USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

DIOXIN ANALYSIS

Multi-Media

Multi-Concentration

SOW. 9/86

Rev. 8/87

Form IFB Series: WA86-K357

STATEMENT OF WORK

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SCOPE OF WORK

The purpose of this contract is to provide EPA with chemical analytical services using selected ion monitoring (SIM) gas chromatography/mass spectrometry/data system (GC/MS/DS) techniques for the analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) in soil or sediment and water samples. The majority of these samples are from areas of suspected 2,3,7,8-TCDD contamination. The methods required in this contract are effective for a concentration range of 1 to 1000 parts per billion for soil or sediment and 0.01 to 10 parts per billion for water.

The Contractor shall use safe handling procedures and generally accepted good laboratory practices to prepare and analyze for the presence of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) in soil or sediment and water samples.

The data obtained will be used by EPA to determine the existence and extent of threats to the public and the environment posed by hazardous waste disposal sites. The data may be used in civil and/or criminal litigation, therefore the strictest adherence of chain-of-custody protocol, document control, and quality assurance procedures is required.

SUMMARY OF DIOXIN SOW CHANGES

In Updating the 9/83 Version to the 9/86 Version

The method changes that have been made resulted in a large number of changes throughout the text. A summary of the changes is given below, a line by line listing of all changes has not been made.

1. The method has been extended to include water samples by adding the extraction and concentration steps from Method 613.

2. Spiking solutions are now mixed with 1.5 mL of acetone before addition to samples and blanks. This was required by the inclusion of water in the method.

3. The surrogate concentration has been changed and it is now used to monitor method detection limits.

4. The S/N ratios of the $^{13}C_{12}$-TCDD and native TCDD are no longer reportables. The S/N ratio of $^{37}Cl_4$-2,3,7,8-TCDD m/e 328 is a reportable.

5. The "Detection Limit" has been retitled "Maximum Possible Concentration" and redefined. The formula remains the same.

6. Concentration Calibration (CC) solution 5 has been eliminated, reducing the number of CC solutions to four and reducing the calibration range and working range of the method to 1 to 1000 ug/kg for soil/sediment and 0.01 to 10 ug/L for water. The working range includes using a smaller sample aliquot for the higher concentration samples (Exhibit D Section 1.1).

7. $^{13}C_{12}$-1,2,3,4-TCDD has been added as a recovery standard; added to the extract just before analysis to monitor performance of the analytical train. It is present in the CC solutions at 60% of the internal standard ($^{13}C_{12}$-2,3,7,8-TCDD) concentration, an advisory recovery window of 40-120% has been set for internal standard recovery. An action window may be set when sufficient data is avaiable.

8. The confirmatory period scan requirement and the DFTPP tune requirement have been dropped.

9. The forms have been redesigned.

10. The requirements for an acceptable method blank have been modified.

11. Two concentration calibration options have been provided to facilitate the inclusion of the recovery standard and use of existing stocks of CC solutions.

12. Numerous changes have been made for clarity and consistency with other CLP methods.

EXHIBIT A

SUMMARY OF REQUIREMENTS

Exhibit A - Summary of Requirements

1. The Contractor shall provide appropriate equipment and experienced personnel to identify and measure 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) in soil/sediment and water samples. These analyses require GC/MS/DS instrumentation including the capability to acquire, store, and retrieve selected-ion-monitoring data for six ions. Required equipment and expertise is specified in IFB Pre-Award Bid Confirmations. Samples to be analyzed may contain high levels of toxic or hazardous materials and must be stored and handled with appropriate precautions.

   Specific analytical procedures to be used are provided in Exhibit D. Specific QA/QC Requirements are specified in Exhibit E. These procedures must be followed explicitly without deviation, except as authorized in writing by the Contracting Officer. Required activities include:

   1.1 Sample receipt and handling under Chain-of-Custody procedures.

   1.1.1 Adherence to Chain-of-Custody procedures described in Exhibit G. Documentation described therein shall be required to show that all procedures are being strictly followed. This documentation shall be reported as the complete Case File Purge.

   1.2 Extract samples, perform column chromatography procedures, and concentrate extracts. Analyze extract aliquots with GC/MS procedures. Required equipment: capillary column, low resolution MS, and selected ion-monitoring data acquisition software. (See Exhibit D.)

   1.3 Acquire appropriate selected-ion-current profiles from MS data files. (See Exhibit D.)

   1.4 Using criteria specified in Exhibit D, determine the presence or absence of 2,3,7,8-TCDD in each sample. If present, calculate its concentration; if absent, calculate the maximum possible concentration.

   1.5 For each sample extract or blank, measure signal to noise ratio of the surrogate compound, $^{37}Cl_4$-2,3,7,8-TCDD, and determine if the analyte detectability requirement has been met. (See Exhibit E.)

   1.6 For each sample extract or blank calculate the percent recovery of the internal standard ($^{13}C_{12}$-2,3,7,8-TCDD) using the recovery standard ($^{13}C_{12}$-1,2,3,4-TCDD).

   1.7 Periodically analyze performance check solution and appropriate QC samples (blanks, duplicates, fortified samples, and performance evaluation samples), as specified in Exhibit E.

   1.8 Perform all required sample rerun extractions and analyses, as specified in Exhibit C.

1.9 Provide all reports and documentation within the applicable delivery requirement, as specified in Exhibit B.

1.10 After receipt of samples, retain unused portions of samples and sample extracts for six months as specified in Exhibit B.

2. The Contractor shall receive field samples in groups designated as sample "batches." There will be up to twenty-four (24) samples in a batch, as defined below.

2.1 Sample: A sample is defined as a solid material (soil or sediment) or a liquid material (water or rinsate) that is shipped to the Contractor for detection and measurement of 2,3,7,8-tetrachloro-dibenzo-p-dioxin (TCDD).

2.2 Field Blanks (described in Exhibit E, Sections 4.2 and 4.2.2): One or more field blank samples will be included in each sample batch.

2.2.1 Field Blank for Spiking -- One field blank for each matrix will be designated for spiking. The Contractor shall fortify this field blank with 2,3,7,8-TCDD at a concentration of 1 ug/kg for soil/sediment or 10 ng/L for water. After spiking, this field blank shall be analyzed by SIM GC/MS; it shall not be analyzed prior to spiking. If a sample is not designated for spiking, the laboratory shall call SMO immediately.

2.2.2 Unspiked Field Blanks -- Other field blanks, if any are included in the batch, shall be extracted and analyzed as routine samples per contract requirements.

2.3 Rinsate Sample (described in Exhibit E, Section 4.2.3): Normally, one rinsate sample will be included in each batch of samples, though more than one may be included. The rinsate sample is a portion of organic solvent that was used to rinse sampling equipment, and therefore, will be liquid in nature, rather than soil/sediment. The rinsate sample is to be prepared and analyzed as a routine sample per contract requirements.

3. Extraction/Analysis Requirements: Each sample in a batch shall be extracted and analyzed with selected ion-monitoring (SIM) GC/MS procedures. Additionally, for each batch of samples, three other analyses are required:

3.1 Laboratory Method Blank (described in Exhibit E, Section 4.1): The Contractor shall extract and analyze a laboratory method blank for each matrix in each batch of samples (or each time a group of samples are extracted) using contract-specified extraction and analysis procedures. NOTE: Method blank analysis is considered part of the required internal laboratory QA/QC (included in the sample unit price); method blank analysis is not considered as a separate sample analysis for contract accounting and/or billing purposes.

3.2 Duplicate Sample Analysis (described in Exhibit E, Section 5): One sample of each matrix in each batch will be designated for duplicate analysis. (In the event that no sample in the batch is marked for duplicate analysis, then the Contractor shall select sample(s) from the batch and perform duplicate analysis. DO NOT USE THE FIELD BLANK FOR DUPLICATE ANALYSIS.) A duplicate aliquot of this sample shall be extracted and analyzed using contract-specified extraction and analysis procedures. NOTE: Duplicate sample analysis is accountable and billable as a separate sample analysis.

3.3 Fortified Field Blank Analysis: One sample for each matrix in each batch should be identified as a field blank for spiking (see Exhibit A section 2.2.1). If no sample(s) are so designated the laboratory should contact SMO immediately.

4. Automatic Rerun Analyses (described in Exhibit C): Certain samples may require sample reruns (reextraction and/or reanalysis) either due to problems with the sample matrix or Contractor insufficiencies. NOTE: Sample reruns may be considered either as billable or non-billable under the terms of this contract, as defined in Exhibit C. For the purposes of this contract, the term "automatic rerun" shall signify only billable rerun analyses.

5. Summary of Batch Analyses:

5.1 Extractions --

24 or less field samples (including field blanks spiked at 1 ug/kg or 10 ng/L as appropriate)
1 or more laboratory method blanks*
1 or 2 duplicate samples
Plus, undetermined number of rerun samples.**

5.2 Analyses --

24 or less field samples (including field blanks spiked at 1 ug/kg or 10ng/L as appropriate)
1 or more laboratory method blanks*
1 or 2 duplicate samples
Plus, undetermined number of rerun samples.**

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*Not billable as separate sample extraction/analysis (see Section 3.1).
**All sample reruns may not be billable as separate sample extraction/analyses, depending on basis for rerun (see Section 4).

6. Contract Bid Lot: One bid lot consists of analysis of a maximum number of three thousand (3,000) samples, which will be received and analyzed in batches of less than or equal to twenty-four (24) field samples. The contractor will be required to perform a maximum number of 200 sample analyses per calendar month period.

7. Sample shipments to the Contractor's facility will be scheduled and coordinated by the EPA CLP Sample Management Office (SMO).

   7.1 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. This shall include immediately notifying SMO personnel of any irregularities with samples or sample paperwork received (noting discrepancies from verbal order placed by SMO), problems encountered in sample analyses that will affect the data produced, and laboratory conditions that impact on timeliness of analyses and data reporting. In particular, the Contractor shall notify SMO personnel in advance regarding sample data that will be late and shall specify an estimated delivery date.

   7.2 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case and Batch number. A Case signifies a group of samples collected at one site or geographical area over a predetermined 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. If a Case consists of multiple shipments, each shipment is considered a Batch.

   7.3 Each sample received by the Contractor should be labeled with an EPA sample number, and accompanied by a Dioxin Shipment Record bearing the sample number and descriptive information regarding each sample. The Contractor shall complete and sign the Dioxin Shipment Record, recording the date of sample receipt and sample condition on receipt for each sample container. The Contractor shall submit the signed copy of each Shipment Record to SMO within seven (7) calendar days following sample receipt (see contract delivery schedule). If there are problems either with the samples (e.g., mixed media, containers broken or leaking) or paperwork (e.g., Shipment Record not with shipment, sample and Shipment Record numbers do not correspond) the Contractor shall immediately contact SMO for resolution.

   7.4 The EPA Case and sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

   7.5 Samples will routinely be shipped to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing required for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station or other carrier service within the

Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays.

7.6 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.

7.7 The Contractor shall be required to return sample shipping containers (e.g., coolers) to the return addressee indicated on or within the container, within a period of fourteen (14) days following receipt of the sample shipment. The government will pay reasonable costs for the return of sample shipping containers by ground carrier service.

EXHIBIT B

REPORTING REQUIREMENTS AND DELIVERABLES

Exhibit B - Reporting Requirements and Deliverables

1. The Contractor shall provide reports and other deliverables as specified in the Contract Schedule. These reports are described below. All reports shall be submitted in legible form or resubmission shall be required. All reports and documentation required, including chromatograms, shall be clearly labeled with the EPA Case number, Batch number and associated sample/Dioxin Shipment Record number(s). If documentation is submitted without the required identification, as specified above, resubmission shall be required.

The Contractor shall provide all reports and deliverables as described below. The Contract Reporting Schedule (Section 2) specifies the number of copies required, delivery schedule and distribution of all required deliverables.

1.1 Dioxin Shipment Record: Copy of SMO Dioxin Shipment Record with lab receipt information and original Contractor signature.

1.2 Sample Data Summary Package: Hard copy analytical data and documentation are required from the Sample Data Package (Section 1.3).

1.2.1 Case Narrative

1.2.2 Completed data reporting sheets consisting of Forms B-1, B-2, B-3, and B-4. Original and rerun sample data shall be provided on Form B-1.

1.3 Sample Data Package: Hard copy analytical data and documentation are required as described below. NOTE: This analytical protocol is designed for receipt and analysis of samples by batch. Therefore, it is desired that sample data from samples in one batch be reported together, i.e., on the same reporting form. However, contract accounting and billing are based on the sample unit.

1.3.1 Case Narrative (laboratory cover letter) contains the Case number, Dioxin Shipment Record numbers, Contract number and detailed documentation of any Quality Control, sample, shipment and/or analytical problems encountered in a specific Case. Also included should be documentation of any internal decision tree process used along with a summary of corrective actions taken. The Case narrative must be signed in original signature by the Laboratory Manager or his designate.

1.3.2 Copies of completed Dioxin Shipment Records for all samples reported in data package.

1.3.3 Results of initial triplicate analyses of four (4) concentration calibration solutions, including all Selected Ion Current

Profiles (SICPs), Calculated Response Factors, plotted concentration calibration curves (see Section 9.2.6.5.7, Exhibit D), and computer generated quantitation reports.

1.3.4 Completed data reporting sheets (Forms B-1, B-2, B-3, and B-4) with appropriate SICPs. Data results of levels less than 10 ug/kg or 100 ng/L shall be reported to two (2) significant figures; results greater than 10 ug/kg or 100 ng/L shall be reported to three (3) significant figures. Apply the rounding rules found in Section 7.2.2, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019. Each SICP must include the following header information: date and time of analysis; instrument ID; and sample ID, i.e., EPA sample number, calibration solution number (CC1, CC2, CC3, or CC4) or column performance check solution (PC). When samples are analyzed more than once, all sample data shall be reported. (Note: Original and rerun data must be submitted.)

1.3.5 SICPs generated during each performance check solution analysis and each concentration calibration solution analysis.

1.3.6 A chronological list of all analyses performed. If more than one GC/MS system is used, a chronological list is required for each system. The list must provide the Data System File Name, the EPA sample number, and (if appropriate) the contractor laboratory sample number for each sample, blank, concentration calibration solution, and performance check solution. This list shall specify date and time of beginning of analysis. All sample/blank analyses performed during a 12-hour period must be accompanied by two performance check solution analyses, one preceding and one following sample/blank analyses. If multiple shifts are used, the ending performance check sample analysis from one 12-hour period may serve as the beginning analysis for the next 12-hour period.

1.4 Monthly Sample Status Report: The Monthly Sample Status Report shall provide the status of all samples the Contractor has received or has had in-house during the calendar month. Required status information includes: samples received, samples extracted, samples analyzed, samples rerun. All samples shall be identified by appropriate EPA Sample, Case and Batch/Shipment numbers.

1.5 Daily Sample Status Report: In response to verbal request from the Sample Management Office or the Project Officer, the Contractor must verbally provide sample status information on a same-day basis. Should written confirmation be requested, the Contractor must send daily sample status information in a written form that same day using first-class mail service. Required Daily Sample Status information shall include the items noted for the Monthly Sample Status Report and, in addition, shall require information on sample analysis reports in progress and analysis reports submitted/mailed.

1.6 GC/MS Tapes: The Contractor must store all raw GC/MS data (including samples, blanks, concentration calibration solutions, performance check solutions, and performance evaluation samples) on magnetic tape in appropriate instrument manufacturer's format. The Contractor shall maintain a written reference/logbook of tape files to EPA sample number, calibration data, standards and blanks. The reference must include EPA sample numbers identified by Case numbers and batch numbers. This reference/logbook shall accompany tapes when submitted.

The Contractor shall submit GC/MS tapes and associated reference/logbook within 7 days following receipt of written request by the EPA Project Officer or Sample Management Office. The Contractor shall retain tapes for at least 365 days after data submission unless submission is requested during that time.

1.7 Extracts and Unused Sample Volume: Unused portions of samples and all sample extracts shall be retained by Contractor for a period of 365 days after data is submitted. Extracts shall be stored at 4°C; unused portions of samples can be sealed and stored at ambient temperature. Extracts and unused sample volume containers shall be labeled with EPA sample number, Case number and Batch number. A logbook of stored extracts and sample volume shall be maintained, listing EPA sample numbers and associated Case and Batch numbers.

The Contractor shall submit sample extracts and/or unused sample volume and associated logbook(s) within 7 days following receipt of written request by the EPA Project Officer or Sample Management Office. The Contractor shall retain extracts and unused sample volume for at least 365 days after data submission unless submission is requested during that time.

NOTE: The Contractor is responsible for shipment of these materials in accordance with applicable Department of Transportation regulations. Whenever the Contractor disposes of such materials, the Contractor is responsible for disposition of these materials in accordance with applicable environmental regulations.

1.8 Complete Case File Purge: (formerly called the Document Control and Chain-of-Custody Package) The Complete Case File Purge package includes all laboratory records received or generated for a specific Case or sample batch, that have not been previously submitted to EPA as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence, and the document inventory. (See Exhibit F.)

2. The following table (2.1) reiterates the contract reporting and deliverables requirements specified in the Contract Schedule and specifies the distribution that is required for each deliverable. Recipients include the CLP Sample Management Office, EM~~SL~~/LV QA.

Division, the appropriate Regional Technical Officer, and NEIC Contract Evidence Audit Team. NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The Project Offier will notify the contractor in writing of such changes when they occur.

2.1 Contract Reporting Schedule

CONTRACT REPORTING SCHEDULE

ITEM NO.	REPORT	# COPIES	DELIVERY SCHEDULE	REPORT DISTRIBUTION SMO	EMSL/LV	REGION	NEIC
1	Dioxin Shipment Record	1	7 Days After Validated Time of Sample Receipt (VTSR)	X			
2	Sample Data Summary Package	1	21 days after VTSR	X			
3	Sample Data Package	3	21 days after VTSR	X	X	X	
4	Monthly Sample Status Report	2	5 days following end of each calendar month	X	X		
5	Daily Sample Status Report	1	Verbal and/or written; upon request by PO or SMO; maximum frequency is daily	As Directed			
6	GC/MS Tapes	Lot	Retain for 365 days after data submission or submit within 7 days after receipt of written request by PO or SMO	As Directed			
7	Extracts & Unused Sample Volume	Lot	Retain for 365 days after data submission or submit within 7 days after receipt of written request by PO or SMO	As Directed			
8	Complete Case File Purge	1 Pkg	Submit 180 days after data submission or within 7 days after receipt of written request by PO or SMO				X

NOTE: ALL RESULTS SHALL BE REPORTED TOTAL AND COMPLETE. Concurrent delivery of items 2 and 3 is required.

2.2 Addresses for Distribution

SMO

CLP Sample Management Office
P. O. Box 818
Alexandria, VA 22313

For overnight deliveries, use street address:
300 N. Lee St., Suite 200
Alexandria, VA 22314

EMSL/LV

USEPA EMSL/LV QA Division
Box 15027
Las Vegas, NV 89114
ATTN: Data Audit Staff

For overnight deliveries, use street address:
944 E. Harmon Ave.
Executive Center, Rm. 226
Las Vegas, NV 89109

Region

Following contract award and prior to Contractor's receipt of the first batch of samples, the Sample Management Office, acting on behalf of the Project Officer, will provide the Contractor with the list of Deputy Project Officers for the 10 EPA Regions. SMO will provide the Contractor with updated Regional address/name lists as necessary throughout the period of the contract.

NEIC

NEIC Contract Evidence
Audit Team (CEAT)
12600 West Colfax, Suite C310
Lakewood, CO 80215

FORM B-1S. TCDD SOIL DATA REPORT FORM

Lab: _____ Report Date: _____

Case/Batch No: _____ Column: _____

Instrument ID: _____

EPA Sample No.	Extr. Date	Wet wt	ug/kg Meas.	TCDD MPC	GC/MS Date	Analysis Time	Surr. S/N Ratio	* % REC(IS)

MB = Method Blank
N = Native TCDD Spike
D = Duplicate/Fortified Field Blank
PE = EMSL-LV Performance Evaluation Sample
MPC = Maximum Possible Concentration
*Note: Relative to $^{13}C_{12}$-1,2,3,4-TCDD

FB = Field Blank
IS = Internal Standard
RR = Rerun
RS = Recovery Standard
ND = Not Detected

Lab: _____ Report Date: _____

Case/Batch No: _____ Column: _____

Instrument ID: _____

EPA Sample Number	Rel. Response Ratios 320/322	332/334IS	332/334RS	Response (Area) 259	320	322	328	332IS	334IS	332RS	334RS

MB = Method Blank
N = Native TCDD Spike
D = Duplicate/Fortified Field Blank
PE = EMSL-LV Performance Evaluation Sample
MPC = Maximum Possible Concentration

FB = Field Blank
IS = Internal Standard
RR = Rerun
ND = Not Detected
RS = Recovery Standard

Lab: _____ Report Date: _____

Case/Batch No: _____ Column: _____

Instrument ID: _____

EPA Sample No.	Extr. Date	volume	ng/L Meas.	TCDD MPC	GC/MS Date	Analysis Time	Surr. S/N Ratio	* % REC(IS)

MB = Method Blank
N = Native TCDD Spike
D = Duplicate/Fortified Field Blank
PE = EMSL-LV Performance Evaluation Sample
MPC = Maximum Possible Concentration
*Note: Relative to $^{13}C_{12}$-1,2,3,4-TCDD

FB = Field Blank
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ND = Not Detected

Lab: _____ Report Date: _____

Case/Batch No: _____ Column: _____

Instrument ID: _____

EPA Sample Number	Rel. Response Ratios 320/322	332/334IS	332/334RS	Response (Area) 259	320	322	328	332IS	334IS	332RS	334RS

MB = Method Blank
N = Native TCDD Spike
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PE = EMSL-LV Performance Evaluation Sample
MPC = Maximum Possible Concentration
FB = Field Blank
IS = Internal Standard
RR = Rerun
ND = Not Detected
RS = Recovery Standard

A. TCDD REPORT FORM (Form B-1)

This form is used for tabulating and reporting case results.

Complete the header information at the top of the page including instrument ID, laboratory name, case/batch number, report date, and column used.

EPA sample number is tabulated along with date sample was extracted, and weight (wet) extracted to the nearest tenth (0.1) of a gram or volume extracted (water) to the nearest 10 milliliters.

Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_X = \frac{A_X \cdot Q_{IS}}{A_{is} \cdot RRF_n \cdot W}$$

C_X = 2,3,7,8-TCDD concentration in ug/kg or ug/L

A_X = the sum of integrated ion abundance detected for m/z 320 and 322

A_{is} = the sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of $^{13}C_{12}$-2,3,7,8-TCDD the internal standard).

Q_{is} = quantity (in ng) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction

RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD

W = The weight (in g) of soil/sediment extracted or volume of water extracted (in mL)

Positive samples are quantitated with values >10.0 ug/kg or 100 ng/L recorded to three (3) significant figures and those values <10.0 ug/kg or 100 ng/L reported to two (2) significant figures.

For samples in which unlabeled 2,3,7,8-TCDD was not detected calculate the estimated maximum possible concentration, which is the concentration required to produce a signal with a peak height of 2.5 times the background signal height.

Use the formula:

$$MPC = \frac{2.5 \cdot H_X \cdot Q_{is}}{H_{is} \cdot RRF_n \cdot W}$$

where: MPC = maximum possible concentration of unlabeled 2,3,7,8-TCDD required to produce H_X.

H_x = peak height for m/z 320 or 322 in the same group of >5 scans used to measure A_{is}.

H_{is} = peak height for the appropriate ion characteristic of the internal standard, m/z 332 when 320 is used to determine A_x, and m/z 334 when 322 is used to determine A_x.

Q_{is} = quantity (in ng) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction.

RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD.

W = weight (in g) of wet soil/sediment sample or volume of water extracted (in mL).

Report GC/MS Instrument ID, the date and time the analysis was performed, and the signal-to noise ratio for the surrogate compound.

INITIAL CALIBRATION SUMMARY

Laboratory: _____ CC Solution Alternative: _____

Case/Batch No.: _____ Instrument ID: _____

Date	Time	Sol. ID	AREA 320	322	328	332IS	334IS	332RS	334RS
		CC1 CC1 CC1							
		CC2 CC2 CC2							
		CC3 CC3 CC3			*				
		CC4 CC4 CC4			† / / † / / / † / †				

Solution ID Codes:

CC1 = Concentration calibration solution #1
CC2 = Concentration calibration solution #2
CC3 = Concentration calibration solution #3
CC4 = Concentration calibration solution #4

* Not present in CC Solution Alternative One.

INITIAL CALIBRATION SUMMARY

Laboratory: _____ CC Solution Alternative: _____

Case/Batch No.: _____ Instrument ID: _____

Date	Time	Sol. ID	Measured RRF_n	Mean RRF_n	Measured RRF_i	Mean RRF_i
		CC1 CC1 CC1				
		CC2 CC2 CC2				
		CC3 CC3 CC3				
		CC4 CC4 CC4				

Solution ID Codes:

CC1 = Concentration calibration solution #1
CC2 = Concentration calibration solution #2
CC3 = Concentration calibration solution #3
CC4 = Concentration calibration solution #4

%RSD:	RRF_n	RRF_i
CC1=	_____	_____
CC2=	_____	_____
CC3=	_____	_____
CC4=	_____	_____

Native Mean of Means: _____

IS Mean of Means: _____

B. Initial Calibration Summary (Form B-2)

Record all routine calibrations (PCS and CCl) performed during initial calibration on form B-3.

Complete all header information including laboratory, case/batch number, and instrument ID and EPA CC Solution Alternative.

Date and time along with response for each ion is recorded for each calibration solution. The response factors are calculated with the following equations:

RRF_n (native Response Factor) RRF_i (internal Standard Response Factor)

$$RRF_n = \frac{A_x \cdot Q_{is}}{A_{is} \cdot Q_n}$$

$$RRF_i = \frac{A_{is} \cdot Q_{rs}}{A_{rs} \cdot Q_{is}}$$

Where:

A_x = the sum of integrated ion abundance of m/z 320 and 322 for unlabeled 2,3,7,8-TCDD

A_{is} = the sum of integrated ion abundancces of m/z 332 and m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD

A_{rs} = the sum of integrated ion abundance of m/z 332 and m/z 334 for $^{13}C_{12}$-1,2,3,4-TCDD

Q_n = quantity of unlabeled 2,3,7,8-TCDD injected

Q_{is} = quantity of $^{13}C_{12}$-2,3,7,8-TCDD injected

Q_{rs} = quantity of $^{13}C_{12}$-1,2,3,4-TCDD

Calculate the mean RRF and the percent relative standard deviation for the triplicate runs of each calibration solution.

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

Where:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(X_i - \bar{X})^2}{N - 1}}$$

$\bar{X}$ = mean of each of the three Response Factors respectively

From the 4 mean native response factors and 4 mean internal standard response factors: calculate the mean of means for each respective RRF's.

FORM B-3

ROUTINE CALIBRATION SUMMARY

Laboratory: _____ CC Solution Alternative: _____

Case/Batch No.: _____ Instrument ID: _____

	(PCS) PERFORMANCE CHECK SOL.						(CC1) CON. CALIB. SOL. #1		
Date									
Time									
Response									
259									
320									
322									
328									
332IS									
334IS									
332RS									
334RS									
Ratios									
320/322									
332/334IS									
332/334RS									
RRF_n	—	—	—	—	—	—			
RRF_i	—	—	—	—	—	—			
% Valley							—	—	—

C. Routine Calibration Summary (Form B-3)

Complete the header information including the laboratory, instrument ID Case/Batch number and EPA CC Solution Alternative.

For each performance check solution analyzed complete the date and time of analysis, the response for m/z 259, 320, and 322 for unlabeled 2,3,7,8-TCDD, 328 for $^{37}Cl_4$-2,3,7,8-TCDD, and 332 and 334 for $^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-1,2,3,4-TCDD.

Ion ratios for m/z 320/322, m/z 332/334 for $^{13}C_{12}$-2,3,7,8-TCDD and m/z 332/334 for $^{13}C_{12}$-1,2,3,4-TCDD are to be calculated and recorded.

Response factors are to be calculated as in the Initial Calibration Summary (Section B).

For calculation of valley percent see Section D, Section 9.2.6.1.

For each Concentration Calibration Solution #1 used in Routine Calibration, complete all the above information.

FORM B-4

QUALITY CONTROL SUMMARY

Laboratory Name_____ Case/Batch No._____

Instrument ID_____

SOIL

Accuracy, Fortified/
Spike Field Blank: _____ EPA Sample Number: _____

Relative Difference (%),
Duplicate Analysis: _____ EPA Sample Number: _____

WATER

Accuracy, Fortified/
Spike Field Blank: _____ EPA Sample Number: _____

Relative Difference (%),
Duplicate Analysis: _____ EPA Sample Number: _____

D. QC Summary

Complete all the header information.

Report the sample number for the fortified field blank and the % accuracy of the fortified/spike field blank by using the following equation:

$$\% \text{ accuracy} = \frac{\text{amount measured}}{1.0} \times 100$$

Record the sample used for duplicate and the Relative Percent Difference which is calculated as follows:

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

Where:

S_1 and S_2 represent sample and duplicate sample results.

EXHIBIT C

SAMPLE RERUN REQUIREMENTS

Exhibit C - Sample Rerun Requirements

1. Scope and Application

The Contractor shall be required to reextract and reanalyze certain samples or batches of samples in a variety of situations that may occur in the process of contract performance. (For purposes of this contract, the term "rerun" shall indicate sample reextraction, cleanup and reanalysis.)

In situations where the rerun is required due to matrix effects, interferences or other problems encountered because of difficult samples, the Government will pay the Contractor for the reruns. Such reruns shall be billable and accountable under the specified contract allotment of automatic reruns.

In situations where the rerun is required due to Contractor materials, equipment or instrumentation problems, or lack of contractor's adherence to specified contract procedures, the rerun shall not be billable nor accountable under the terms of the contract.

Contractor's failure to perform any of the sample reruns specified herein, either billable or non-billable, shall be construed as Contractor nonperformance and may result in termination of the contract for default by Contractor.

NOTE: The only circumstance that may require more than one rerun per sample is a contaminated method blank.

2. Required Sample Reruns

2.1 Automatic sample reruns, billable as such under the contract.

2.1.1 If the calculated unlabeled TCDD amount was outside the upper initial calibration range, the Contractor shall reextract the sample using a smaller sample aliquot, and reanalyze the sample. (See Section 12.1.2, Exhibit D.)

2.1.2 If the internal standard was not found to be present with at least 10/1 signal to noise ratio at mass 332 and 334, the Contractor shall reextract and reanalyze the sample.
NOTE: This rerun is billable only if the Contractor can demonstrate that the internal standard was added to the sample in accordance with contract specifications. (See Sections 3.11 and 11.6.3, Exhibit D.)

2.1.3 If the internal standard 332/334 ratio was outside of the contract specified control limits of 0.67-0.90, the Contractor shall reextract and reanalyze the sample. (See Section 11.6.5, Exhibit D.) This reanalysis is billable, only if the internal standard 332/334 ratio is still outside the 0.67-0.90 control limits.

2.1.4 If the isotope abundance ratio for m/z 320/322 is less than 0.67 or greater than 0.90 and all other criteria contained in Section 11.6.1 through 11.6.5 of Exhibit D are met, then the sample must be rerun unless the MPC is $\leq$0.3 ug/Kg for soil or 0.003 ug/L. (See Section 6.2, Exhibit E.) NOTE: This reanalysis is billable only if the isotope abundance ratio of m/z 320/322 on the rerun is still outside the criteria.

2.1.5 If the estimated maximum possible concentration is greater than 1 ug/kg (soil) or 10 ng/L (water), the Contractor shall reextract and reanalyze the sample. This reanalysis is billable only if the maximum possible concentration is still greater than 1 ug/kg or 10 ng/L.

2.2 Sample reruns to be performed at Contractor's expense (i.e., not billable under terms of contract).

2.2.1 Acceptable laboratory method blanks must not contain any signal at 320, 322 or 259 which is greater than 2% of the m/z 332 response within $\pm$5 scans of the m/z 332 peak maximum. The Contractor shall reanalyze the affected positive samples associated with a contaminated blank (see Section 4.1.5, Exhibit E).

2.2.2 If the performance check (PC) solution does not meet specified criteria, the Contractor shall reanalyze all positive samples run during the time period between the last acceptable PC run and the unacceptable PC run (see Section 2.4, Exhibit E).

2.2.3 If a false positive is reported for an uncontaminated soil or water (blind QC) sample, upon notification by the Sample Management Office the Contractor shall reextract and reanalyze all positive samples in the associated batch of samples (see Section 7.1.1, Exhibit E).

2.2.4 If the analytical results for a performance evaluation blind QC sample fall outside of EPA-established acceptance windows, upon notification by the Sample Management Office the Contractor shall reextract and reanalyze the entire associated batch of samples (see Section 7.4.1, Exhibit E).

2.2.5 If the accuracy of the measured concentration of native 2,3,7,8-TCDD in the spiked (fortified) field blank is not between 60-140%, the contractor shall reextract and reanalyze a second aliquot of the fortified field blank sample.

3. Sample Rerun QC

3.1 A native spike and duplicate shall be performed for each batch of samples reanalyzed as specified in Section 2.

3.1.1 If a concurrent Dioxin Case is being processed, the native spike and duplicate from that case may be shared with the rerun samples if the total number of samples does not exceed 24. If the total number of samples exceeds 24 an additional native spike and duplicate must be analyzed as in Section 3.1.2 below. The native spike and duplicate data shall be reported in each Case Data Package. (Note: The QC samples are to be billed only under the Case number in which the QC sample was received.)

3.1.2 If no other Dioxin Case is being processed at the time of reanalysis, the native spike and duplicate shall be chosen from the Case and batch in which the rerun sample belongs. The QC samples are to be billed only if the rerun samples are billable according to Section 2.

EXHIBIT D

ANALYTICAL METHODS

INDEX

Exhibit D - Analytical Methods

2,3,7,8-Tetrachlorodibenzo-p-dioxin in Soil, Sediment and Water by High Resolution Gas Chromatography/ Low Resolution Mass Spectrometry

1. SCOPE AND APPLICATION

1.1 This method provides procedures for detection and measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD; CAS Registry Number 1746-01-6; STORET Number 34675) at concentrations of 1 ug/kg to 100 ug/kg in 10-g aliquots of wet soil/sediment and 10 ng/L to 1000 ng/L in 1 L aliquots of water. The use of 1 g aliquots permits measurement of concentration up to 1000 ug/kg in soil/sediment. The use of a 100 mL aliquot volume permits measurement of concentrations up to 10 ug/L in water.

1.2 CAUTION: The analysis of water samples includes whatever particulates may be present. The estimated solubility of 2,3,7,8-TCDD in water is less than 50 ng/L[7], therefore positive values above this level should be considered to be a function of the TCDD associated with the particulates rather than the water.

1.3 The minimum measurable concentration is estimated to be 0.3 ug/kg or 3 ng/L, but is dependent on interfering compounds present in the sample matrix.

1.4 This method is designed for use by analysts who are experienced in the use of a gas chromatograph/mass spectrometer.

1.5 CAUTION: Because 2,3,7,8-TCDD is extremely toxic, safety procedures described in Section 5 of this method should be followed to prevent exposure of laboratory personnel to materials containing this compound.

2. SUMMARY OF METHOD

2.1 Soil/Sediment Extraction: For purposes of this contract a soil/sediment sample is defined as a portion of wet soil or sediment which may contain other solids such as stones, vegetation etc., but should not contain an obvious liquid phase (See Exhibit D, Section 8.3.2). Fifty (50) ng of $^{13}C_{12}$-labeled 2,3,7,8-TCDD and 1.4 ng of $^{37}Cl_4$-labeled 2,3,7,8TCDD are added to a 10 g aliquot of wet soil or sediment sample, the sample aliquot is mixed with 20 g of anhydrous sodium sulfate and is extracted with a mixture of hexane and methanol by agitating the sample aliquot and solvent continually in a glass jar.

2.2 Water Extraction: For the purpose of this contract a water sample is defined as a single phase system that is primarily water but may contain small amounts of floating, suspended, and settled particulate matter. Multiple phases should not be present (See Exhibit D, Section 8.3.2.). Fifty (50) ng of $^{13}C_{12}$-labeled 2,3,7,8-TCDD and 1.4 ng of $^{37}Cl_4$-labeled 2,3,7,8TCDD are added to approximately 1 L of water and extracted with methylene chloride using a separatory funnel. The methylene chloride extract is exchanged to hexane during concentration.

2.3 Cleanup and Analysis: Column chromatographic procedures are used to help eliminate sample components that may interfere with detection and measurement of 2,3,7,8-TCDD. The extract is concentrated to 50 uL, 50 ng $^{13}C_{12}$-1,2,3,4-TCDD are added, and a 2 uL aliquot is injected into a fused silica capillary column in a gas chromatograph (GC) interfaced to a mass spectrometer (MS) that has at least unit resolution at m/z 334. Identification of 2,3,7,8-TCDD is based on detection of three ions, measurement of the appropriate relative abundances of two characteristic ions in the molecular ion cluster, and determination of the retention time of the sample analyte relative to the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD, contained in the sample extract. The 2,3,7,8-TCDD concentration is determined by measuring the MS response to the sample component relative to the MS response to $^{13}C_{12}$-2,3,7,8-TCDD (the internal standard). The labeled internal standard method presumes that internal standard losses during method procedures are equal to unlabeled TCDD losses. Therefore, the calculated sample 2,3,7,8,-TCDD concentration is corrected for losses during sample preparation.

The $^{37}Cl_4$-2,3,7,8-TCDD is a surrogate compound that is added to each sample and is analyzed exactly the same as unlabeled TCDD. The surrogate compound is used to determine that the detection criteria for unlabeled 2,3,7,8-TCDD in the same sample have been met.

The $^{13}C_{12}$-1,2,3,4-TCDD is a recovery standard that is added to each sample and blank. The recovery of the internal standard ($^{13}C_{12}$-2,3,7,8-TCDD) and the surrogate compound ($^{37}Cl_4$-2,3,7,8-TCDD) are related to the precision and sensitivity of the analysis for unlabeled 2,3,7,8-TCDD in the sample.

3. DEFINITIONS

3.1 Concentration calibration solution -- a solution containing known amounts of the analyte (unlabeled 2,3,7,8-TCDD), the surrogate compound ($^{37}Cl_4$-2,3,7,8-TCDD), the recovery standard ($^{13}C_{12}$-1,2,3,4-TCDD) and the internal standard ($^{13}C_{12}$-2,3,7,8-TCDD); it is used to determine the instrument response of the analyte compound relative to the internal standard and the recovery of the internal standard.

3.2 Field blank -- a portion of soil/sediment or water uncontaminated with 2,3,7,8-TCDD submitted with the samples.

3.3 Rinsate -- a portion of trichloroethylene used to rinse sampling equipment and analyzed to demonstrate that samples were not contaminated during sampling.

3.4 Internal standard -- $^{13}C_{12}$-2,3,7,8-TCDD, which is added to every sample and is present at the same concentration in every blank, quality control sample, and concentration calibration solution. It is added to the samples before extraction and is used to measure the concentration of the analyte.

3.5 Recovery standard -- $^{13}C_{12}$-1,2,3,4-TCDD is added to every extract and is present in all standards. It is added to every extract just before analysis and is used to measure the recovery of the internal standard.

3.6 Surrogate compound -- A known concentration of $^{37}Cl_4$-2,3,7,8-TCDD, which is added to all samples before analysis. Its signal to noise ratio is measured in each sample, and is used to indicate that unlabeled 2,3,7,8-TCDD is detectable at less than 1.0 ug/kg or 10 ng/L.

3.7 Laboratory method blank -- a blank prepared in the laboratory by performing all analytical procedures except addition of a sample aliquot to the extraction vessel.

3.8 Performance check mixture -- a mixture of known amounts of selected standard compounds; it is used to demonstrate continued acceptable performance of the GC/MS/DS system.

3.9 Performance evaluation sample -- a soil/sediment or water sample containing a known amount of unlabeled 2,3,7,8-TCDD. It is distributed by EPA to potential contractor laboratories who must analyze it and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). It may also be included as an unspecified QC sample in any sample batch submitted to the laboratory for analysis.

3.10 Response factor -- response of the mass spectrometer to a known amount of an analyte relative to a known amount of an internal standard.

3.11 Signal-to-noise (for the purpose of this contract) is defined as the ratio of analyte signal to random background signal. Display each characteristic ion using a window 20 scans wide and centered around the elution time of 2,3,7,8-TCDD. Draw a base line from the lowest point in the 20 scan window. The noise is defined as the height of the largest signal (excluding signal due to TCDD or other chemicals) on either side of the 2,3,7,8-TCDD peak, within the 20 scan window. The signal is defined as the height of 2,3,7,8-TCDD peak. Chemical noise is left to the judgement of the analyst.

3.12 Abbreviations

TCDD - Tetrachlorodibenzo-p-dioxin
GC - Gas Chromatograph
MS - Mass Spectrometer
DS - Data System
SIM - Selected Ion Monitoring
SMO - Sample Management Office
SICP - Selected Ion Current Profile
S/N - Signal to Noise
PC - Performance Check Standard
CC - Calibration Check Standard
RRF - Relative Response Factor
IS - Internal Standard
RS - Recovery Standard
K-D - Kuderna-Danish Apparatus
OD - Outside Diameter
m/z - Mass to Charge Ratio
MPC - Maximum Possible Concentration
VTSR - Verified Time of Sample Receipt

4. INTERFERENCES

Any compound that yields ions at m/z 259, 320, 322, or 328 and also elutes within 10 scans of the internal standard is a potential interference. Most frequently encountered interferences are other sample components that are extracted along with TCDD. Because very low levels of TCDD must be measured, elimination of interference is essential. High purity reagents and solvents must be used and all equipment must be scrupulously cleaned. Laboratory method blanks (Exhibit E, Quality Control, Section 4) must be analyzed to demonstrate lack of contamination that would interfere with TCDD measurement. Column chromatographic procedures are used to remove some coextracted sample components; these procedures must be performed carefully to minimize loss of TCDD during attempts to enrich its concentration relative to other sample components.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a file of current OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are identified.[1-3] 2,3,7,8-TCDD has been identified as a suspected human or mammalian carcinogen.

5.2 Each laboratory must develop a strict safety program for handling 2,3,7,8-TCDD. The following laboratory practices are recommended:

5.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.

5.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or through a trap containing oil or highboiling alcohols.

5.3 The following precautions for safe handling of 2,3,7,8-TCDD in the laboratory are presented as guidelines only, and are based on safe handling practices included in USEPA Method 613.[4] The precautions for safe handling and use are necessarily general in nature because detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual usage. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. Although 2,3,7,8TCDD is extremely toxic to laboratory animals, it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

5.3.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and lab hood adequate for radioactive work.

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

5.3.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift) with any mild soap and plenty of scrubbing action.

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

5.3.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors should not handle wastes.

5.3.6 Disposal of Wastes: 2,3,7,8-TCDD decomposes above 800°C. Low level waste, such as the absorbent paper and plastic gloves, may be burned in a good incinerator. Waste containing gross quantities (milligrams) of 2,3,7,8-TCDD should be packaged securely and disposed through commercial or governmental

channels that are capable of handling high-level or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container; residues may then be handled as above.

5.3.7 Glassware, Tools, and Surfaces: Satisfactory cleaning may be accomplished by rinsing with 1,1,1-trichloroethane, then washing with any detergent and water. Dishwater may be disposed to the sewer. (Also see Section 6.5.)

5.3.8 Laundry: Clothing known to be contaminated should be disposed with the precautions described under Section 5.3.6. Lab coats or other clothing worn in 2,3,7,8-TCDD work may be laundered. Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing. Disposable garments may be used to avoid a laundry problem, but they must be properly disposed or incinerated.

5.3.9 Wipe Tests: A useful method to determine cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, which is extracted and analyzed by gas chromatography (limit of sensitivity of approximately 0.1 ug per wipe). Less than 4 pg/cm^2 2,3,7,8-TCDD indicates acceptable cleanliness; anything higher warrants further cleaning. More than 400 pg/cm^2 indicates an acute hazard that requires prompt cleaning before further use of the equipment or work space and indicates that unacceptable work practices have been employed in the past.

5.3.10 Inhalation: Any procedure that may produce airborne contamination should be performed with good ventilation. Gross losses to a ventilation system should not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in case of an accident. Finely divided soils contaminated with 2,3,7,8-TCDD are hazardous because of the potential for inhalation. Such samples should be handled in a confined environment, such as a hood or glove box, or laboratory personnel should wear masks fitted with a particulate filter and charcoal sorbent.

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

6. APPARATUS AND EQUIPMENT

6.1 Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/DS)

6.1.1 The GC must be capable of temperature programming and be equipped with all required accessories, such as syringes, gases, and a capillary column. The GC injection port must be designed for capillary columns. Splitless or on-column injection technique is recommended. With this method, a 2 uL injection volume is used consistently. However, with some GC injection ports other volumes may be more appropriate. Any volume that produces adequate precision sensitivity, and chromatographic separation may be used. A 1 uL injection volume may be used if adequate sensitivity and precision can be achieved. CAUTION: The injection volume for all extracts, blanks, calibration solutions and the performance check samples must be the same.

6.1.2 Mass spectral data are obtained with electron ionization at a nominal electron energy of 70 eV. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five data points for each of six ions while a sample component elutes from the GC.

6.1.3 An interfaced data system (DS) is required to acquire, store, reduce and output mass spectral data. The DS must be equipped with a selected ion monitoring (SIM) program to acquire data for at least six ions that are characteristic of labeled and unlabeled 2,3,7,8-TCDD. (The mass spectrum of unlabeled 2,3,7,8-TCDD is shown in Figure 1 at the end of this Exhibit.) The same integration time must be used for each ion monitored, and the integration time used for sample analyses must be the same as the time used to analyze concentration calibration solutions and the performance check solution. Total data acquisition time per cycle (six ions) must not exceed 1.5 seconds.

6.1.4 The Contractor shall use a magnetic media storage device capable of recording data suitable for long-term off-line storage. The Contractor shall record all raw GC/MS data acquired during the entire contract period on magnetic media in appropriate instrument manufacturer format. The Contractor shall provide the data on 9-track magnetic tape in appropriate instrument manufacturers format to the US EPA within seven (7) days of request by the Project Office or SMO. The tapes must be retained by the contractor for 180 days after data package submission unless requested by EPA.

6.2 GC Column -- Two fused silica capillary columns are recommended; one is a 60-m SP-2330 and the other is a 50-m CP-SIL 88. Any capillary column that separates 2,3,7,8-TCDD from all other TCDDs may be used, but this separation must be demonstrated. Minimum acceptance criteria must be determined per Section 9.2.6.1. At the beginning and end of

each 12-hour period during which sample or concentration calibration solutions will be analyzed, column operating conditions must be demonstrated to achieve the required separation on the column to be used for samples. Operating conditions known to produce acceptable results with the recommended columns are shown in Table 1 at the end of this Exhibit. It is the Contractor's responsibility to verify whether the information in Table 1 is suitable for the laboratory's instrument(s).

6.3 Miscellaneous Equipment

6.3.1 Nitrogen evaporation apparatus with variable flow rate.

6.3.2 Mechanical shaker -- A magnetic stirrer or a wrist-action or platform-type shaker that produces vigorous agitation.

6.3.3 Analytical balance capable of accurately weighing 0.01 g.

6.3.4 Centrifuge capable of operating at 400 x G.

6.3.5 Water bath -- equipped with concentric ring cover and temperature controlled within $\pm 2^{\circ}C$.

6.3.6 Stainless steel spatulas or spoons.

6.3.7 Stainless steel (or glass) pan large enough to hold contents of 1-pint sample containers.

6.3.8 Glove box.

6.4 Glassware

6.4.1 Extraction jars -- amber glass with Teflon-lined screw cap; minimum capacity of approximately 500 mL; must be compatible with mechanical shaker to be used.

6.4.2 Kuderna-Danish apparatus -- 500-mL evaporating flask, 10-mL graduated concentrator tubes with ground-glass stoppers, 3-ball macro-Snyder column, and 2-ball micro-Snyder column.

6.4.3 Culture tubes -- 8-mL glass.

6.4.4 Mini-vials -- 1-mL amber borosilicate glass with conical-shaped reservoir and screw caps lined with Teflon-faced silicone disks.

6.4.5 Funnels -- glass; appropriate size to accommodate filter paper used to filter jar extract (volume of approximately 170 mL).

6.4.6 Chromatography columns -- 1 cm ID x 20 cm long and 1 cm ID x 30 cm long.

6.4.7 Separatory funnels -- 2 L with Teflon stopcock.

6.4.8 Drying column 19 mm ID glass chromatographic column with a coarse frit (a small pad of pyrex glass wool may be substituted for the frit to avoid cross contamination).

6.4.9 Boiling chips -- Approximately 10/40 mesh. Silicon carbide or Teflon may be used. Heat to 400°C for 30 min or Soxhlet extract with methylene chloride as appropriate.

6.5 NOTE: Reuse of glassware should be minimized to avoid the risk of using contaminated glassware. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure. Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water. Drain dry and heat in a muffle furnace at 400°C for 15 to 30 min. Volumetric glassware should not be heated in a muffle furnace, and some thermally stable materials (such as PCBs) may not be removed by heating in a muffle furnace. In these cases, rinsing with high-purity acetone and hexane may be substituted for muffle furnace heating. After glassware is dry and cool, store inverted or capped with aluminum foil in a clean environment.

CAUTION: The analysis for 2,3,7,8-TCDD in water is for much lower concentrations than in soil/sediment. Extreme care must be taken to prevent cross-contamination between soil and water samples. It is strongly recommended that separate glassware be reserved for analyzing water samples. It is recommended that all glassware be rinsed with solvent immediately before use and that the pooled solvent for a set of extractions be concentrated and analyzed as a method of demonstrating that the glassware was free of contamination.

7. REAGENTS AND CONSUMABLE MATERIALS

7.1 Column Chromatography Reagents

7.1.1 Alumina, acidic AG4, Bio Rad Laboratories (catalog #132-1240 or equivalent) -- Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil covered glass container for 24 hours at 190°C.

7.1.2 Silica gel -- high purity grade, type 60, 70-230 mesh; Soxhlet extract with methylene chloride for 21 hours and activate by heating in a foil-covered glass container for 24 hours at 130°C.

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

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

7.1.5 Sulfuric acid, concentrated -- ACS grade, specific gravity 1.84.

7.1.6 Graphitized carbon black (Carbopack C or equivalent), surface area of approximately 12 m2/g, 80/100 mesh.

7.1.7 Celite 545, reagent grade, or equivalent.

7.2 Filter paper -- Whatman No. 1 or equivalent; rinse with hexane before use.

7.3 Glass wool, silanized -- Extract with methylene chloride and then hexane before use.

7.4 Sodium sulfate -- Granular, anhydrous; before use, extract with methylene chloride and dry for >4 h in a shallow tray placed in an oven operated at 120°C.

7.5 Solvents -- High purity, distilled-in-glass; hexane, methanol, methylene chloride, toluene, and isooctane.

7.6 Concentration Calibration Solutions (Table 2) -- EMSL-LV will provide the concentration calibration solutions in either of two formulations depending on the availability of standard materials. Alternative one (Section 7.6.1) includes the $^{13}C_{12}$-1,2,3,4-TCDD recovery standard in the concentration calibration solutions. Alternative two (Section 7.6.2 requires the addition of a specified amount of $^{13}C_{12}$-1,2,3,4-TCDD to 1 mL of each concentration calibration solution. The solutions obtained will be clearly labeled to identify which formulation is supplied.

7.6.1 Alternative One

Four isooctane solutions containing unlabeled 2,3,7,8-TCDD at varying concentrations and $^{13}C_{12}$-2,3,7,8-TCDD (the internal standard, CASRN 80494-19-5) and $^{13}C_{12}$-1,2,3,4-TCDD (the recovery standard) at a constant concentration. Two of these solutions also contain $^{37}Cl_4$-2,3,7,8-TCDD (the surrogate compound, CASRN 85508-50-5) at a constant concentration. Concentration calibration solutions are to be obtained from the Quality Assurance Division, US EPA Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. However, if not available from EMSL-LV, standards may be obtained from commercial sources, and solutions may be prepared in the contractor laboratory. Traceability of standards must be

verified against EPA-supplied standard solutions, by laboratory SOP's as required in IFB Pre-Award Bid Confirmations, part 2.f.(4).

7.6.1.1 Each of solutions #1-#4 contains $^{13}C_{12}$-2,3,7,8-TCDD at a concentration of 1 ng/uL which is equivalent to a 50-uL extract of a 10-g sample to which that compound (the internal standard) was added at a concentration of 5 ug/kg or a 50 uL extract of a 1 L water sample to which the internal standard was added at a concentration of 50 ng/L.

7.6.1.2 Solutions #1-#4 contain unlabeled 2,3,7,8-TCDD at concentrations of 0.2, 1, 5, and 20 ng/uL respectively; those concentrations are equivalent to 50-uL extracts of 10-g samples containing 1, 5, 25, and 100 ppb, respectively, or of 1 L water samples containing 0.01, 0.05, 0.25, and 1.0 ppb, respectively.

7.6.1.3 Solutions #1-#4 contain $^{13}C_{12}$-1,2,3,4-TCDD at a concentration of 0.6 ng/uL.

7.6.1.4 Solutions #1-#2 contain $^{37}Cl_4$-2,3,7,8-TCDD at a concentration of 0.028 ng/uL, this concentration is equivalent to an extract of a sample containing 0.14 ug/kg or 1.4 ng/L the amount of $^{37}Cl_4$-TCDD (the surrogate compound) added to each sample before extraction.

7.6.2 Alternative Two

Four isooctane solutions containing unlabeled 2,3,7,8-TCDD at varying concentrations and $^{13}C_{12}$-2,3,7,8-TCDD (the internal standard (CASRN 80494-19-5) at a constant concentration. Three of these solutions also contain $^{37}Cl_4$2,3,7,8-TCDD (the surrogate compound, CASRN 85508-50-5) at varying concentrations. Concentration calibration solutions are to be obtained from the Quality Assurance Division, US EPA Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. However, if not available from EMSL-LV, standards may be obtained from commercial sources, and solutions may be prepared in the contractor laboratory. Traceability of standards must be verified against EPA-supplied standard solutions, by laboratory SOP's as required in IFB Pre-Award Bid Confirmations, part 2.f.(4).

7.6.2.1 A solution of $^{13}C_{12}$-1,2,3,4-TCDD at a concentration of 10 ng/uL in isooctane is provided to be added to each of the concentration calibration

(CC) solutions. The amount to be added to each CC solution is given below. This will result in all compounds being in the same ratios as Alternative One but the concentrations are 9 percent lower.

10 ng/uL $^{13}C_{12}$-1,2,3,4,-TCDD Solution

CC1 - 1 mL CC1 + 60 uL $^{13}C_{12}$-1,2,3,4-TCDD + 40 uL Isooctane
CC2 - 1 mL CC2 + 60 uL $^{13}C_{12}$-1,2,3,4-TCDD + 40 uL Isooctane
CC3 - 1 mL CC3 + 60 uL $^{13}C_{12}$-1,2,3,4-TCDD + 40 uL Isooctane
CC4 - 1 mL CC4 + 60 uL $^{13}C_{12}$-1,2,3,4-TCDD + 40 uL Isooctane

7.6.2.2 The final CC#1-CC#4 solutions contain $^{13}C_{12}$-2,3,7,8-TCDD at 0.909 ng/uL which is equivalent to a 50-uL extract of a 10 g or 1 L sample to which that compound (the internal standard) was added at a concentration of 4.5 ug/Kg or 45 ng/L.

7.6.2.3 The final CC#1-CC#4 solutions contain unlabeled 2,3,7,8-TCDD at concentrations of 0.182, 0.909, 4.545, and 18.18 ng/uL, respectively.

7.6.2.4 Solutions #1-#4 contain $^{13}C_{12}$-1,2,3,4-TCDD at a concentration of 0.54 ng/uL.

7.6.2.5 Solutions #1-#3 contain $^{37}Cl_4$-2,3,7,8-TCDD at concentrations of 0.054, 0.109 and 0.182 ng/uL, respectively.

NOTE: The surrogate concentrations do not correspond to Alternative One and are not at the level used for the confirmation of the 1.0 ug/kg or 10 ng/L detection criteria.

7.6.3 Store concentration calibration solutions in 1-mL amber mini-vials at room temperature.

7.7 Performance Check Solution -- A mixture containing at a minimum: unlabeled 2,3,7,8-TCDD (CASRN 1746-01-6); 1,2,3,4-TCDD (CASRN 30746-58-8); 1,4,7,8-TCDD (CASRN 40581-94-0); 1,2,3,7-TCDD (CASRN 67028-18-6); 1,2,3,8-TCDD (CASRN 53555-02-5); 1,2,7,8-TCDD (CASRN 34816-53-0) and 1,2,6,7-TCDD (CASRN 40581-90-6) must be obtained from the Quality Assurance Division, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. Note: This solution may vary between lots.

To this dry mixture add 500 uL of the sample fortification solution (Section 7.8) containing $^{13}C_{12}$-2,3,7,8-TCDD at a concentration of 0.5 ng/uL and $^{37}Cl_4$-2,3,7,8-TCDD at a concentration of 0.014 ng/uL and 50

uL of the 10 ng/uL $^{13}C_{12}$-1,2,3,4-TCDD recovery standard solution. Store in 1-mL amber mini-vial at 4°C.

7.8 Sample Fortification Solution -- An isooctane solution containing the internal standard at a concentration of 0.5 ng/uL and the surrogate compound at a concentration of 0.014 ng/uL. Mix 100 uL with 1.5 mL of acetone before adding to each sample and blank.

7.9 Field Blank Fortification Solution -- An isooctane solution containing the internal standard at a concentration of 0.5 ng/uL, the surrogate compound at a concentration of 0.014 ng/uL, and the unlabeled 2,3,7,8-TCDD at a concentration of 0.1 ng/uL. Mix 100 uL with 1.5 mL of acetone before adding to each field blank.

7.10 Recovery Standard Solution -- An isooctane solution containing the recovery standard $^{13}C_{12}$-1,2,3,4-TCDD at a concentration of 10 ng/uL.

7.10.1 For samples to be analyzed using Alternative One CC Solutions, the recovery standard $^{13}C_{12}$-1,2,3,4-TCDD is used at a concentration of 10 ng/uL.

7.10.2 For samples to be analyzed using Alternative Two CC solutions as standards, the recovery standard $^{13}C_{12}$-1,2,3,4-TCDD must be diluted before use; this is done by adding 100 uL of isooctane to a measured 1.0 mL of the 10 ng/uL $^{13}C_{12}$-1,2,3,4-TCDD solution.

8. SAMPLE PRESERVATION AND HANDLING

8.1 Chain-of-custody Procedures (see Exhibit G)

8.2 Sample Preservation

8.2.1 Soil Samples: When received, each sample will be contained in a 1-pint glass jar surrounded by vermiculite in a sealed metal paint can. Until a portion is to be removed for analysis, store the sealed paint cans in a locked limited-access area where ambient temperature is maintained above freezing. After a portion is removed for analysis, return the unused portion of sample to its original containers and store as stated above. Do not freeze samples; they may contain sufficient water to break the sample jar if frozen.

8.2.2 Water Samples: Each water sample received will consist of two (2) 1 liter (or quart) amber glass bottles. Samples may be iced or refrigerated at 4°C from the time of collection until extraction. Do not freeze. Samples must be extracted within 10 days of VTSR.

8.2.3 All samples must be protected from light from the time of collection until extraction to prevent photodecomposition.

8.3 Sample Handling

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

8.3.2 Pre-extraction Sample Treatment

8.3.2.1 For the purpose of this contract a water sample is defined as a single phase system, the primary component of which is water. This may include floating, suspended, and settled particulate matter in quantities that do not cause severe problems with the extraction. If sufficient particulate matter is present to be considered a separate phase or it causes severe extraction problems procede to Section 8.3.2.4.

8.3.2.2 For the purpose of this contract a soil/sediment sample is defined as a single phase solid system composed of soil or sediment. It may contain particulates such as stones, vegetation, etc. but should not contain an obvious liquid phase. If a liquid phase is present, procede to Section 8.3.2.4.

8.3.2.3 Homogenization -- Although sampling personnel will attempt to collect homogeneous samples, the contractor shall examine each sample and judge if it needs further mixing. NOTE: Contractor personnel have the responsibility to take a representative sample aliquot this responsibility entails efforts to make the sample as homogeneous as possible. Stirring is recommended when possible.

8.3.2.4 Centrifugation -- If a soil or water sample contains more than one phase, contact your DPO to determine which phase(s) should be analyzed. If the sample contains obvious aqueous/solid phases, centrifuge it to separate liquid and solid phases (an organic phase is beyond the scope of this method, contact your DPO for instructions). Place the entire sample in suitable centrifuge bottle(s) and centrifuge for 30 minutes at 400 x G. Remove bottle(s) from centrifuge and decant the aqueous phase to be analyzed as a water sample. Mix solid layer with stainless steel spatula and remove a portion to be weighed and analyzed as a soil/sediment sample.

Return the remaining solid portion to original sample bottle and store.

CAUTION: A phase not analyzed may contain TCDD and should be handled and disposed of appropriately.

9. CALIBRATION

9.1 Two types of calibration procedures are required. One type, initial calibration, is required before any samples are analyzed for TCDD, and is required intermittently throughout sample analyses as dictated by results of routine calibration procedures described below. The other type, routine calibration, consists of analyzing the column performance check solution and concentration calibration solution #1 (Section 7.6). No samples are to be analyzed until acceptable calibration as described in Section 9.2 and 9.3 is demonstrated and documented.

9.2 Initial Calibration

9.2.1 Concentration calibration solutions -- the four solutions described in Section 7.6 are required.

9.2.2 Inject an appropriate aliquot of the performance check solution (CAUTION: See Section 6.1.1) and acquire selected-ionmonitoring (SIM) mass spectral data using the MS operating conditions specified in Section 9.2.4. Determine GC operating conditions necessary to achieve separation described in Section 9.2.6.1.

9.2.3 Determine valley percent as described in Section 9.2.6.1 and the m/z ratio according to the criteria in Section 9.2.6.2. If the valley percent and/or m/z ratios are outside requirements, corrective action to meet the criteria must be taken (as described in Section 6.1.1) before further sample analyses are performed.

9.2.4 Using the same GC conditions that produced acceptable results with the performance check solution, analyze a 2-uL or other appropriate aliquot (as described in Section 6.1.1) of each of the four concentration calibration solutions with the following MS operating parameters.

9.2.4.1 Acquire selected-ion-monitoring data for m/z 259, 320, 322, 328, 332 and 334.

9.2.4.2 Total cycle time for data acquisition must be $\leq$1.5 seconds.

9.2.4.3 Acquire at least five data points for each ion during elution of the GC peak.

9.2.4.4 Use the same data acquisition time for each of the six ions being monitored.

9.2.5 Repeat Section 9.2.4 two times to produce triplicate data sets for each solution. NOTE: CC solutions should be analyzed in either random order or in order of increasing concentration to avoid biasing the calibration.

9.2.6 The Laboratory must not proceed with analysis before determining and documenting acceptable calibration with the following criteria:

9.2.6.1 GC Column Performance

9.2.6.1.1 The valley between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers must be resolved with a valley $\leq$25%. Valley (%) = x/y X 100, when y is peak height of 2,3,7,8TCDD, x is measured as shown in Figures 2 and 3 at the end of this Exhibit. The peak representing 2,3,7,8-TCDD shall be labeled and identified as such on the chromatograms.

9.2.6.1.2 Ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be $\geq$0.67 and $\leq$0.90.

9.2.6.1.3 Ratio of integrated ion current for m/z 332 to m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD must be $\geq$0.67 and $\leq$0.90.

9.2.6.2 Calibration solutions must meet the following criteria:

9.2.6.2.1 MS sensitivity -- signal-to-noise (S/N) ratio (Section 3.10) of >2.5 for m/z 259, 320,and 322 for unlabeled 2,3,7,8-TCDD and 328 for $^{37}Cl_4$-2,3,7,8-TCDD and >10 for m/z 332 and 334 for $^{13}C_{12}$-2,3,7,8-TCDD.

9.2.6.2.2 The ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be $\geq$0.67 and $\leq$0.90.

9.2.6.2.3 The ratio of integrated ion current for m/z 332 to m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD must be $\geq$0.67 and $\leq$0.90.

9.2.6.3 Calculate the response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$2,3,7,8-TCDD:

$$RRF_n = \frac{A_x \cdot Q_{is}}{A_{is} \cdot Q_x}$$

Where A_x = the sum of integrated ion abundances of m/z 320 and m/z 322 for unlabeled 2,3,7,8-TCDD,

A_{is} = the sum of integrated abundances of m/z 332 and m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD,

Q_{is} = quantity of $^{13}C_{12}$-2,3,7,8-TCDD, and

Q_x = quantity of unlabeled 2,3,7,8-TCDD injected.

RRF is a dimensionless number; units used to express quantities must be consistent.

9.2.6.4 Calculate the response factor for $^{13}C_{12}$-2,3,7,8-TCDD relative to $^{13}C_{12}$-1,2,3,4-TCDD:

$$RRF_i = \frac{A_{is} \cdot Q_{rs}}{A_{rs} \cdot Q_{is}}$$

Where A_{rs} = The sum of the integrated ion abundance of m/z 332 and m/z 334 for $^{13}C_{12}$-1,2,3,4-TCDD

Q_{rs} = Quantity of $^{13}C_{12}$-1,2,3,4-TCDD Injected

A_{is} and Q_{is} are as in Section 9.2.6.3.

9.2.6.5 Response Factor Criteria:

9.2.6.5.1 Calculate the mean RRF and its percent relative standard deviation (%RSD) from triplicate analysis of each of 4 concentration solutions for unlabeled 2,3,7,8-TCDD and $^{13}C_{12}$-TCDD.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean RRF}} \times 100$$

9.2.6.5.2 The variation of the RRF calculated for unlabeled 2,3,7,8-TCDD at each concentration level must not exceed 10% RSD.

9.2.6.5.3 Calculate the mean and %RSD of the 4 mean RRFs for unlabeled 2,3,7,8-TCDD and for $^{13}C_{12}$-TCDD.

9.2.6.5.4 The %RSD of the 4 mean RRFs for $^{13}C_{12}$-2,3,7,8TCDD should not exceed 10% RSD.

9.2.6.5.5 The %RSD of the 4 mean RRFs for unlabeled TCDD must not exceed 10% RSD.

9.2.6.5.6 The mean of the mean RRFs for each compound must be used for concentration calculations.

9.2.6.5.7 The concentration curves must be plotted (RRF vs concentration) for enclosure in the deliverables package.

9.3 Routine Calibration

9.3.1 Inject an appropriate aliquot (CAUTION: See Section 6.1.1) of the performance check solution (Section 7.7) and acquire selected ion monitoring mass spectral data for m/z 259, 320, 322, 328, 332, and 334 within a total cycle time of <1.5 seconds. Acquire at least five data points for each GC peak and use the same data acquisition time for each of the six ions being monitored. NOTE: The same data acquisition parameters previously used to analyze concentration calibration solutions during initial calibration must be used for the performance check solution. The column performance check solution must be run at the beginning and end of each 12-hour period, if the contractor laboratory operates during consecutive 12-hour periods (shifts), analysis of the performance check solution at the beginning of each 12-hour period and at the end of the final 12-hour period is sufficient.

9.3.2 Determine and document acceptable column performance as described in Section 9.2.6.1.

9.3.3 Inject 2 uL of concentration calibration solution #1 which contains 0.2 ng/uL of unlabeled 2,3,7,8-TCDD once at the begininning of each 12-hour period. Using the same GC/MS/DS conditions as used in Section 9.3.1, acquire data for m/z 259, 320, 322, 328, 332 and 334. Determine and document acceptable calibration as described below.

9.3.3.1 MS sensitivity -- signal-to-noise (S/N) ratio (Section 3.8) of >2.5 for m/z 259, 320, 322, and 328 for unlabeled 2,3,7,8-TCDD and $^{37}Cl_4$-2,3,7,8-TCDD and >10 for m/z 332 and 334 for $^{13}C_{12}$-2,3,7,8-TCDD is required. The ratio of integrated ion current for m/z 320/322 must be $\geq$0.67 and $\leq$0.90.

9.3.3.2 Measured response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD must be within ±10% of the mean value established (Section 9.2.6.5.3) by initial analyses of the concentration calibration solutions.

9.3.4 Further sample analyses must not be performed if the criteria in Section 9.3.2 and 9.3.3 are not met. Possible remedies are listed in Section 9.3.5. Following corrective action, a routine calibration must be performed and criteria listed in Section 9.3.3 must be met before further analysis of samples is performed; if the routine calibration does not meet criteria a new initial calibration must be performed.

9.3.5 Remedial actions shall be taken by Contractor if criteria are not met. Possible remedies are:

9.3.5.1 Check and adjust GC and/or MS operating conditions.

9.3.5.2 Replace GC column (performance of initial calibration procedures are required if acceptance criteria for continuing calibration are not met).

9.3.5.3 Tune MS for greater or lesser resolution.

9.3.5.4 Calibrate MS mass scale.

9.3.5.5 Prepare and analyze new performance check solution.

10. QUALITY CONTROL

See Exhibit E for QA/QC Requirements.

11. PROCEDURES

11.1 Soil Sample Extraction

11.1.1 CAUTION: See Section 5 for safety guidelines and recommendations.

11.1.2 Jar extraction. NOTE: Extremely wet samples may require centrifuging to remove water before addition of sodium sulfate (see Section 8.3.2.2).

11.1.2.1 Accurately weigh to three significant figures a 10 g (±0.5 g) portion of the wet soil or sediment sample, and transfer it to the extraction jar.

11.1.2.2 Note: Additional QC samples are required as specified in exhibit E. These are processed as described in this section with the following exceptions:

a. Laboratory Method Blank - Perform all steps in the analytical procedure but substitute an aliquot of sodium sulfate for the soil/sediment sample.

b. Fortified Field Blank - Perform all steps in the analytical procedure, but spike the designated sample with 1.5 mL of the acetone dilution of the field blank fortification solution (Section 7.9) rather than the sample fortification solution.

11.1.2.3 Add 1.5 mL of the acetone dilution of the sample fortification solution (Section 7.8) to the soil or sediment in the extraction jar. Add small portions of the solution at several sites on the surface of the soil or sediment.

11.1.2.4 Add 20 g of purified anhydrous sodium sulfate, and mix thoroughly using a stainless steel spoon or spatula.

11.1.2.5 Allow the mixture of soil and sodium sulfate to set for 2 hours at ambient temperature; mix again, break all visible lumps, and allow to set for at least 4 more hours.

11.1.2.6 Mix again and add 20 mL of methanol; mix again and add 150 mL of hexane.

11.1.2.7 Place the extraction jar containing the soil, sodium sulfate and solvents in the shaker and shake for at least 3 hours.

11.1.2.8 Remove the jar from the shaker and allow all solids to settle. Decant the solvent through a glass funnel containing hexane-rinsed filter paper into a clean Kuderna-Danish apparatus. Rinse the jar, solid sample residue, and filter residue with four 5-mL portions of hexane.

11.1.2.9 Concentrate the extract volume to approximately 2 to 3 mL with a Kuderna-Danish apparatus. NOTE: Glassware used for more than one sample must be carefully cleaned between samples to prevent cross contamination (see Section 6.5).

11.1.2.10 Rinse the evaporator flask with 3 mL portions of hexane; transfer each rinse to the concentrator tube. Between additions of hexane rinse, reduce the extract volume in the concentrator tube enough to allow addition of another 5 mL volume of rinse. To reduce the volume, place the concentrator tube in a water bath adjusted to operate at 50°C and position the tube so that the surfaces of the extract and the water are at about the same level. Evaporate the solvent with a stream of nitrogen with the tip of the nitrogen delivery tube 2 cm above the solution.

11.1.2.11 After the final rinse has been added, reduce the extract volume to approximately 1 mL. Proceed to section 11.3.2. If further processing will be delayed, quantitatively transfer the extract to a Teflon sealed screw-cap vial and store refrigerated and protected from light.

11.2 Water Sample Extraction

Caution: When using this method to analyze for 2,3,7,8-TCDD, all of the following operations should be performed in a limited access laboratory with the analyst wearing full protective covering for all exposed skin surfaces. See Section 5.

11.2.1 Mark the water meniscus on the side of the sample bottle for later determination of the sample volume. Pour the entire sample into a 2 L separatory funnel. Note: a continuous liquid-liquid extractor may also be used.

11.2.1.1. NOTE: Additional QC samples are required as specified in exhibit E. These are processed as described in this section with the following exceptions:

a. Laboratory Method Blank - Perform all steps in the analytical procedure but substitute an aliquot of reagent water for the sample.

b. Fortified Field Blank - Perform all steps in the analytical procedure, but spike the designated sample with 1.5 mL of the acetone dilution of the field blank fortification solution (Section 7.9) rather than the sample fortification solution.

11.2.2 Add 1.5 mL of the acetone dilution of the sample fortification solution containing 50 ng of the internal standard and 1.4 ng of the surrogate compound to the sample in the separatory funnel.

11.2.3 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250 mL Erlenmeyer flask.

11.2.4 Add a second 60 mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.2.5 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Pour the combined extracts into the K-D concentrator through a drying column containing about 6 cm of sodium sulfate. Rinse the Erlenmeyer flask with 25-30 mL methylene chloride and pour it through the drying column, rinse the drying column with an additional 10 ml methylene chloride. All rinses are added to the concentrator.

11.2.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.2.7 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the

temperature of the water bath to 85 to 90°C. Concentrate the extract as in section 11.2.6 except use hexane to prewet the column. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane.

11.2.8 Add a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 1 mL of hexane to the top. Place the micro-K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches about 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL hexane. Proceed to section 11.3.2. If further processing is to be delayed, the extract should be quantitatively transfered to a Teflon sealed screw-cap vial and store refrigerated and protected from light.

11.2.9 Fill the sample bottle with water to the mark and measure the volume to the nearest 10 mL in a 1 L graduated cylinder.

11.3 Column Chromatograph

11.3.1 Column Preparation

11.3.1.1 Column 1: Place 1.0 g of silica gel into a 1 cm x 20 cm column and tap the column gently to settle the silica gel. Add 2 g sodium hydroxide-impregnated silica gel, 1 g silica gel, 4.0 g of sulfuric acid-impregnated silica gel, and 2 g silica gel. Tap column gently after each addition.

11.3.1.2 Column 2: Place 6.0 g of alumina into a 1 cm x 30 cm column and tap the column gently to settle the alumina. Add a 1-cm layer of purified sodium sulfate to the top of the alumina.

11.3.1.3 Add hexane to each column until the packing is free of channels and air bubbles. A small positive pressure (5 psi) of clean nitrogen can be used if needed.

11.3.2 Quantitatively transfer the hexane sample extract from the concentrator tube to the top of the silica gel in Column 1. Rinse the concentrator tube with two 0.5 mL portions of hexane; transfer rinses to Column 1.

11.3.3 With 90 mL of hexane, elute the extract from Column 1 directly into Column 2 containing alumina and sodium sulfate.

11.3.4 Add 20 mL of hexane to Column 2 and elute until the hexane level is just below the top of the sodium sulfate; discard the eluted hexane.

11.3.5 Add 20 mL of 20% methylene chloride/80% hexane (volume/volume) to Column 2 and collect the eluate.

11.3.6 Reduce the volume of the eluate with a gentle stream of filtered dry nitrogen. When the volume of the eluate is about 1 to 2 mL, transfer the eluate to the Carbopack column (Section 11.4.4). Rinse the eluate container with two 0.5 mL portions of hexane; transfer the rinses to the Carbopack column. CAUTION: Do not evaporate the sample extract to dryness. NOTE: The carbopack cleanup is not required for water samples unless needed to meet detection sensitivity criteria.

11.4 Carbopack Column Chromatography Procedure

11.4.1 Thoroughly mix 3.6 g of Carbopack C (or equivalent) with 16.4 g of Celite 545 (or equivalent) in a 40 mL vial and activate by heating in an oven at 130°C for 6 hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopack/Celite to ensure TCDD recovery of >50%. Subject the low level concentration calibration solution to this procedure and measure the quantity of labeled and unlabeled 2,3,7,8-TCDD.

11.4.2 Insert a small plug of glass wool into a disposable pipet approximately 15 cm long by 7 mm O.D. Apply suction with a vacuum aspirator attached to the pointed end of the pipet, and add the Carbopack/Celite mixture until a 2 cm packing is obtained.

11.4.3 Pre-elute the column with:

11.4.3.1 2 mL toluene

11.4.3.2 1 mL of mixture of 75% (by volume) methylene chloride, 20% methanol and 5% benzene

11.4.3.3 1 mL of 50% (by volume) cyclohexane and 50% methylene chloride

11.4.3.4 2 mL of hexane

11.4.4 While the column is still wet with hexane add the sample extract from section 11.2.6. Elute the column with the following sequence of solvents and discard the eluates.

11.4.4.1 2 mL hexane

11.4.4.2 1 mL of 50% (by volume) cyclohexane and 50% methylene chloride

11.4.4.3 1 mL of 75% (by volume) methylene chloride, 20% methanol and 5% benzene

11.4.5 Elute with 2 mL of toluene and collect the elutate, which contains the TCDD. Transfer the rinses to a 1-mL amber minivial with conical reservoir with further concentration as necessary. CAUTION: Do not evaporate the sample extract to dryness.

11.3.6 Store the sample extract in the dark at 4°C until just before GC/MS analysis.

11.5 GC/MS Analysis

11.5.1 Remove the sample extract or blank from storage and allow it to warm to ambient laboratory temperature. With a stream of dry, filtered nitrogen, reduce the extract/blank volume to near dryness. Immediately before GC/MS analysis, add 5 uL of the 10 ng/uL recovery standard solution and adjust the extract or blank volume to 50 uL with isooctane.

11.5.2 Inject a 2-uL aliquot of the extract into the GC, operated under conditions previously used (Section 9) to produce acceptable results with the performance check solution.

11.5.3 Acquire mass spectral data for the following selected characteristic ions: m/z 259, 320, and 322 for unlabeled 2,3,7,8-TCDD; m/z 328 for $^{37}Cl_4$-2,3,7,8-TCDD; and m/z 332 and 334 for $^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-1,2,3,4-TCDD. Use the same data acquisition time and MS operating conditions previously used (Section 9.2.6) to determine response factors.

11.6 Identification Criteria. NOTE: Refer to Exhibit E, Section 7, for application of identification criteria.

11.6.1 Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the $^{13}C_{12}$-2,3,7,8-TCDD. Retention times are required for all chromatograms, but scan numbers are optional. These parameters should be printed next to the appropriate peak.

11.6.2 The integrated ion currents detected for m/z 259, 320, and 322 must maximize simultaneously. If there are peaks that will affect the maximization or quantitation of peaks of interest, attempts should be made to narrow the scan window to eliminate the interfering peaks. This should be reported on a separate chromatogram.

11.6.3 The integrated ion current for each analyte ion (m/z 259, 320 and 322) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background noise and must not have saturated the detector.

11.6.4 Abundance of integrated ion counts detected for m/z 320 must be $\geq$67% and $\leq$90% of integrated ion counts detected for m/z 322.

11.6.5 Abundance of integrated ion counts detected for m/z 332 must be $\geq$67% and $\leq$90% of integrated ion counts detected for m/z 334.

11.6.6 The recovery of the internal standard $^{13}C_{12}$-2,3,7,8-TCDD should be within a 40 percent to 120 percent recovery window. This is an advisory limit only, an action window may be set when sufficient data is available.

12. CALCULATIONS

12.1 Concentration

12.1.1 Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_x = \frac{A_x \cdot Q_{is}}{A_{is} \cdot RF_n \cdot W}$$

where C_x = 2,3,7,8-TCDD concentration in ug/kg or ug/L

A_x = the sum of integrated ion abundance detected for m/z 320 and 322

A_{is} = the sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of $^{13}C_{12}$-2,3,7,8-TCDD, the internal standard)

Q_{is} = quantity (in ng) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction

RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD

W = weight (in g) of wet soil or sediment sample or volume of water extracted (in mL).

12.1.2 If the calculated concentration of unlabeled 2,3,7,8-TCDD exceeds 100 ug/kg for soil/sediment or 1 ug/L for water, which is the maximum concentration of the concentration calibration solutions, the linear range may have been exceeded, and a smaller aliquot of that sample must be analyzed. Accurately weigh to three significant figures a 1-g aliquot of the wet soil/sediment or measure a 100 mL aliquot of water. Add the 1.5 mL acetone dilution of 100 uL of the sample fortification solution (Section 7.8), just as for the larger sample aliquot. Extract and analyze.

12.1.3 Calculate the concentration of the internal standard $^{13}C_{12}$-2,3,7,8-TCDD using the formula:

$$C_{is} = \frac{A_{is} \cdot Q_{rs}}{A_{rs} \cdot RF_i \cdot W}$$

where

C_{is} = concentration of $^{13}C_{12}$-2,3,7,8-TCDD in ug/kg or ug/L

A_{is} = sum of integrated ion abundances for m/z 332 and 334 for $^{13}C_{12}$-2,3,7,8-TCDD

A_{rs} = sum of integrated ion abundances for m/z 332 and 334 for $^{13}C_{12}$-1,2,3,4-TCDD

Q_{rs} = quantity (in ng) of $^{13}C_{12}$-1,2,3,4-TCDD added to the sample before injection

RF_i = calculated mean response factor for $^{13}C_{12}$-1,2,3,4-TCDD

W = weight (in g) of wet soil or sediment sample or volume of water extracted (in mL).

12.2 Estimated Maximum Possible Concentration -- For samples in which no unlabeled 2,3,7,8-TCDD was detected, calculate the estimated maximum possible concentration, which is the concentration required to produce a signal with peak height of 2.5 times the background signal level. The background level is determined by measuring the range of the noise (minimum to maximum) for either m/z 320 or 322 in the appropriate region of the SICP (as defined in section 3.11), multiplying that noise height by 2.5, and relating the product height to an estimated concentration that would produce that product height.

Use the formula:

$$MPC = \frac{2.5 \cdot H_x \cdot Q_{is}}{H_{is} \cdot RF_n \cdot W}$$

where MPC = estimated maximum possible concentration of unlabeled 2,3,7,8-TCDD required to produce H_x in ug/kg or ug/L

H_x = peak height for either m/z 320 or 322 within $\pm$ 5 scans of the internal standard peak used to measure H_{is}

H_{is} = peak height of the appropriate ion characteristic of the internal standard m/z 332 when m/z 320 is used to determine H_x, and m/z 334 when m/z 322 is used to determine H_x

Q_{is}, RF and W retain the definitions previously stated in Section 12.1.1

12.4 The relative percent difference (RPD) is calculated as follows: (See Section 5.1.1, Exhibit E.)

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

S_1 and S_2 represent sample and duplicate sample results.

12.6 Percent Recovery of 2,3,7,8-TCDD in spiked field blanks =

$$\frac{\text{concentration found}}{\text{concentration added}} \times 100$$

12.7 Percent Recovery of internal standard, $^{13}C_{12}$-2,3,7,8-TCDD =

$$\frac{\text{concentration found}}{\text{concentration added}} \times 100$$

12.8 Standard deviation = $S = \sqrt{\sum_{i=1}^{N} \frac{(X_i - \bar{X})^2}{N - 1}}$

12.9 Percent relative standard deviation =

$$\frac{\text{Standard Deviation}}{\text{Mean}} \times 100 = \frac{S}{\bar{X}} \times 100$$

TABLE 1. OPERATING CONDITION GUIDELINES

Column coating	SP-2330	CP-SIL 88
Film thickness	0.2 um	0.22 um
Column dimensions	60 m x 0.24 mm	50 m x 0.22 mm
Helium* linear velocity	28-29 cm/sec at 240°C	28-29 cm/sec at 240°C
Initial temperature	70°C	45°C
Initial time	4 min	3 min
Temperature program	Rapid increase to 200°C 200°C to 240°C at 4°C/min	Rapid increase to 190°C 190°C to 240°C at 5°C/min
2,3,7,8-TCDD retention time	24 min	26 min

*Hydrogen is an acceptable carrier gas.

TABLE 2. COMPOSITION OF CONCENTRATION CALIBRATION SOLUTIONS

Alternative One

	(TCDD)			
Solution #	Unlabeled 2,3,7,8	$^{37}Cl_4$-2,3,7,8	$^{13}C_{12}$-2,3,7,8	$^{13}C_{12}$-1,2,3,4
CC1	0.2 ng/uL	0.028 ng/uL	1.0 ng/uL	0.6 ng/uL
CC2	1.0	0.028	1.0	0.6
CC3	5.0	----	1.0	0.6
CC4	20.0	----	1.0	0.6

Alternative Two

These are the final concentrations obtained. All compounds except $^{37}Cl_4$-2,3,7,8-TCDD, are in the same ratios as Alternative One but are 9% lower.

	(TCDD)			
Solution #	Unlabeled 2,3,7,8	$^{37}Cl_4$2,3,7,8	$^{13}C_{12}$-2,3,7,8	$^{13}C_{12}$-1,2,3,4
CC1	0.182 ng/uL	0.054 ng/uL	0.909 ng/uL	0.545 ng/uL
CC2	0.909	0.109	0.909	0.545
CC3	4.545	0.182	0.909	0.545
CC4	18.18	-----	0.909	0.545

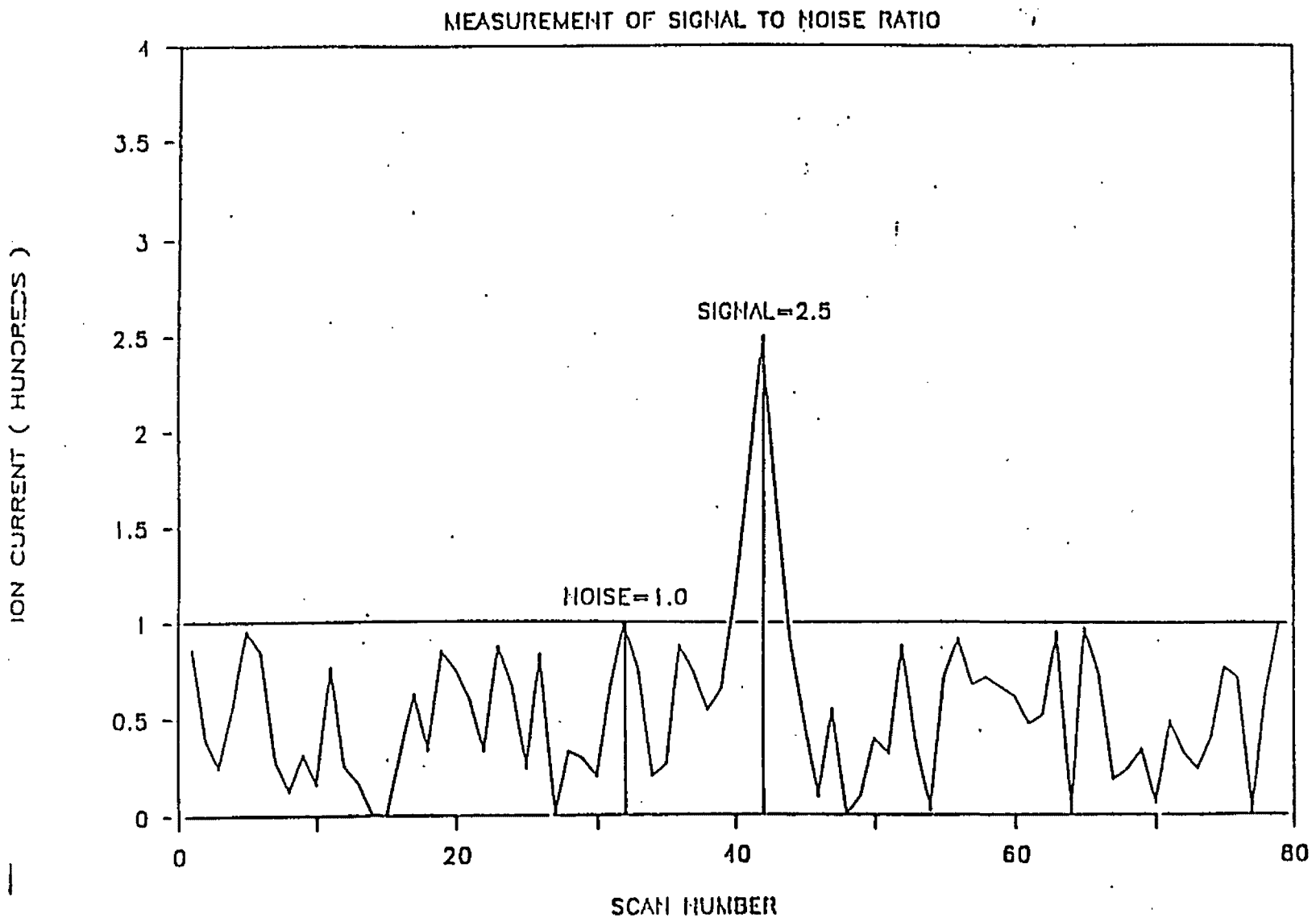

Figure 1. Measurement of signed to noise ratio.

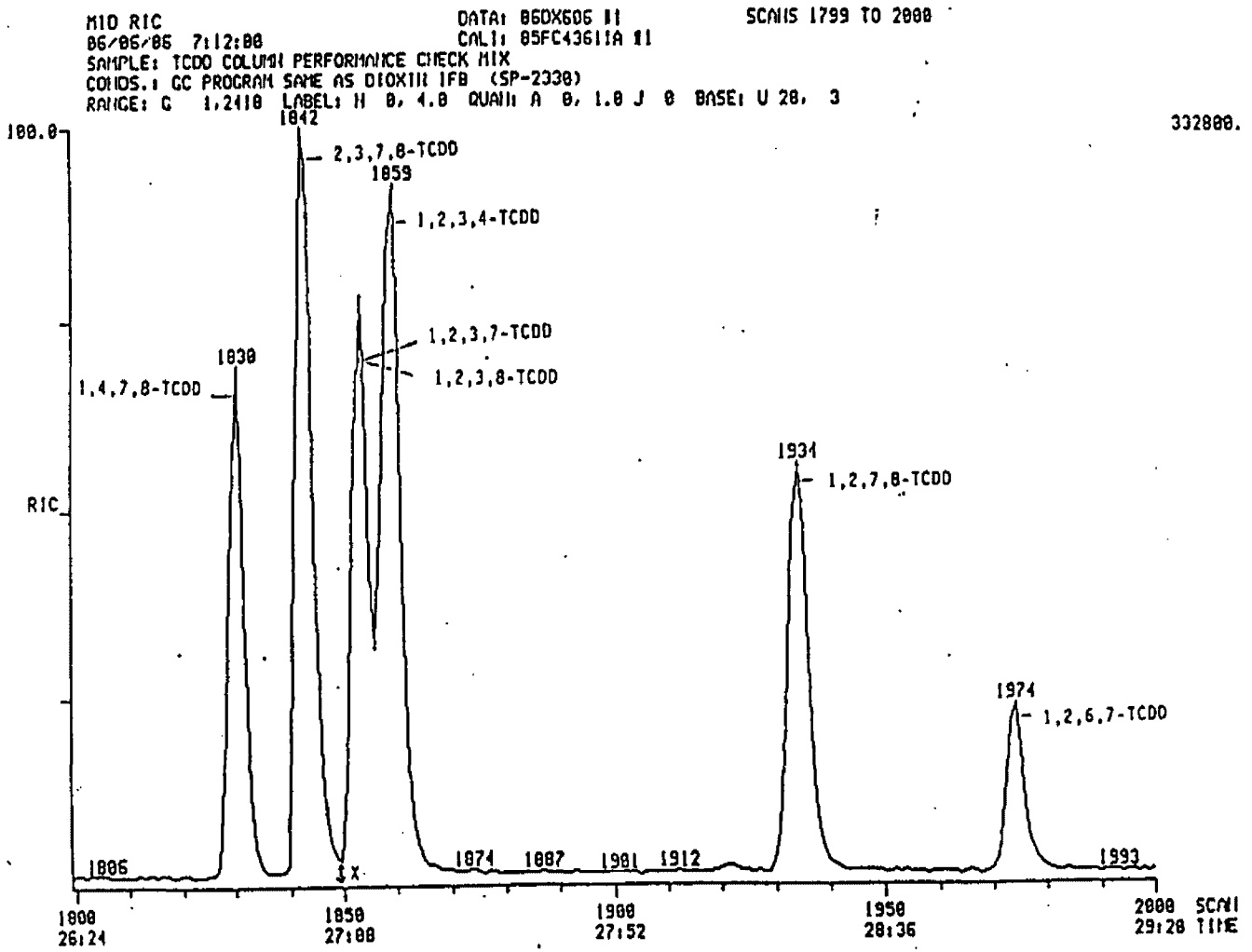

Figure 2. Selected ion current profile for m/z 320 and 322 produced by MS analysis of performance check solution using a 60-m SP-2330 fused silica capillary column and conditions listed in Table 1.

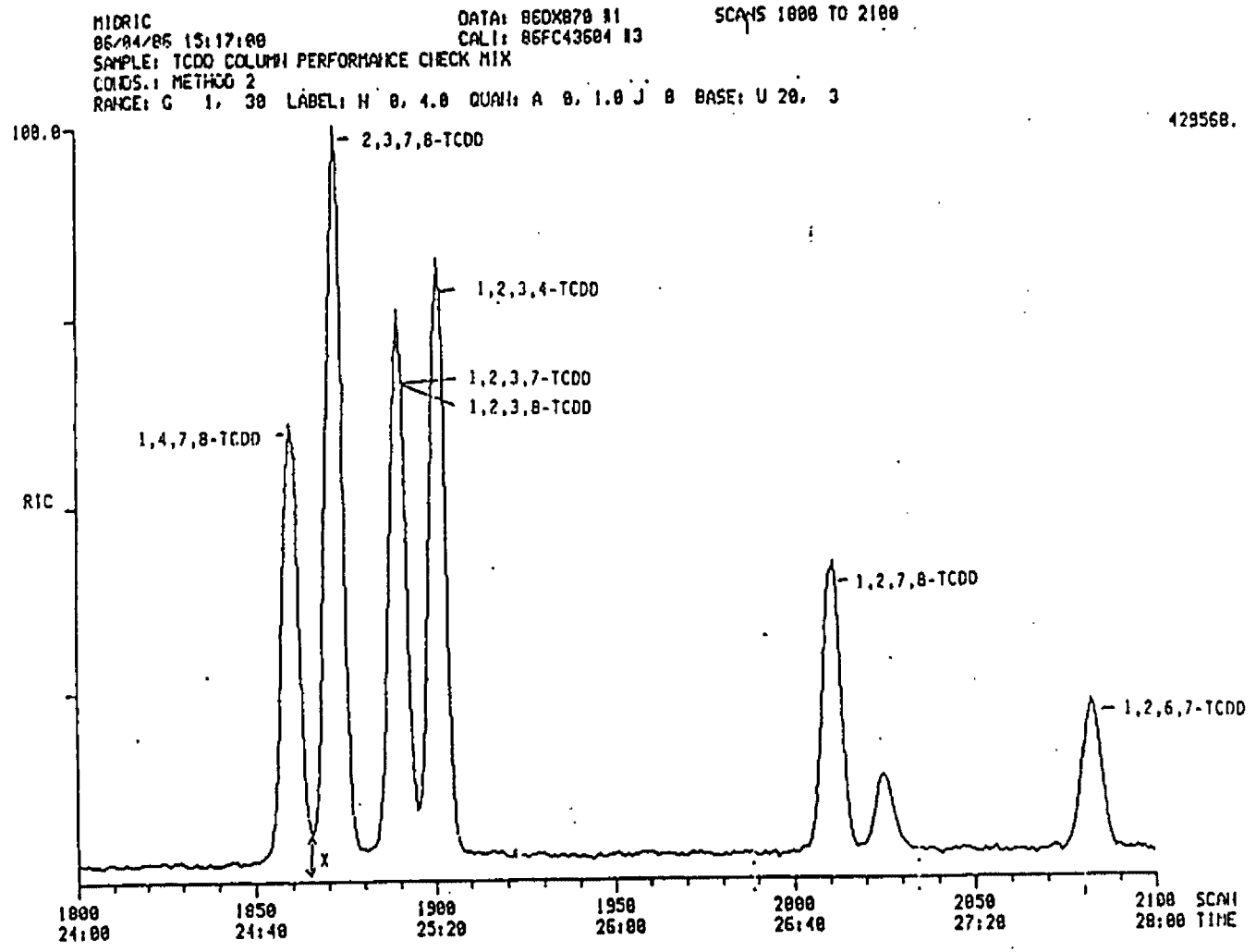

Figure 3. Selected ion current profile for m/z 320 and 322 produced by MS analysis of performance check solution using a 50-m CP-SIL 88 fused silica capillary column and conditions listed in Table 1.

REFERENCES

1. "Carcinogens-Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977.

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3. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

4. Method 613, "2,3,7,8-Tetrachlorodibenzo-p-dioxin," Federal Register, 44 (233) 69529, December 3, 1979.

5. "Quality Assurance Plan for 2,3,7,8-TCDD Monitoring Project," R. D. Kleopfer and C. J. Kirchmer, presented by the Division of Environmental Chemistry, American Chemical Society, Washington, D.C., September 1983.

6. "Determination of 2,3,7,8-TCDD in Soil," R. D. Kleopfer, K. Yue, and W. W. Bunn, presented before the Division of Environmental Chemistry, American Chemical Society, Washington, D.C., September 1983.

7. "Water Solubility of 2,3,7,8-Tetrachlorodibenzo-p-dioxin," Leland Marple, Robert Brunck, and Lewis Throop, Environmental Science and Technology, Vol. 20, No. 2, 180-182, 1986

EXHIBIT E

QA/QC REQUIREMENTS

Exhibit E - QA/QC Requirements

SUMMARY OF QC ANALYSES

1. Initial and periodic calibration and instrument performance checks.

2. Laboratory method blank analyses (Section 4.1 of QUALITY CONTROL); minimum of one blank per matrix shall be analyzed with each sample batch; an additional blank shall be analyzed when new reagents are used and with each set of samples rerun.

3. Analysis of a batch of samples with accompanying QC analyses:

   3.1 Sample Batch -- <24 samples, including field blank(s), rinsate sample(s) and any reruns generated by prior batch analyses.

   NOTE: See Exhibit C, Section 3, if total samples exceed 24, additional QC analyses are required.

   3.2 Additional QC Analyses Per Batch:

Laboratory method blank for each matrix	1-2
Duplicate sample analysis for each matrix	1-2
TOTAL	2-4

4. "Blind" QC samples may be submitted to contractor as an ordinary soil or sediment or water sample included among the batch of samples. Blind samples include:

   4.1 uncontaminated soil or water,

   4.2 split samples,

   4.3 unlabeled duplicates, and

   4.4 performance evaluation samples.

QUALITY CONTROL

1. Performance Evaluation Samples -- Included among samples in some batches will be samples containing known amounts of unlabeled 2,3,7,8-TCDD.

2. Performance Check Solution and Concentration Calibration Solutions

   2.1 At the beginning of each 12-hour period during which samples are to be analyzed, an aliquot of the performance check solution and an aliquot of concentration calibration solution #1 shall be analyzed to demonstrate adequate GC and MS resolution and sensitivity, response factor reproducibility, and mass range calibration.

These procedures are described in Section 9 of Exhibit D. If any required criteria are not met, remedial action must be taken before any samples are analyzed.

2.2 To validate sample data, the performance check solution must be analyzed at the end of each 12-hour period during which samples are analyzed.

2.2.1 If the contractor laboratory operates only during one 12-hour period (shift) each day, the performance check solution must be analyzed twice (at the beginning and end of the 12-hour period) to validate data acquired during the interim period.

2.2.2 If the contractor laboratory operates during consecutive 12-hour periods (shifts), analysis of the performance check solution at the beginning of each 12-hour period and at the end of the final 12-hour period is sufficient.

2.3 Results of at least two analyses of the performance check solution must be reported with sample data collected during a 12-hour period.

2.4 Deviations from criteria specified for the performance check solution (Section 9.2.6.1, Exhibit D) invalidate all sample data collected between analyses of the performance check solution, and samples shall be rerun (see Exhibit C).

3. The performance check mixture, concentration calibration solutions, and the sample and field blank fortification solutions are to be obtained from EMSL-LV. However, if not available from EMSL-LV, standards can be obtained from other sources, and solutions can be prepared in the contractor laboratory. Concentrations of all solutions containing unlabeled 2,3,7,8-TCDD and not obtained from EMSL-LV must be verified by comparison to the unlabeled 2,3,7,8-TCDD standard solution (concentration of 7.87 ug/mL) that is available from EMSL-LV.

4. Blanks

4.1 Laboratory method blank -- Perform all steps in the analytical procedure (Section 11, Exhibit D) using all reagents, standards, equipment, apparatus, glassware, and solvents that would be used for a sample analysis, using an aliquot of reagent water for the water blank and an aliquot of sodium sulfate for the soil blank.

4.1.1 Except in the case noted below in Section 4.1.3, a laboratory method blank must contain the same amount of $^{37}Cl_4$-2,3,7,8-TCDD and $^{13}C_{12}$-2,3,7,8-TCDD that is added to samples before extraction.

4.1.2 Extract and analyze a laboratory method blank before any samples are extracted and analyzed.

4.1.3 Extract and analyze two laboratory method blanks before new solvents or reagents are used for sample extraction or for column chromatographic procedures. Do not add any $^{37}Cl_4$-2,3,7,8-TCDD or $^{13}C_{12}$-2,3,7,8-TCDD to one blank, to demonstrate that reagents contain no impurities producing an ion current above the level of background noise for m/z 328, 332 and 334.

4.1.4 In addition to the specification in preceding section 4.1.2, extract and analyze a laboratory method blank for each matrix along with each batch of samples.

4.1.5 Acceptable laboratory method blanks must not contain any signal at 320, 322, or 259 which is greater than 2% of the m/z 332 response within ±5 scans of the m/z 332 peak maximum. If the method blank that was extracted along with a batch of samples is contaminated, the associated positive samples must be rerun. (See Exhibit C.)

4.1.5.1 If the above criterion is not met, check solvents, reagents, apparatus, and glassware to locate and eliminate the source of contamination before any samples are extracted and analyzed.

4.1.5.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

4.2 Field Blanks -- Each batch of samples contains a sample of uncontaminated soil/sediment and/or water that is to be fortified with unlabeled 2,3,7,8-TCDD at a concentration of 1 ug/kg for soil or 10 ng/L for water before analysis. In addition to that field blank, a batch of samples may include a rinsate sample, that is a portion of solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that samples have not been contaminated by sampling equipment.

4.2.1 Unfortified field blank -- Analyze with procedures used for environmental samples (Section 11, Exhibit D). This blank may or may not be labeled as such (i.e., it may be a "blind" QC sample).

4.2.2 Fortified (Spiked) Field Blank

4.2.2.1 Weigh a 10-g or measure a 1 L aliquot of the specified field blank sample and add 1.5 mL of the acetone dilution of the 100 uL of field blank fortification solution which contains 0.1 ng/uL of unlabeled 2,3,7,8-TCDD, 0.5 ng/uL of $^{13}C_{12}$-2,3,7,8TCDD, and 0.014 ng/uL of $^{37}Cl_4$-2,3,7,8-TCDD.

4.2.2.2 Extract with the appropriate method from Exhibit D and analyze a 2-uL aliquot.

4.2.2.3 Calculate the concentration (Section 12.1, Exhibit D) of unlabeled 2,3,7,8-TCDD, and the internal standard recovery (Section 12.1.3, Exhibit D) of the measured concentration.

4.2.3 Rinsate Sample

4.2.3.1 To a 100-mL aliquot (or entire sample if less than 100 mL is provided) of equipment rinse solvent (trichloroethylene-rinsate sample), add 1.5 mL of the acetone dilution of 100 uL of the sample fortification solution which contains 0.5 ng/uL of $^{13}C_{12}$-2,3,7,8-TCDD and 0.014 ng/uL of $^{37}Cl_4$-2,3,7,8-TCDD.

4.2.3.2 Using a Kuderna-Danish apparatus, concentrate the volume to approximately 5 mL.

4.2.3.3 Transfer the total 5-mL concentrate in 1-mL portions to a 1 mL-amber mini-vial, reducing volume as necessary with a gentle stream of dry nitrogen.

4.2.3.4 Rinse container with two 0.5 mL portions of hexane and transfer rinses to the 1-mL amber mini-vial.

4.2.3.5 Just before analysis, reduce volume to near dryness, add 5 uL of the recovery standard solution and make to a final volume of 50 uL with isooctane. (Column chromatography is not required.)

4.2.3.6 Analyze an aliquot with the same procedures used to analyze samples (Section 11.5, Exhibit D).

5. Duplicate Analyses

5.1 Laboratory Duplicates -- In each batch of samples, locate the sample specified for duplicate analyses and analyze a second sample aliquot. If no sample is specified for duplicate analysis the laboratory shall select one and analyze it in duplicate. The sample chosen must <u>not</u> be the field blank.

5.1.1 Results of laboratory duplicates must agree within 50% relative difference (difference expressed as percentage of the mean). If the RPD is >50%, Contractor shall immediately contact the Sample Management Office for resolution of the problem. Report all results.

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

Where S_1 and S_2 represent sample and duplicate sample results.

5.1.2 Recommended actions to help locate problem:

5.1.2.1 Analyze an aliquot of the performance check standard to verify satisfactory instrument performance (Section 9, Exhibit D).

5.1.2.2 If possible, determine that no error was made while weighing or measuring sample aliquots.

5.1.2.3 Review analytical procedures with performing laboratory personnel.

6. Identification Criteria

6.1 If any of the four initial identification criteria (Sections 11.6.1-11.6.4, Exhibit D) are not met, the sample is reported not to contain unlabeled 2,3,7,8-TCDD at the maximum possible concentration limit (Section 12.2, Exhibit D).

6.2 When the four initial identification criteria are met, but the fifth criteria, the isotopic abundance ratio for m/z 320 and 322 (Section 11.6.4, Exhibit D) is not met, that sample is presumed to contain interfering contaminants. Contractor shall reextract, clean-up, and reanalyze the sample.

6.3 The recovery of the internal standard $^{13}C_{12}$-2,3,7,8-TCDD should be within a 40 percent to 120 percent recovery window. This is an advisory limit only, an action window may be set when sufficient data is available.

7. Blind QC Samples -- Included among soil and sediment or water samples may be QC samples that are not specified as such to the performing laboratory. Types that may be included are:

7.1 Uncontaminated Soil or Water.

7.1.1 If a false positive is reported for this sample, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C).

7.2 Split Samples -- composited sample aliquots sent to more than one laboratory.

7.3 Unlabeled Field Duplicates -- two aliquots of a composited sample.

7.4 Performance Evaluation Sample -- soil/sediment or water sample containing a known amount of unlabeled 2,3,7,8-TCDD.

7.4.1 If the performance evaluation sample result falls outside the acceptance windows established by EPA, the Contractor shall be required to rerun the entire associated batch of samples (see Exhibit C). NOTE: EPA acceptance windows are based on historical data results.

LABORATORY EVALUATION PROCEDURES

1. On a continuing basis, the EPA Project Officer and/or designated representatives may conduct an evaluation of the laboratory to ascertain that the laboratory is meeting contract requirements. This section outlines the procedures which may be used by the Project Officer or his authorized representative in order to conduct a successful evaluation of laboratories conducting dioxin analyses according to this protocol. The evaluation process consists of the following steps: 1) analysis of a performance evaluation (PE) sample, and 2) on-site evaluation of the laboratory to verify continuity of personnel, instrumentation, and quality assurance/quality control functions. The following is a description of these two steps.

2. Performance Evaluation Sample Analysis

The PE samples are supplied by EMSL-LV to the EPA Regions who include them with the cases submitted to the laboratories. The PE samples are sent in this manner to assure that they are processed and reported in a routine manner by laboratory personnel. The EPA Region client will evaluate the results to verify that the laboratory is continuing to produce acceptable analytical results. The acceptance windows provided by EMSL-LV are based on PE sample performance data and may be updated periodically as the size of the database increases. The PE samples will be representative of the types of samples that will be subject to analysis under this contract.

3. On-Site Laboratory Evaluation

3.1 An on-site laboratory evaluation is performed to verify that the laboratory is maintaining the necessary minimum level in instrumentation and levels of experience in personnel committed to the contract and that the necessary quality control/assurance activities are being carried out. It also serves as a mechanism for discussing laboratory weaknesses identified through routine data audits, PE sample analyses results, and prior on-site evaluation.

3.2 The sequence of events for the on-site evaluations is shown in Figure 1. The Site Evaluation Sheet (SES) (Figure 2) is used to document the results of the evaluation.

Figure 1

EXAMPLE

EVENT SEQUENCE FOR SITE EVALUATION

I. MEETING WITH LABORATORY MANAGER AND PROJECT MANAGER

General discussion of purpose of site visit, purpose of analyses and current contract award status.

II. VERIFICATION OF PERSONNEL

Review qualifications of contractor personnel in place and committed to project (Section I, SES).

III. VERIFICATION OF INSTRUMENTATION

Review equipment in place and committed to project (Section II, SES). The bidder must demonstrate adequate equipment redundancy to ensure capability to perform required analyses in the required time.

IV. QUALITY CONTROL PROCEDURES

Walk through laboratory to review:

1. Sample reception and logging procedures
2. Sample and extract storage area,
3. Procedures to prevent sample contamination,
4. Security procedures for laboratory and samples,
5. Safety procedures,
6. Conformance to written Standard Operating Procedures,
7. Instrument records and logbooks,
8. Sample and data control systems,
9. Procedures for handling and disposing of hazardous materials,
10. Glassware cleaning procedures,
11. Status of equipment and its availability,
12. Procedures for data handling, analysis, reporting, and case file preparation and
13. Chain-of-custody procedures.

V. REVIEW OF STANDARD OPERATING PROCEDURES (SOPs)

Review SOPs with project manager to assure that the laboratory understands the dimensions and requirements of this program.

VI. IDENTIFICATION OF NEEDED CORRECTIVE ACTIONS

Discuss with project manager the actions needed to correct weaknesses identified during site inspection, PE sample analysis or production of reports (hard copies and magnetic tapes) and documentation. Determine how and when corrective actions will be documented, how and when improvements will be demonstrated, and the contractor employee responsible for corrective actions.

Figure 2

EXAMPLE

LABORATORY SITE EVALUATION SHEET (SES)

Laboratory: _____

Date: _____

Type of Evaluation: _____

Contract Number: _____

Contract Title: _____

Personnel Contacted:

Name	Title

Laboratory Evaluation Team:

Name	Title

EXAMPLE

I. Organization and Personnel (Page 1 of 2)

ITEM	YES	NO	COMMENT
Laboratory or Project Manager (individual responsible for overall technical effort): Name: _____			
GC/MS Operator: Name: _____ Experience: 1 year minimum requirement per appropriate instrument			
GC/MS Spectral Interpretation Expert: Name: _____ Experience: 2 years minimum requirement			
Extraction Concentration Expert: Name: _____ Experience: 6 months minimum requirement			
Do personnel assigned to this project have the appropriate educational background to successfully accomplish the objectives of the program?			
Do personnel assigned to this project have the appropriate level and type of experience to successfully accomplish the objectives of this program?			
Is the organization adequately staffed to meet project commitments in a timely manner?			

I. Organization and Personnel (Page 2 of 2)

ITEM	YES	NO	COMMENT
Does the laboratory Quality Assurance Supervisor report to senior management levels?			
Was the Project Manager available during the evaluation?			
Was the Quality Assurance Supervisor available during the evaluation?			

A. General Facilities (Page 1 of 6)

Does the laboratory appear to have adequate workspace (120 sq. feet, 6 linear feet of unencumbered bench space per analyist)?			
Are voltage control devices used on major instrumentation?			
Does the laboratory have a source of distilled/ demineralized water?			
Is the conductivity of distilled/demineralized water routinely checked and recorded?			
Is the analytical balance located away from draft and areas subject to rapid temperature changes?			
Has the balance been calibrated within one year by a certified technician?			
Is the balance routinely checked with class S weights before each use and the results recorded in a logbook?			

A. General Facilities (Page 2 of 6)

ITEM	YES	NO	COMMENT
Are properly filtered exhaust hoods provided to allow efficient work with hazardous/toxic materials?			
Is the laboratory maintained in a clean and organized maner?			
Is a glove box available to allow efficient work with hazardous/toxic materials?			
Are contamination-free work areas provided for the handling of toxic materials?			
Are the toxic chemical handling areas either a stainless steel bench or an impervious material covered with absorbent material?			
Are adequate facilities provided for storage of samples, extracts, and calibration standards, including temperature controlled storage?			
Is the temperature of the cold storage units recorded daily in logbooks?			
Are chemical waste disposal policies/procedures adequate?			
Are contamination-free areas provided for trace level analytical work?			
Can the laboratory supervisor document that trace-free water is available for preparation of standards and blanks?			

A. General Facilities (Page 3 of 6)

ITEM	YES	NO	COMMENT
Is the laboratory secure?			
Can the laboratory supervisor document that organic solvents used are free of trace contaminants?			

Comments on Laboratory Facilities

EXAMPLE

B. Equipment (Page 4 of 6)

1. GC/MS/DS Instrumentation

	Manufacturer	Model	Installation Date
HRGC/MS HRMS ID #			
GC/MS ID #			
Peak Matching Unit ID #			
GC (interfaced with MS) ID#			
Data System ID#			
Data System ID #			

Comments on GC/MS/DS Instrumentation:

EXAMPLE

B. Equipment (Page 5 of 6)

ITEM	YES	NO	COMMENT
Are manufacturer's operating manuals readily available to the operator?			
Is there a calibration protocol available to the operator?			
Are calibration results kept in a permanent record?			
Is service maintenance by contract?			
Is preventative maintenance applied?			
Is a permanent service record maintained in a logbook?			
Has the instrument been modified in any way?			
Is the instrument properly vented?			
Is a 9-track mag tape available?			
Is a split/splitless capillary injector in place?			
Is the column direct to the source?			
Are sufficient in-house replacement parts available?			

B. Equipment (Page 6 of 6)

Comments on GC/MS Instrumentation

III. Documentation (Page 1 of 2)

When reviewing documentation, give special attention to:

a) traceability
b) neatness and completion

A. Documentation/Tracking

ITEM	YES	NO	COMMENT
Is a sample custodian designated? If yes, name of sample custodian. Name: _____			
Are the sample custodian's procedures and responsibilities documented? If yes, where are these documented?			
Are written Standard Operating Procedures (SOP) developed for receipt of samples? If yes, where are the SOP documented (laboratory manual, written instructions, etc.)?			
Are quality assurance procedures documented and available to the analysts? If yes, where are these documented?			
Are written Standard Operating Procedures (SOP) developed for compiling and maintaining sample document files? If yes, where are the SOP documented (laboratory manual, written instructions, etc.)?			
Are the magnetic tapes stored in a secure area?			
Is a permanently-bound notebook with preprinted, consecutively-numbered pages being used?			

B. Documentation/Notebooks (Page 2 of 2)

ITEM	YES	NO	COMMENT
Is the type of work clearly displayed on the notebook (i.e., EPA Extraction)?			
Is the notebook maintained in a legible manner?			
Are entries noting anomalies routinely recorded?			
Has the analyst avoided obliterating entries?			
Are inserts (i.e., chromatograms, computer printout, etc.) permanently affixed in notebook and signed across insert edge and page?			
Has the supervisor of the individual maintaining the notebook personally examined and reviewed the notebook periodically, and signed his/her name therein, together with the date and appropriate comments as to whether or not the notebook is being maintained in an appropriate manner?			
Where applicable, is the notebook holder referencing reports or memoranda pertinent to the contents of an entry?			

EXAMPLE

IV. Analytical Methodology (Page 1 of 2)

ITEM	YES	NO	COMMENT
Are the required methods used?			
Is there any unauthorized deviation from contract methodology?			
Are written analytical procedures provided to the analyst?			
Are distilled-in-glass grade or other high purity chemicals used to prepare standards?			
Are fresh analytical standards prepared at a frequency consistent with good QA?			
Are reference materials properly labeled with concentrations, date of preparation, and the identity of the person preparing the sample?			
Is a standards preparation and tracking logbook maintained?			
Do the analysts record bench data in a neat and accurate manner?			
Is the appropriate instrumentation used in accordance with the required protocol(s)?			

EXAMPLE

Comments on Analytical Methods and Practices (Page 2 of 2)

V. Quality Control Manual Checklist (Page 1 of 2)

ITEM	YES	NO	COMMENT
Does the laboratory maintain a Quality Control Manual?			
Does the manual address the important elements of a QC program, including the following:			
a. Personnel?			
b. Facilities and equipment?			
c. Operation of instruments?			
d. Documentation of procedures?			
e. Procurement and inventory practices?			
f. Preventive maintenance?			
g. Reliability of data?			
h. Data validation?			
i. Feedback and corrective action?			
j. Instrument calibration?			
k. Recordkeeping?			
l. Internal audits?			

EXAMPLE

V. Quality Control Manual Checklist (Page 2 of 2)

ITEM	YES	NO	COMMENT
Are QC responsibilities and reporting relationships clearly defined?			
Have standard curves been adequately documented?			
Are laboratory standards traceable?			
Are quality control charts maintained for each routine analysis?			
Do QC records show corrective action when analytical results fail to meet QC criteria?			
Do supervisory personnel review the data and QC results?			

EXAMPLE

VII. Summary

A. Summary Checksheet (Page 1 of 2)

ITEM	YES	NO	COMMENT
Do responses to the evaluation indicate that project and supervisory personnel are aware of QA and its application to the project?			
Do project and supervisory personnel place positive emphasis on QA/QC?			
Have responses with respect to QA/QC aspects of the project been open and direct?			
Has a cooperative attitude been displayed by all project and supervisory personnel?			
Does the organization place the proper emphasis on quality assurance?			
Have any QA/QC deficiencies been discussed before leaving?			
Is the overall quality assurance adequate to accomplish the objectives of the project?			
Have corrective actions recommended during previous evaluations been implemented?			
Are any corrective actions required? If so, list the necessary actions below.			

EXAMPLE

VI. Data Handling Checklist (Page 1 of 1)

ITEM	YES	NO	COMMENT
Are data calculations checked by a second person?			
Are data calculations documented?			
Do records indicate corrective action that has been taken on rejected data?			
Are limits of detection determined and reported properly?			
Are all data and records retained for the required amount of time?			
Are quality control data (e.g., standard curve, results of duplication and spikes) accessible for all analytical results?			

EXAMPLE

B. Summary Comments and Corrective Actions (Page 2 of 2)

EXHIBIT F

SPECIFICATIONS FOR
CHAIN-OF-CUSTODY, DOCUMENT CONTROL,
AND STANDARD OPERATING PROCEDURES

NOTE: The Contractor shall not deviate from the procedures described herein without the prior written approval of the Contracting Officer: Provided, that the Contracting Officer may ratify in writing such deviation and such ratification shall constitute the approval required herein.

SPECIFICATIONS FOR CHAIN-OF-CUSTODY, DOCUMENT CONTROL PROCEDURES, AND STANDARD OPERATING PROCEDURES

1. SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of the hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following chain-of-custody procedures have been established.

1.1 Sample Identification

To assure the traceability of samples through the laboratory, a method for sample identification shall be developed and documented in the laboratory SOPs (see Section 3). Each sample or sample preparation container shall be labelled with a unique number identifier (or the EPA sample number). This identifier shall be cross-referenced to the sample tag number and the EPA sample number. There shall be a written description of the method of assigning this identifier and attaching it to the sample bottle, included in the laboratory SOPs.

1.2 Chain-of-Custody Procedures

Because of the nature of the data being collected, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample custody, the chain-of-custody procedures described below shall be followed.

1.2.1 A sample is under custody if:

1.2.1.1 It is in your actual possession,

1.2.1.2 It is in your view after being in your physical possession,

1.2.1.3 It was in your possession and then you locked or sealed it up to prevent tampering, or

1.2.1.4 It is in a secure area.

1.2.2 Upon receipt of the samples in custody, the contractor shall inspect the shipping container and sample bottles, and shall document receiving information as specified

in Section 3.2. The sample custodian or a designated representative shall sign and date all appropriate receiving documents at the time of receipt (i.e., EPA chain-of-custody forms, traffic reports, airbills, etc.). The contractor shall contact SMO if documents are absent, information on receiving documents does not agree, custody seals are not intact, or the sample is not in good condition. The contractor shall document resolution of any discrepancies.

2. DOCUMENT CONTROL PROCEDURES

The goal of the laboratory document control program is to assure that all documents for a specified case will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to EPA or are available upon request form EPA prior to the delivery schedule.

2.1 Preprinted Data Sheets and Logbooks

Preprinted data sheets shall contain the name of the laboratory and be dated and signed by the analyst or individual performing the work. All documents produced by the laboratory which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the case file. For that reason, all observations and results recorded by the laboratory but not on preprinted data sheets are entered into permanent laboratorey logbooks. The person responsible for the work shall sign and date each entry and/or page in the logbook. When all data from a case is compiled, copies of all EPA case-related logbook entries shall be included in the documentation package. Analysts' logbook entries must be in chronological order and shall include only one case per page. Instrument run logs shall be maintained so as to enable a reconstruction of the run sequences of individual instruments.

Because the laboratory must provide copies of the instrument run logs to EPA, the laboratory may exercise the option of using only laboratory or SMO sample identification numbers in the logs for sample ID rather than government agency or commercial client names.

Using laboratory or SMO sample IDs only in the run sequences will assist the laboratory in preserving the confidentiality of commercial clients.

2.2 Error Correction Procedure

All documentation in logbooks and other documents shall be in ink. If an error is made, corrections shall be made by crossing a line through the error

and entering the correct information. Changes shall be dated and initialed. No information shall be obliterated or rendered unreadable.

2.3 Consistency of Documentation

Before releasing analytical results, the laboratory shall assemble and cross-check the information on sample tags, custody records, lab bench sheets, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or case is consistent throughout the case file.

2.4 Document Numbering and Inventory Procedure

In order to provide document accountability of the completed analysis records, each item in a case shall be inventoried and assigned a serialized number and identifier associating it to the case and Region.

Case # - Region - Serialized number (For example: 75-2-0240)

The number of pages of each item must be accounted for if each page is not individually numbered. All documents relevant to each case, including logbook pages, bench sheets, mass spectra, chromatograms, custody records, library search results, etc., shall be inventoried. The laboratory shall be responsible for ensuring that all documents generated are placed in the file for inventory and are delivered to EPA. Figure 1 is an example of a document inventory.

2.5 Shipping Data Packages and Case Files

The contractor shall have written procedures to document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that it cannot be opened without damaging or breaking the seal. The contractor shall also document what was sent, to whom, the date, and the method (carrier) used.

3. SPECIFICATIONS FOR STANDARD OPERATING PROCEDURES

The contractor must have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample storage, tracking the analysis of samples and assembly of completed data.

An SOP is defined as a written narrative step-wise description of laboratory operating procedures including examples of laboratory documentation. The SOPs must accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1 The contractor shall have a designated sample custodian responsible for receipt of samples and have written SOPs describing his/her duties and responsibilities.

3.2 The contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.2.1 Presence or absence of EPA chain-of-custody forms

3.2.2 Presence or absence of airbills

3.2.3 Presence or absence of traffic reports or SAS packing lists

3.2.4 Presence or absence of custody seals on shipping and/or sample containers and their condition

3.2.5 Presence or absence of sample tags

3.2.6 Sample tag ID numbers if not recorded on the chain-of-custody record(s) or packing list(s)

3.2.7 Condition of the shipping container

3.2.8 Condition of the sample bottles

3.2.9 Verification of agreement or non-agreement of information on receiving documents

3.2.10 Resolution of problems or discrepancies with the Sample Management Office

3.3 The contractor shall have written SOPs for maintenance of the security of samples after log-in and shall demonstrate security of the sample storage areas and laboratory. The SOPs shall specifically include descriptions of all storage areas for EPA samples in the laboratory. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.4 The contractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include the following:

3.4.1 A description of the documentation used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.

3.4.2 A description of the documentation used to record calibration and QA/QC laboratory work.

3.4.3 Examples of the document formats and laboratory documentation used in the sample receipt, sample storage, sample transfer, and sample analyses.

3.5 The contractor shall have written SOPs for organization and assembly of all documents relating to each EPA case. Documents shall be filed on a case-specific basis. The procedures must ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the case are compiled in one location for submission to EPA. The system must include a document numbering and inventory procedure.

4. HANDLING OF CONFIDENTIAL INFORMATION

A contractor conducting work under this contract may receive EPA-designated confidential information from the agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

4.1 All confidential documents shall be under the supervision of a designated document control officer (DCO).

4.2 Confidential Information

-Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO logs these documents into a Confidential Inventory Log. The information is then made available to authorized personnel but only after it has been signed out to the person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA contracting officer. The DCO will enter all copies into the document control system. In addition, this information may not be disposed of except upon approval by the EPA contracting officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record of the disposition in the Confidential Inventory Log.

Figure 1

232-2-0001

Case No. 232

Example

DOCUMENT INVENTORY

Document Control #*	Document Type	# Pages
232-2-0001	Case File Document Inventory Sheet	1
232-2-0002	Chain-of-Custody Records	2
232-2-0003	Shipping Manifests	2
232-2-0004	Sample Tags	50
232-2-0005	SMO Organics Traffic Reports	10
232-2-0006	GC/MS spectra for sample B0310	20
232-2-0007	GC/MS spectra for sample B0311	20
232-2-0008	GC/MS spectra for sample B0319	20
232-2-0009	Analyst's logbook pages	6
232-2-0010	GC/MS library search worksheets	15
232-2-0011	GC instrument log pages	5
232-2-0012	GC/MS QC data sheets	4
etc.	etc.	etc.

* This number is to be recorded on each set of documents.