stationary phase of the GC column, and the internal diameter of the GC column in millimeters (mm).

3.3.9 Rounding Rule

For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up).

3.4 CDD/CDF Data Reporting Forms

3.4.1 CDD/CDF Sample Data Summary [Form I-HR CDD-1]

3.4.1.1 This form is used for tabulating and reporting the sample analysis results for target analytes in samples, blanks, LCS, and LCSD. It is related to Form I-HR CDD-2, and for each sample for which there is a Form I-HR CDD-1, there must be a corresponding Form I-HR CDD-2. In addition, a Form I-HR CDD-3 may be associated.

Exhibit B -- Section 3
Form Instructions
Form I-HR CDD-1 (Con't)

- 3.4.1.2 Complete all header information according to the instructions in Section 3.3 and as follows:
- 3.4.1.2.1 Enter the "Matrix" of the sample being analyzed. The designation of matrix must reflect which one of the matrix-specific extraction procedures in Exhibit D was used for extraction of the sample.
- 3.4.1.2.2 For "Sample wt/vol", enter the number of grams (for soil, tissue, oil, and ash) or milliliters (for water) of sample used in the first blank line, and the units, either "G" or "ML", in the second blank.
- 3.4.1.2.3 For water samples, indicate the extraction procedure used by entering "SEPF" for separatory funnel extraction or "SPE" for solid phase extraction in the field labeled "Water Sample Prep".
- 3.4.1.2.4 Enter the actual volume of the most concentrated sample extract, in microliters, under "Conc. Extract Volume" This volume will typically be 20 microliters after addition of internal standard solution.
- 3.4.1.2.5 Enter "GC Column", "ID (mm)", "Lab Sample ID", and "Lab File ID" as described in Section 3.3.
- 3.4.1.2.6 "Date Received" is the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the validated time of sample receipt, VTSR) for that sample. It must be entered as MM/DD/YYYY.
- 3.4.1.2.7 "Date Extracted" and "Date Analyzed" must also be entered as MM/DD/YYYY.
- 3.4.1.2.8 "Date Analyzed" must be the date of the analysis for which the results are reported on Form I-HR. (If the sample requires a second column confirmation and is reported on Form I-HR CDD-3, the "Date Analyzed" on Form I-HR CDD-3 must be the date of the second analysis, while the date on Form I-HR CDD-1 and CDD-2 will be the date of the first analysis.)
- 3.4.1.2.9 If the sample has been diluted for analysis, enter the "Dilution Factor" as a single number, not a fraction. For example, enter "100.0" for a 1 to 100 dilution of the extract. Enter "0.1" for a concentration of 10 to 1. If the sample was not diluted, enter "1.0".
- 3.4.1.2.10 Enter the volume of the sample extract injected into the HRGC in the "Injection Volume" field. Report this volume in microliters (µL).
- 3.4.1.2.11 Enter the value for percent solid as described in Exhibit D for soil/sediment/sludge samples in the "% Solid/Lipids" field. For tissue samples enter the value for percent lipids, as determined in Exhibit D, in this field. For all other matrices, leave this field blank.
- 3.4.1.2.12 The appropriate concentration units, "PG/L" for water samples, or "NG/KG" for all other matrices, must be entered in the field for "CONCENTRATION UNITS".

- 3.4.1.3 For each analyte detected in a sample, enter the absolute retention time of the detected peak under "PEAK RT". Enter the retention time in minutes and decimal minutes, not seconds or minutes and seconds. The retention time must be entered even if the peak did not meet all of the identification criteria in Exhibit D.
- 3.4.1.4 Enter the ion abundance ratio for the two m/z's (listed under "Selected Ions") in the column labeled "ION RATIO". If the ion abundance ratio falls outside the acceptance limits listed in Exhibit D, place an asterisk (*) in the column under the number (#) symbol. For target analytes that meet all the identification criteria in Exhibit D, the Contractor will report the concentrations detected as uncorrected for blank contaminants in the column labeled "CONCENTRATION". Report all results to three significant figures.
- 3.4.1.5 Under the column labeled "Q" for qualifier, flag each result with the specific data reporting qualifiers listed below. The Contractor is encouraged to use additional flags as needed, but the definition of such flags must be explicit, must not contradict the qualifiers listed below, and must be included in the accompanying Narrative.
- 3.4.1.6 For reporting results to USEPA, the following contract-specific qualifiers are to be used. The seven qualifiers listed below are not subject to modification by the laboratory. Up to five qualifiers may be reported on Form I for each analyte. The seven EPA-defined qualifiers to be used are as follows:
- 3.4.1.6.1 U - Indicates compound was analyzed for, but not detected. The CONCENTRATION column is left blank in this instance, and an estimated detection limit (EDL) must be calculated based on the signal-to-noise ratio, as described in Exhibit D. This calculation takes into account the sample weight/volume extracted, the volume of the most concentrated extract, the injection volume, and dilution of the most concentrated extract prior to analysis.
- 3.4.1.6.2 J - Indicates an estimated value. This flag is used when the mass spectral data indicate presence of an analyte meeting all the identification criteria in Exhibit D, but the result is less than the Contract Required Quantitation Limit (CRQL), as listed in Exhibit C, but greater than zero.
- 3.4.1.6.3 B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- 3.4.1.6.4 E - This flag identifies analytes whose concentrations exceed the calibration range of the HRGC/HRMS instrument for that specific analysis. If one or more compounds have a response greater than fullscale, except as noted in Exhibit D, a smaller sample size must be extracted and analyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged "E" on the Form I for the original analysis. If the dilution causes any compounds identified in the first analysis to be below the calibration range in the second analysis, the results of both analyses will be reported on separate copies of

Exhibit B -- Section 3
Form Instructions
Form I-HR CDD-1 (Con't)

Form I. The Form I for the diluted sample will have the "DL" suffix appended to the EPA sample number.

- 3.4.1.6.5 D - This flag indicates all compounds identified in an analysis at a secondary dilution factor. If a smaller sample size is analyzed, as in the "E" flag above, the "DL" suffix is appended to the EPA sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample extract.
- 3.4.1.6.6 H - This flag indicates that the analyte in question was quantitated using peak heights rather than peak areas for both the analyte and its internal standard (see Exhibit D, Section 11).
- 3.4.1.6.7 X - Other specific flags may be required to properly define the results. If used, they must be fully described, and such description must be attached to the Sample Data Package and the SDG Narrative. Begin using "X". If more than one flag is needed, use "Y" and "Z" as needed. The laboratory-defined flags are limited to the letters "X", "Y", and "Z".
- 3.4.1.7 The combination of flags "BU" or "UB" is expressly prohibited. Blank contaminants are flagged "B" only when they are detected in the sample associated with the blank.
- 3.4.1.8 If a peak detected in the sample meets all of the identification criteria except the ion abundance ratio, flag the ion ratio as indicated above, and report the "Estimated Maximum Possible Concentration" as calculated in Exhibit D under the "EMPC/EDL" column. Do not report the value of the EMPC under the column labeled "CONCENTRATION", as that column is only for analytes meeting all the identification criteria.
- 3.4.1.9 If an analyte was not detected in the sample, enter "U" in the qualifier column, as described above, and report the Estimated Detection Limit" as calculated in Exhibit D under the "EMPC/EDL" column. Do not report the value of the EDL if there is an entry under "CONCENTRATION". The presence of the "U" alerts the data user that the reported value is an EDL, otherwise it is assumed to be an EMPC. EMPC values must be reported with "Peak Retention Times" and "Ion Ratios" with a flag in the "Q" column.
- 3.4.1.10 The bottom portion of Form I-HR CDD-1 contains the fields for reporting the recoveries of the labeled compounds and the cleanup standard. The recoveries of these standards are crucial in evaluating the effectiveness of this isotope dilution method. For each labeled compound and the cleanup standard, enter the absolute retention time of the standard in the sample in minutes and decimal minutes. Report the ion abundance ratio under the "ION RATIO" column. Flag any ion ratios that fall outside the ion ratio limits listed on the form by placing an asterisk (*) in the column under the number (#) symbol. There is no ion abundance ratio for the cleanup standard, as only one ion is monitored. Report the percent recovery of the labeled compounds and the cleanup standard, calculated according to Exhibit D, under the "%REC" column. The quality control limits for recovery are listed on the form. Flag any recovery outside those limits by placing an asterisk (*) under the number (#) symbol in the recovery column.

Requirements for re-analysis of samples due to poor recoveries are provided in Exhibit D.

3.4.2 Toxicity Equivalence Summary [Form I-HR CDD-2]

This page of Form I-HR reports the results of the toxicity equivalence calculations for each sample analyzed. The concentration of each of the 2,3,7,8-substituted CDD and CDF isomers is multiplied by a toxicity equivalence factor (TEF) to arrive at an equivalent toxicity concentration of 2,3,7,8-TCDD.

3.4.2.1 Complete the header information as specified in Section 3.3. The header of Form I-HR CDD-2 must match the header of Form I-HR CDD-1 for the same sample.

3.4.2.2 For each 2,3,7,8-substituted isomer positively identified in the sample, enter the concentration found in the column labeled "CONCENTRATION". If an isomer was not detected (e.g., flagged "U" on Form I CDD-1) for the purposes of this calculation, enter 0.0 (zero) (EDLs and EMPCs are entered as 0.0) as the concentration. Multiply each concentration times the TEF listed on the form for that isomer, and enter the product of the two in the column labeled "TEF-ADJUSTED CONCENTRATION". Add all 17 TEF-adjusted concentrations together, including any zeros for non-detected, and enter the total on the line at the bottom of the form.

3.4.3 Second Column Confirmation Results [Form I-HR CDD-3]

This page of Form I reports the results of all second column confirmation analyses performed. The requirements for second column confirmation are discussed in Exhibit D. Each time a second column confirmation is performed, the results are reported on Form I-HR CDD-3. Second column confirmation is not required for LCS and LCSD, therefore Form I-HR CDD-3 is not submitted for LCS and LCSD.

3.4.3.1 Complete the header information as specified in Section 3.3, except note that the field for "GC Column" must correspond to the second column confirmation analysis, i.e., it must not match that in the header of Form I-HR CDD-1 or CDD-2. Other fields such as "Date Analyzed", "Instrument ID", "Dilution Factor", and "Lab File ID" may also differ and must correspond to the second column confirmation analysis.

3.4.3.2 Complete the information in the lower portion of the form in a fashion similar to that for Form I-HR CDD-1, but enter the results of the second column confirmation.

3.4.3.3 Enter the data on recovery of the labeled compounds and cleanup standard from the second column confirmation analysis in a fashion similar to that for the original analysis.

3.4.4 CDD/CDF Total Homologue Concentration Summary [Form II-HR CDD]

This form reports the total concentration of all CDD/CDF isomers in a given homologue that are detected in the sample, including those isomers that do not represent the 2,3,7,8-substituted isomers of greatest toxicological concern. Because there are many isomers in each homologue, it is necessary to indicate the number of peaks that represent isomers within the congener. Enter the number of peaks detected in each congener under "PEAKS". For instance, if three

Exhibit B -- Section 3
Form Instructions
Forms III-HR CDD-1 and III-HR CDD-2

PeCDD peaks are detected and summed together, enter "3" under "PEAKS".

3.4.4.1 Enter the total concentration of the homologue, as calculated in Exhibit D, under "CONCENTRATION". Enter qualifiers under the "Q" column, as described in Section 3.4.1.6. If no isomers in a homologue were detected, enter "U" as the qualifier, and enter the lowest EDL of any of the 2,3,7,8-substituted isomers under the "EMPC/EDL" column.

3.4.4.2 If any of the peaks in a congener meet all the identification criteria except the ion abundance ratio, report the total concentration as an EMPC under the "EMPC/EDL" column.

3.4.5 CDD/CDF Lab Control Sample Summary [Form III-HR CDD-1]

This page of Form III reports the results of the Lab Control Sample (LCS) analysis.

3.4.5.1 Complete the header information as in Section 3.3. Enter the EPA sample number in the box at the top of the form. Similarly, the lab sample ID and lab file ID must refer to the LCS.

3.4.5.2 Under the "Spike Added" column, enter the calculated concentration of each of the 17 analytes in the LCS in pg/L or ng/Kg (according to the matrix) that results from dividing each spike compound amount added by the aliquot weight or volume. In the column labeled "Amount Recovered", enter the concentration (or EMPC) of each analyte detected in the lab control sample. The concentration units must be those indicated at the top of the form and be appropriate to the sample matrix listed in the header. Calculate the recovery of each spiked analyte as described in Exhibit D, and enter this value to the nearest whole percentage point in the column labeled "Percent Recovery". Flag any recoveries outside the quality control limits listed on the form by placing an asterisk (*) in the column under the number (#) symbol.

3.4.5.3 In addition to Form III CDD-1, a copy of Form I-HR CDD-1 must also be completed for the lab control sample analysis, following the procedures described above.

3.4.6 CDD/CDF Lab Control Sample Duplicate Summary [Form III-HR CDD-2]

This page of Form III reports the results of the lab control sample duplicate (LCSD) analysis.

3.4.6.1 Complete the header information as in Section 3.3. Enter the EPA sample number in the box at the top of the form. Similarly, the lab sample ID and lab file ID must refer to the LCSD.

3.4.6.2 In the columns labeled "Lab Control Sample Concentration" and "Lab Control Sample Duplicate Concentration" enter the concentration of each analyte from the LCS and LCSD, respectively. Calculate and report the Relative Percent Difference (RPD) under the "RPD" column. Flag any RPD outside the QC limits listed on the form by placing an asterisk (*) in the column under the number (#) symbol.

3.4.6.3 In addition to Form III CDD-1, a copy of Form I-HR CDD-1 must be completed for the lab control sample duplicate analysis as well, following the procedures described above.

3.4.7 CDD/CDF Method Blank Summary [Form IV-HR CDD]

This form summarizes the samples associated with each method blank analysis. A copy of Form IV-HR is required for each blank.

3.4.7.1 Complete the header information as described in Section 3.3. The EPA sample number entered in the box at the top of the form will be the number assigned to the method blank. The matrix entered on this form refers to the matrix of the associated samples, as one blank is required each time that samples of a similar matrix are extracted together. Therefore, samples of differing matrices cannot be mixed together on a single Form IV-HR.

3.4.7.2 Summarize the samples associated with a given method blank in the box in the lower portion of the form, entering the EPA sample number, lab sample ID, lab file ID, and date of analysis of each sample. Include lab control samples and lab control sample duplicates as well.

3.4.8 CDD/CDF Window Defining Mix Summary [Form V-HR CDD-1]

This page of Form V reports the results of the analysis of the window defining mixture that precedes each calibration verification on each GC column and instrument used for analysis. The analysis of this mixture is used to document the retention time window for the CDD/CDF congeners.

3.4.8.1 Complete the header information as described in Section 3.3, entering the EPA sample number of the window defining mixture injection in the box at the top of the form. The header information must correspond to the analysis of the window defining mixture.

3.4.8.2 In the box in the lower portion of the form, enter the absolute retention times of the first and last eluting isomers in each congener. Enter the retention times in minutes and decimal minutes, not minutes and seconds, nor seconds.

NOTE: As there is only one possible octachlorinated dioxin and furan, the retention times of these analytes are not contained in the window defining mixture, and are not reported here.

3.4.9 CDD/CDF Chromatographic Resolution Summary [Form V-HR CDD-2]

This page of Form V reports the chromatographic resolution of selected analytes in one of two solutions, depending on the GC column. The chromatographic resolution of these analytes is crucial to evaluating the results for the CDDs/CDFs reported in the samples. This evaluation is made every 12 hours during which samples or standards are analyzed. The Form V-HR CDD-2 shall be submitted for each column used.

3.4.9.1 For the DB-5 (or equivalent) column and for the DB-225 (or equivalent) column, the chromatographic resolution is judged from the analysis of the isomer specificity check that precedes the analysis of the calibration verification (see Exhibit D).

3.4.9.2 Complete one copy of Form V-HR CDD-2 for each analysis of the isomer specificity check on each GC column. Complete the header information, as described in Section 3.3, entering the EPA sample number of the isomer specificity check in the box at the top of

Exhibit B -- Section 3
Form Instructions
Forms V-HR CDD-3 and VI-HR CDD-1

the form. Enter the date and time of analysis of the standard in the header.

- 3.4.9.3 Calculate the chromatographic resolution for the GC column identified in the header according to the procedures in Exhibit D. For the DB-5 (or equivalent) column, enter only the results from the isomer specificity check analysis. For the DB-225 (or equivalent) column, enter only the results from the isomer specificity check analysis.

3.4.10 CDD/CDF Analytical Sequence Summary [Form V-HR CDD-3]

This page of Form V reports the sequence of analyses, including the analysis of the window defining mixture, isomer specificity check, the calibration standards, blanks, samples, lab control samples, and lab control sample duplicates. One copy of Form V CDD-3 is required for each 12-hour period during which samples, blanks, standards, etc. associated with the SDG are analyzed.

- 3.4.10.1 Complete the header information as described in Section 3.3. Enter the inclusive dates and times of the analyses of the first and last initial calibration standards in the fields for "Init. Calib. Date(s)" and "Init. Calib. Times". Dates must be in the format MM/DD/YYYY, and all times are expressed as HHMM, in military time (i.e., a 24-hour clock).
- 3.4.10.2 In the box in the lower portion of the form, enter the EPA sample number, lab sample ID, lab file ID, and date and time of analysis of all standards, samples, blanks, lab control samples, lab control sample duplicates, dilutions, re-analyses, etc. All analyses in the 12-hour period must be listed on Form V in chronological order. If analysis is not associated with the SDG being reported, enter the EPA sample number as "ZZZZZZ", as described in Section 3.3.
- 3.4.10.3 If the analytical sequence includes the analysis of the initial calibration standards, these standards and the window defining mix must be included on that copy of Form V, identified by the EPA sample numbers described in Section 3.3. A copy of the analytical sequence that includes these initial calibration standards and the window defining mix must be submitted with each data package to which the initial calibration applies, but the Case number and Client number must match those of each data package in which these initial calibration data are reported.
- 3.4.11 CDD/CDF Initial Calibration Response Factor Summary [Form VI-HR CDD-1]
- This form summarizes the relative response or relative response factors for each target analyte, labeled compound, and cleanup standard calculated from the initial calibration. Complete the header information as described in Section 3.3. Enter the inclusive initial calibration date(s) and times, as described for Form V-HR CDD-3. One copy of Form VI-HR CDD-1 must be completed for each initial calibration, for each instrument and GC column used for analysis of samples, and must be accompanied by a corresponding Form VI-HR CDD-2.
- 3.4.11.1 Enter the Relative Response (RR) and Relative Response Factors (RRF) determined from the analysis of each of the calibration standards (CS1 through CS5). Enter RR/RRF values to three decimal

places. Calculate the mean RR/RRF, as described in Exhibit D, and enter in the column "MEAN RR/RRF". Calculate the relative standard deviation (%RSD), and enter under "%RSD". Note that as the internal standards are used to determine the RRFs of the labeled compounds, no RRF values can be calculated for the internal standards, and therefore, they do not appear on Form VI CDD-1.

3.4.12 CDD/CDF Initial Calibration Ion Abundance Ratio Summary [Form VI-HR CDD-2]

This page of Form VI reports the ion abundance ratios for each of the initial calibration standards. Because the ratio of the abundances of the two ions monitored for each analyte is crucial to the identification of these analytes, the ion abundance ratios must meet the quality control limits.

3.4.12.1 For each native analyte, labeled compound and internal standard, the two ions monitored for each analyte are listed in the column labeled "Selected Ions". Calculate the ratio of the abundances of these two ions and enter the ion abundance ratio of each analyte in each of the initial calibration standards to two decimal places.

3.4.12.2 Compare the ion abundance ratios to the quality control limits shown on the form, and flag any analyte which did not meet these limits in one or more of the standards.

NOTE: The cleanup standard does not appear on Form VI-HR CDD-2, as only one ion is monitored for this analyte, and therefore, no ion abundance ratio can be calculated.

3.4.12.3 One copy of Form VI-HR CDD-2 must be completed for each initial calibration, for each instrument and GC column used for analysis of samples, and must accompany a corresponding copy of Form VI CDD-1.

3.4.13 CDD/CDF Continuing Calibration Summary [Form VII-HR CDD-1]

This page of Form VII summarizes the results of the continuing calibration that must occur in each 12-hour analytical sequence. The form is used to report the RR/RRF values and ion abundance ratios of each analyte in the CS3 standard, and to compare these values to the initial calibration data reported on Form VI-HR CDD-1.

3.4.13.1 One copy of Form VII-HR CDD-1 must be completed for each continuing calibration performed, and must be accompanied by a corresponding copy of Form VII-HR CDD-2.

3.4.13.2 Complete the header information as described in Section 3.3. The date and time of analysis and lab file ID in the header must correspond to the analysis of the CS3 standard. Enter the dates and times of the associated initial calibration in the fields for "Init. Calib. Date(s)" and "Init. Calib. Times" respectively. If the calendar date changed during the initial calibration, enter the inclusive dates of the first and last standards in the associated initial calibration in the fields for "Init. Calib. Date(s)".

3.4.13.3 For each of the native analytes, labeled compounds, and the cleanup standard in the CS3 Standard, enter the Relative Response

(RR) or Relative Response Factor (RRF) determined from the analysis of the continuing calibration standard in the column labeled "RR/RRF". Enter the mean RR/RRF for each analyte from the associated initial calibration, in the column labeled "MEAN RR/RRF". The values reported in this column must match those reported on the Form VI for the associated initial calibration. Calculate the percent difference (%D) between the RR/RRF and the mean RR/RRF for each analyte, and report under "%D". If the percent difference exceeds the quality control limits specified in Exhibit D, flag that analyte by placing an asterisk (*) in the "%D FLAG" column. Report the ion abundance ratio of each analyte under the "ION RATIO" column. Flag any ion ratio that falls outside the quality control limits shown on the form by placing an asterisk (*) in the "ION RATIO FLAG" column.

NOTE: Because only one ion is monitored for the cleanup standard, no ion ratio is determined for this analyte. For the internal standards, relative response factors are not calculated or reported on Form VII-HR CDD-1, but the ion abundance ratios for these standards must be reported on Form VII-HR CDD-1.

3.4.14 CDD/CDF Continuing Calibration Retention Time Summary [Form VII-HR CDD-2]

This page of Form VII summarizes the absolute and relative retention times of the analytes in the continuing calibration standards that must be analyzed in each 12-hour analytical sequence. Absolute retention times and relative retention times are critical to the identification of CDDs/CDFs by this method. One copy of Form VII-HR CDD-2 must be completed for each continuing calibration performed and must be accompanied by a corresponding copy of Form VII-HR CDD-1.

3.4.14.1 Complete the header information as described in Section 3.3. The date and time of analysis and lab file ID in the header must correspond to the analysis of the CS3 standard. Enter the date of the associated initial calibration in the field for "Init. Calib. Date(s)". If the calendar date changed during the initial calibration, enter the inclusive dates of the analyses of the first and last standards in the associated initial calibration in the fields for "Init. Calib. Date(s)".

3.4.14.2 For each of the native and labeled analytes, enter the relative retention time (RRT) and absolute retention time (RT) of the analyte in the calibration standard. RRT is calculated as the RT of the native analyte divided by the RT of appropriate labeled compound and the RT of the labeled compound divided by the RT of the appropriate internal standard. For the internal standards, report only the absolute retention times. Enter all RTs in minutes and decimal minutes. RRTs are reported to two decimal places.

3.5 Sample Log-In Sheet [Form DC-1]

This form documents the receipt and inspection of sample containers and samples. One original of Form DC-1 is required for each sample shipping container. If the samples in a single sample shipping container must be assigned to more than one SDG, the original Form DC-1 will be placed with the deliverables for the lowest alpha numeric SDG number, and a copy of Form DC-1 must be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)", and the location of the original should be noted on the copies.

- 3.5.1 Sign and date the airbill (if present). Examine the shipping container and record the presence/absence of custody seals and their condition (e.g., intact, broken) in item 1 of Form DC-1. Record the custody seal numbers in Item 2.
- 3.5.2 Open the container, remove the enclosed sample documentation, and record the presence/absence of a chain-of-custody record(s), SMO forms (e.g., Traffic Reports, Packing Lists), and airbills or airbill stickers in Items 3-5. Specify if there is an airbill present or an airbill sticker in Item 5. Record the airbill or sticker number in Item 6.
- 3.5.3 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (e.g., intact, broken, leaking) and presence or absence of sample tags in Items 7 and 8.
- 3.5.4 Review the sample shipping documents and complete the header information as described in Section 3.3. Report the temperature of the cooler under Item 9. Compare the information recorded on all the documents and samples and circle the appropriate answer in Item 10.
- 3.5.5 If there are no problems observed during receipt, sign and date (include time) Form DC-1, the chain-of-custody record, and the Traffic Report, and write the sample numbers on Form DC-1. Record the appropriate sample tags and assigned laboratory numbers, if applicable. The log-in date should be recorded at the top of Form DC-1 and the date and time of cooler receipt at the laboratory should be recorded in Items 11 and 12. Record the specific area designation (e.g., refrigerator number) in the Sample Transfer block located in the bottom left corner of Form DC-1. Sign and date the Sample Transfer block. Cross out unused columns and spaces.
- 3.5.6 If there are problems observed during receipt or an answer marked with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact and the resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.6 CDD/CDF Complete SDG File (CSF) Inventory Sheet [Form DC-2]

This form is used to record the inventory of the CSF documents and the count of documents in the original Sample Data Package that is sent to the Region.

- 3.6.1 Organize all CSF documents, as described in Section 2. Assemble the documents in the order specified on Form DC-2 (high resolution) and Section 2, and stamp each page with a consecutive number. (Do not number the DC-2 form.) Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided in the Form DC-2 (high resolution). If there are no documents for a specific document type, enter "NA" in the empty space.
- 3.6.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly-defined category. The laboratory should review Form DC-2 (high resolution) to determine if it is most appropriate to place them under Item 5, 6, 7, or 8. Item 8 should be used if there is no appropriate previous item. These types of documents should be described or listed in the blanks under each appropriate item.

Exhibit B -- Section 4
Data Reporting Forms

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

EXHIBIT B
CDD/CDF FORMS

1DFA
CDD/CDF SAMPLE DATA SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) _____ Date Received: _____

Concentrated Extract Volume: _____ (uL) _____ Date Extracted: _____

Injection Volume: _____ (uL) %Solid/Lipids: _____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) _____ Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

TARGET ANALYTE	SELECTED IONS	PEAK RT	ION RATIO #	CONCENTRATION	Q	EMPC/EDL
2378-TCDD	320/322					
2378-TCDF	304/306					
12378-PeCDF	340/342					
12378-PeCDD	356/358					
23478-PeCDF	340/342					
123478-HxCDF	374/376					
123678-HxCDF	374/376					
123478-HxCDD	390/392					
123678-HxCDD	390/392					
123789-HxCDD	390/392					
234678-HxCDF	374/376					
123789-HxCDF	374/376					
1234678-HpCDF	408/410					
1234678-HpCDD	424/426					
1234789-HpCDF	408/410					
OCDD	458/460					
OCDF	442/444					

NOTE: Concentrations, EMPCs, and EDLs for solid samples are calculated on a dry weight basis (except tissues, which are reported on a wet weight basis with % lipids).

LABELED COMPOUNDS	SELECTED IONS	PEAK RT	ION RATIO #	ION RATIO LIMITS	% REC #	RECOVERY LIMITS
13C-2378-TCDD	332/334			0.65-0.89		25-164
13C-12378-PeCDD	368/370			1.32-1.78		25-181
13C-123478-HxCDD	402/404			1.05-1.43		32-141
13C-123678-HxCDD	402/404			1.05-1.43		28-130
13C-1234678-HpCDD	424/426			0.88-1.20		23-140
13C-OCDD	470/472			0.76-1.02		17-157
13C-2378-TCDF	316/318			0.65-0.89		24-169
13C-12378-PeCDF	352/354			1.32-1.78		24-185
13C-23478-PeCDF	352/354			1.32-1.78		21-178
13C-123478-HxCDF	384/386			0.43-0.59		26-152
13C-123678-HxCDF	384/386			0.43-0.59		26-123
13C-123789-HxCDF	384/386			0.43-0.59		29-147
13C-234678-HxCDF	384/386			0.43-0.59		28-136
13C-1234678-HpCDF	418/420			0.37-0.51		28-143
13C-1234789-HpCDF	418/420			0.37-0.51		26-138
37C1-2378-TCDD	328/NA		NA	NA		35-197

Column to be used to flag values outside QC limits

1DFB
CDD/CDF TOXICITY EQUIVALENCE SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil/ _____) Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) Date Received: _____

Concentrated Extract Volume: _____ (uL) Date Extracted: _____

Injection Volume: _____ (uL) %Solid/Lipids: _____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

TARGET ANALYTE	CONCENTRATION	TEF*	TEF-ADJUSTED CONCENTRATION
2378-TCDD		x 1.0 =	
2378-TCDF		x 0.1 =	
12378-PeCDF		x 0.05 =	
12378-PeCDD		x 0.5 =	
23478-PeCDF		x 0.5 =	
123478-HxCDF		x 0.1 =	
123678-HxCDF		x 0.1 =	
123478-HxCDD		x 0.1 =	
123678-HxCDD		x 0.1 =	
123789-HxCDD		x 0.1 =	
234678-HxCDF		x 0.1 =	
123789-HxCDF		x 0.1 =	
1234678-HpCDF		x 0.01 =	
1234678-HpCDD		x 0.01 =	
1234789-HpCDF		x 0.01 =	
OCDD		x 0.001 =	
OCDF		x 0.001 =	
		Total =	

* TEF - Toxicity Equivalent Factors from EPA/625/3-89/016 March 1989 - Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxins and Chlorinated-Dibenzofurans (CDDs and CDFs) and 1989 Update.

1DFC
CDD/CDF SECOND COLUMN CONFIRMATION
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) Date Received: _____

Concentrated Extract Volume: _____ (uL) Date Extracted: _____

Injection Volume: _____ (uL) %Solid/Lipids: _____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

ANALYTE	SELECTED IONS	PEAK RT	ION RATIO #	CONCENTRATION	Q	EMPC/EDL
2378-TCDD	320/322					
2378-TCDF	304/306					

NOTE: Concentrations, EMPCs, and EDLs for solid samples are calculated on a dry weight basis (except tissues, which are reported on a wet weight basis with % lipids).

LABELED COMPOUNDS	SELECTED IONS	PEAK RT	ION RATIO #	ION RATIO LIMITS	% REC #	RECOVERY LIMITS
13C-2378-TCDD	332/334			0.65-0.89		25-164
13C-2378-TCDF	316/318			0.65-0.89		24-169
37Cl-2378-TCDD	328/NA		NA	NA		35-197

Column to be used to flag values outside QC limits

2DF
CDD/CDF TOTAL HOMOLOGUE CONCENTRATION SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) Date Received: _____

Concentrated Extract Volume: _____ (uL) Date Extracted: _____

Injection Volume: _____ (uL) %Solid/Lipids: _____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

HOMOLOGUE	PEAKS	CONCENTRATION	Q	EMPC/EDL
DIOXINS				
Total TCDD				
Total PeCDD				
Total HxCDD				
Total HpCDD				
FURANS				
Total TCDF				
Total PeCDF				
Total HxCDF				
Total HpCDF				

NOTE: Concentrations, EMPCs, and EDLs for solid samples are calculated on a dry weight basis (except tissues, which are reported on a wet weight basis with % lipids). The total homologue concentrations do not affect the TEF calculations.

3DFA
CDD/CDF LAB CONTROL SAMPLE SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) Date Received: _____

Concentrated Extract Volume: _____ (uL) Date Extracted: _____

Injection Volume: _____ (uL) Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

SPIKE ANALYTE	SPIKE ADDED	AMOUNT RECOVERED	PERCENT RECOVERY	#	QC LIMITS
2378-TCDD					67-158
2378-TCDF					75-158
12378-PeCDF					80-134
12378-PeCDD					70-142
23478-PeCDF					68-160
123478-HxCDF					72-134
123678-HxCDF					84-130
123478-HxCDD					70-164
123678-HxCDD					76-134
123789-HxCDD					64-162
234678-HxCDF					70-156
123789-HxCDF					78-130
1234678-HpCDF					82-132
1234678-HpCDD					70-140
1234789-HpCDF					78-138
OCDD					78-144
OCDF					63-170

Column to be used to flag values outside QC limits.

LCS Recovery: _____ Outside limits out of _____ total.

3DFB
CDD/CDF LAB CONTROL SAMPLE DUPLICATE SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Sample wt/vol: _____ (g/ml) _____ Lab File ID: _____

Water Sample Prep: _____ (Sepf/SPE) Date Received: _____

Concentrated Extract Volume: _____ (uL) Date Extracted: _____

Injection Volume: _____ (uL) %Solid: _____ Date Analyzed: _____

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

CONCENTRATION UNITS: (pg/L or ng/Kg) _____

SPIKE ANALYTE	LAB CONTROL SAMPLE CONCENTRATION	LAB CONTROL SAMPLE DUPLICATE CONCENTRATION	RPD #	QC LIMITS
2378-TCDD				# 30
2378-TCDF				# 30
12378-PeCDF				# 30
12378-PeCDD				# 30
23478-PeCDF				# 30
123478-HxCDF				# 30
123678-HxCDF				# 30
123478-HxCDD				# 30
123678-HxCDD				# 30
123789-HxCDD				# 30
123789-HxCDF				# 30
234678-HxCDF				# 30
1234678-HpCDF				# 30
1234678-HpCDD				# 30
1234789-HpCDF				# 30
OCDD				# 30
OCDF				# 30

If an analyte is not detected in either analysis, enter 0 (zero) as the concentration.

column to be used to flag values outside QC limits.

QC limits are advisory.

4DF
CDD/CDF METHOD BLANK SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

--

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

Matrix: (soil/water/ash/tissue/oil) _____ Lab Sample ID: _____

Water Sample Prep: _____ (Sepf/SPE) Lab File ID: _____

GC Column: _____ ID: _____ (mm) Date Extracted: _____

Instrument ID: _____ Date Analyzed: _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, LAB CONTROL SAMPLES, AND
LAB CONTROL SAMPLE DUPLICATES.

EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED

5DFA
CDD/CDF WINDOW DEFINING MIX SUMMARY
HIGH RESOLUTION

EPA SAMPLE NO.

--

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Lab File ID: _____

Instrument ID: _____ Date Analyzed: _____

Time Analyzed: _____

CDD/CDF	RT FIRST ELUTING	RT LAST ELUTING
TCDD		
TCDF		
PeCDD		
PeCDF		
HxCDD		
HxCDF		
HpCDD		
HpCDF		

5DFB
CDD/CDF CHROMATOGRAPHIC RESOLUTION SUMMARY
HIGH RESOLUTIONEPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Lab File ID: _____

Instrument ID: _____ Date Analyzed: _____

Time Analyzed: _____

Percent Valley determination for DB-5 (or equivalent) column -
For the column performance solution beginning the 12-hour period:

1238-TCDD/2378-TCDD: _____

QC LIMITS:

Percent Valley between the TCDD isomers must be less than or equal to 25%.

Percent Valley Determination for DB-225 (or equivalent) column -
For the column Performance Solution beginning the 12-hour period:

2347-TCDF/2378-TCDF: _____

QC LIMITS:

Percent Valley between the TCDD/TCDF isomers must be less than or equal to
25%.

USEPA - CLP

5DFC
CDD/CDF ANALYTICAL SEQUENCE SUMMARY
HIGH RESOLUTION

Lab Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____
GC Column: _____ ID: _____ (mm) Instrument ID: _____
Init. Calib. Date(s): _____
Init. Calib. Times: _____

THE ANALYTICAL SEQUENCE OF STANDARDS, SAMPLES, BLANKS, LAB CONTROL SAMPLES,
AND LAB CONTROL SAMPLE DUPLICATES IS AS FOLLOWS:

EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED

6DFA
CDD/CDF INITIAL CALIBRATION RESPONSE FACTOR SUMMARY
HIGH RESOLUTION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Instrument ID: _____

Init. Calib. Date(s): _____

Init. Calib. Times: _____

TARGET ANALYTES	RR/RRF					MEAN RR/RRF	%RSD	QC LIMITS
	CS1	CS2	CS3	CS4	CS5			
2378-TCDD								± 20%
2378-TCDF								± 20%
12378-PeCDF								± 20%
12378-PeCDD								± 20%
23478-PeCDF								± 20%
123478-HxCDF								± 20%
123678-HxCDF								± 20%
123478-HxCDD								± 20%
123678-HxCDD								± 20%
123789-HxCDD ¹								± 20%
234678-HxCDF								± 20%
123789-HxCDF								± 20%
1234678-HpCDF								± 20%
1234678-HpCDD								± 20%
1234789-HpCDF								± 20%
OCDD								± 20%
OCDF ²								± 20%
LABELED COMPOUNDS								
13C-2378-TCDD								± 35%
13C-12378-PeCDD								± 35%
13C-123478-HxCDD								± 35%
13C-123678-HxCDD								± 35%
13C-1234678-HpCDD								± 35%
13C-OCDD								± 35%
13C-2378-TCDF								± 35%
13C-12378-PeCDF								± 35%
13C-23478-PeCDF								± 35%
13C-123478-HxCDF								± 35%
13C-123678-HxCDF								± 35%
13C-123789-HxCDF								± 35%
13C-234678-HxCDF								± 35%
13C-1234678-HpCDF								± 35%
13C-1234789-HpCDF								± 35%
37C1-2378-TCDD								± 35%

¹The relative response is calculated based on the labeled analogs of the other two HxCDDs.

²The relative response is calculated based on the labeled analog of OCDD.

6DFB
CDD/CDF INITIAL CALIBRATION ION ABUNDANCE RATIO SUMMARY
HIGH RESOLUTION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Instrument ID: _____

Init. Calib. Date(s): _____

Init. Calib. Times: _____

TARGET ANALYTES	SELECTED IONS	ION ABUNDANCE RATIO					FLAG	ION RATIO QC LIMITS
		CS1	CS2	CS3	CS4	CS5		
2378-TCDD	320/322							0.65-0.89
2378-TCDF	304/306							0.65-0.89
12378-PeCDF	340/342							1.32-1.78
12378-PeCDD	356/358							1.32-1.78
23478-PeCDF	340/342							1.32-1.78
123478-HxCDF	374/376							1.05-1.43
123678-HxCDF	374/376							1.05-1.43
123478-HxCDD	390/392							1.05-1.43
123678-HxCDD	390/392							1.05-1.43
123789-HxCDD	390/392							1.05-1.43
234678-HxCDF	374/376							1.05-1.43
123789-HxCDF	374/376							1.05-1.43
1234678-HpCDF	408/410							0.88-1.20
1234678-HpCDD	424/426							0.88-1.20
1234789-HpCDF	408/410							0.88-1.20
OCDD	458/460							0.76-1.02
OCDF	442/444							0.76-1.02
LABELED COMPOUNDS								
13C-2378-TCDD	332/334							0.65-0.89
13C-12378-PeCDD	368/370							1.32-1.78
13C-123478-HxCDD	402/404							1.05-1.43
13C-123678-HxCDD	402/404							1.05-1.43
13C-1234678-HpCDD	436/438							0.88-1.20
13C-OCDD	470/472							0.76-1.02
13C-2378-TCDF	316/318							0.65-0.89
13C-12378-PeCDF	352/354							1.32-1.78
13C-23478-PeCDF	352/354							1.32-1.78
13C-123478-HxCDF	384/386							0.43-0.59
13C-123678-HxCDF	384/386							0.43-0.59
13C-123789-HxCDF	384/386							0.43-0.59
13C-234678-HxCDF	384/386							0.43-0.59
13C-1234678-HpCDF	418/420							0.37-0.51
13C-1234789-HpCDF	418/420							0.37-0.51
INTERNAL STANDARDS								
13C-1234-TCDD	332/334							0.65-0.89
13C-123789-HxCDD	402/404							1.05-1.43

QC limits represent $\pm 15\%$ window around the theoretical ion abundance ratio. The laboratory must flag any analyte in any calibration solution which does not meet the ion abundance ratio QC limit by placing an asterisk in the flag column.

USEPA - CLP

7DFA
CDD/CDF CONTINUING CALIBRATION SUMMARY
HIGH RESOLUTION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Instrument ID: _____

Lab File ID: _____ Date Analyzed: _____ Time Analyzed: _____

Init. Calib. Times: _____ Init. Calib. Dates: _____

TARGET ANALYTES	SELECTED IONS	RR/RRF	MEAN RR/RRF	%D	%D FLAG	ION RATIO	ION RATIO FLAG	ION RATIO QC LIMITS
2378-TCDD	320/322							0.65-0.89
2378-TCDF	304/306							0.65-0.89
12378-PeCDF	340/342							1.32-1.78
12378-PeCDD	356/358							1.32-1.78
23478-PeCDF	340/342							1.32-1.78
123478-HxCDF	374/376							1.05-1.43
123678-HxCDF	374/376							1.05-1.43
123478-HxCDD	390/392							1.05-1.43
123678-HxCDD	390/392							1.05-1.43
123789-HxCDD	390/392							1.05-1.43
234678-HxCDF	374/376							1.05-1.43
123789-HxCDF	374/376							1.05-1.43
1234678-HpCDF	408/410							0.88-1.20
1234678-HpCDD	424/426							0.88-1.20
1234789-HpCDF	408/410							0.88-1.20
OCDD	458/460							0.76-1.02
OCDF	442/444							0.76-1.02
LABELED COMPOUNDS								
13C-2378-TCDD	332/334							0.65-0.89
13C-12378-PeCDD	368/370							1.32-1.78
13C-123478-HxCDD	402/404							1.05-1.43
13C-123678-HxCDD	402/404							1.05-1.43
13C-1234678-HpCDD	424/426							0.88-1.20
13C-OCDD	470/472							0.76-1.02
13C-2378-TCDF	316/318							0.65-0.89
13C-12378-PeCDF	352/354							1.32-1.78
13C-23478-PeCDF	352/354							1.32-1.78
13C-123478-HxCDF	384/386							0.43-0.59
13C-123678-HxCDF	384/386							0.43-0.59
13C-123789-HxCDF	384/386							0.43-0.59
13C-234678-HxCDF	384/386							0.43-0.59
13C-1234678-HpCDF	418/420							0.37-0.51
13C-1234789-HpCDF	418/420							0.37-0.51
CLEAN-UP								
37Cl-2378-TCDD	328/NA					NA	NA	NA
INTERNAL STANDARDS								
13C-1234-TCDD	332/334	NA	NA	NA	NA			0.65-0.89
13C-123789-HxCDD	402/404	NA	NA	NA	NA			1.05-1.43

The laboratory must flag any analyte which does not meet criteria for %D or ion abundance ratio by placing an asterisk in the appropriate flag column.

7DFB

CDD/CDF CONTINUING CALIBRATION RETENTION TIME SUMMARY
HIGH RESOLUTION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Client No.: _____ SDG No.: _____

GC Column: _____ ID: _____ (mm) Instrument ID: _____

Date Analyzed: _____ Time Analyzed: _____

Lab File ID: _____ Init. Calib. Date(s): _____

TARGET ANALYTES	RRT	RT
2378-TCDD		
2378-TCDF		
12378-PeCDF		
12378-PeCDD		
23478-PeCDF		
123478-HxCDF		
123678-HxCDF		
123478-HxCDD		
123678-HxCDD		
123789-HxCDD		
234678-HxCDF		
123789-HxCDF		
1234678-HpCDF		
1234678-HpCDD		
1234789-HpCDF		
OCDD		
OCDF		
LABELED COMPOUNDS		
13C-2378-TCDD		
13C-12378-PeCDD		
13C-123478-HxCDD		
13C-123678-HxCDD		
13C-1234678-HpCDD		
13C-OCDD		
13C-2378-TCDF		
13C-12378-PeCDF		
13C-23478-PeCDF		
13C-123478-HxCDF		
13C-123678-HxCDF		
13C-123789-HxCDF		
13C-234678-HxCDF		
13C-1234678-HpCDF		
CLEAN-UP STANDARD		
37C1-2378-TCDD	NA	
INTERNAL STANDARD		
13C-1234-TCDD	NA	
13C-123789-HxCDD	NA	

RRT = (RT of analyte) / (RT of appropriate labeled compound)

CDD/CDF
SAMPLE LOG-IN SHEET (DC-1)

Lab Name				Page ____ of ____	
Received By (Print Name)				Log-in Date	
Received By (Signature)					
Contract No.				Client Number	
Case Number		Sample Delivery Group No.			
Remarks:		EPA Sample #	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____				
3. Chain-of Custody Records	Present/Absent*				
4. Traffic Reports or Packing Lists	_____				
5. Airbill	Airbill/Sticker Present/Absent*				
6. Airbill No.	_____				
7. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of- Custody				
8. Sample Condition	Intact/Broken*/ Leaking				
9. Cooler Temperature					
10. Does information on custody records and sample tags agree?	Yes/No*				
11. Date Received at Lab	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

CDD/CDF COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____
CITY/STATE _____
CASE NO. _____ SDG NO. _____ SDG NOS. TO FOLLOW _____
CLIENT NO. _____
CONTRACT NO. _____
SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section II and Section III)

	<u>PAGE NOS.</u>		<u>CHECK</u>	
	<u>FROM</u>	<u>TO</u>	<u>LAB</u>	<u>EPA</u>
1. <u>Inventory Sheet</u> (DC-2) (Do not number)	_____	_____	_____	_____
2. <u>SDG Narrative</u>	_____	_____	_____	_____
3. <u>Traffic Report</u>	_____	_____	_____	_____
4. <u>CDD/CDF Data</u>				
a. Sample Data				
Sample Data Summary (Form I-HR CDD-1)	_____	_____	_____	_____
Toxicity Equivalence Summary (Form I-HR CDD-2)	_____	_____	_____	_____
Second Column confirmation Summary (Form I-HR CDD-3)	_____	_____	_____	_____
Selected Ion Current Profile (SICP) for each sample	_____	_____	_____	_____
Quantitation Reports and Area Summaries	_____	_____	_____	_____
Total Homologue Concentration Summary (Form II-HR CDD)	_____	_____	_____	_____
b. Quality Control Data				
Lab Control Sample Summary (Form III-HR CDD-1)	_____	_____	_____	_____
Lab Control Sample Duplicate Summary (Form III-HR CDD-2)	_____	_____	_____	_____
Method Blank Summary (Form IV-HR CDD)	_____	_____	_____	_____
Window Defining Mix Summary (Form V-HR CDD-1)	_____	_____	_____	_____
Chromatographic Resolution Summary (Form V-HR CDD-2)	_____	_____	_____	_____
Analytical Sequence Summary (Form V-HR CDD-3)	_____	_____	_____	_____
c. Calibration Data				
Initial Calibration Data (Form VI-HR CDD-1 and Form VI-HR CDD-2), PFK mass resolution, CDD/CDF standard(s) SICPs, Quantitation Reports, and Area Summaries for the initial (five-point) calibration	_____	_____	_____	_____
Continuing Calibration Data (Form VII-HR CDD-1 and Form VII-HR CDD-2), PFK mass resolution, SICPs, Quantitation Reports, and Area Summaries	_____	_____	_____	_____
d. Raw Quality Control Data				
Blank Data Form I-HR CDD-1, CDD-2, CDD-3 (if applicable)	_____	_____	_____	_____

		<u>PAGE NOS.</u>		<u>CHECK</u>	
		<u>FROM</u>	<u>TO</u>	<u>LAB</u>	<u>EPA</u>
Blank Data including SICPs, Quantitation Reports, and Area Summaries for each blank analyzed		_____	_____	_____	_____
LCS/LCSD Form I-HR CDD-1 and CDD-2		_____	_____	_____	_____
LCS/LCSD Data including SICPs, Quantitation Reports, and Area Summaries		_____	_____	_____	_____
5.	<u>Miscellaneous Data</u>				
	Original preparation and analysis forms or copies of preparation and analysis logbook pages	_____	_____	_____	_____
	Internal sample and sample extract transfer chain-of-custody records	_____	_____	_____	_____
	Screening records	_____	_____	_____	_____
	All instrument output, including strip charts from screening activities (describe or list)				

6.	<u>EPA Shipping/Receiving Documents</u>				
	Airbills (No. of shipments _____)	_____	_____	_____	_____
	Chain-of-Custody Records	_____	_____	_____	_____
	Sample Tags	_____	_____	_____	_____
	Sample Log-In Sheet (Lab & DC-1)	_____	_____	_____	_____
	TR Cover Sheet	_____	_____	_____	_____
	Miscellaneous Shipping/Receiving Records (describe or list)				

7.	<u>Internal Lab Sample Transfer Records and Tracking Sheets</u> (describe or list)				

8.	<u>Other Records</u> (describe or list)				
	Telephone Communication Log				

9.	<u>Comments:</u> _____				

Completed by:

(CLP Lab) _____
(Signature)

(Print Name & Title) _____

(Date) _____

Audited by:

(USEPA) _____
(Signature)

(Print Name & Title) _____

(Date) _____

EXHIBIT C

TARGET COMPOUND LIST (TCL)
AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

THIS PAGE INTENTIONALLY LEFT BLANK

EXHIBIT C - TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

Table of Contents

<u>Section</u>	<u>Page</u>
1.0 CDD/CDF TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)	5
2.0 TOTAL HOMOLOGUES	6

THIS PAGE INTENTIONALLY LEFT BLANK

1.0 CDD/CDF TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

CDD/CDF	CAS No.	WATER (pg/L)	SOLIDS* (ng/Kg)
2378-TCDD	1746-01-6	10	1.0
12378-PeCDD	40321-76-4	50	5.0
123678-HxCDD	57653-85-7	50	5.0
123478-HxCDD	39227-28-6	50	5.0
123789-HxCDD	19408-74-3	50	5.0
1234678-HpCDD	35822-46-9	50	5.0
OCDD	3268-87-9	100	10
2378-TCDF	51207-31-9	10	1.0
12378-PeCDF	57117-41-6	50	5.0
23478-PeCDF	57117-31-4	50	5.0
123678-HxCDF	57117-44-9	50	5.0
123789-HxCDF	72918-21-9	50	5.0
123478-HxCDF	70648-26-9	50	5.0
234678-HxCDF	60851-34-5	50	5.0
1234678-HpCDF	67562-39-4	50	5.0
1234789-HpCDF	55673-89-7	50	5.0
OCDF	39001-02-0	100	10

* Solids include soil, sediment, sludge, tissue (no human tissue), ash, oil, and oil matrices.

NOTE: The values in these tables are quantitation limits, not absolute detection limits. The amount of material necessary to produce a detector response that can be identified and reliably quantified is greater than that needed to be simply detected above the background noise. The quantitation limits in these tables are set at the concentrations in the sample equivalent to the concentration of the lowest calibration standard analyzed for each analyte.

Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

These CRQL values are based on the analysis of samples according to the specifications given in Exhibit D. Sample data are reported on a dry weight basis for all non-aqueous samples (except tissues which are reported on a wet weight basis, along with their percent lipid content).

Exhibit C -- Section 2
Total Homologues

2.0 TOTAL HOMOLOGUES

Data are reported for the total concentration of all detected CDDs or CDFs in the following homologues. However, because the number of non-2378-substituted isomers that might be detected in a sample is unpredictable, it is not possible to assign CRQL values to the total homologue concentrations.

Homologue	CAS No.	No. of Possible Isomers	No. of 2378- Substituted Isomers
Total TCDD	41903-57-5	22	1
Total PeCDD	36088-22-9	14	1
Total HxCDD	34465-46-8	10	3
Total HpCDD	37871-00-4	2	1
Total TCDF	55722-27-5	38	1
Total PeCDF	30402-15-4	28	2
Total HxCDF	55684-94-1	16	4
Total HpCDF	38998-75-3	4	2

There is only one isomer in both the OCDD and OCDF homologues, hence the total concentration is the same as the 2378-substituted concentration.

Homologue	Definition
TCDD =	Tetrachlorinated dibenzo-p-dioxin
TCDF =	Tetrachlorinated dibenzofuran
PeCDD =	Pentachlorinated dibenzo-p-dioxin
PeCDF =	Pentachlorinated dibenzofuran
HxCDD =	Hexachlorinated dibenzo-p-dioxin
HxCDF =	Hexachlorinated dibenzofuran
HpCDD =	Heptachlorinated dibenzo-p-dioxin
HpCDF =	Heptachlorinated dibenzofuran
OCDD =	Octachlorinated dibenzo-p-dioxin
OCDF =	Octachlorinated dibenzofuran