Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)

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Annual Review

Reviewed by: _____ Date: _____
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I.0 Scope

I.I This Standard Operating Procedure (SOP) applies to the evaluation of Routine Analytical Services (RAS) inorganic data generated in accordance with the EPA Contract Laboratory Program (CLP) protocols.

1.2 This Region 2 inorganic data validation SOP is used to determine the usability of analytical data generated from water and soil/sediment samples collected from Superfund sites in EPA Region 2.

1.3 Data should be generated and validated in accordance with the site specific Project Quality Objectives (PQOs) developed prior to the sample collection event. This SOP can be customized to validate the data according to the site specific PQOs. If the site specific DQOs are not available, this SOP must be used in its entirety.

1.4 This SOP is based, for the most part, upon analytical and quality assurance requirements specified in the Statement of Work SOW-ILM05.3, as well as in the final (October 2004) of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. The SOP Checklist, Appendix A.1, provides guidance in conducting the data validation. The result of the use of this SOP is a **Total Review** of the data: **Technical plus Contract - Compliance Review.**

2.0 Contract Compliance Review

This type of review is the first step in data validation which is carried out to ensure that the CLP laboratory has analyzed the environmental samples in accordance with the Statement of Work (SOW), and provided a data package which is both complete and compliant. This means that laboratory's procedures were performed exactly as specified in the CLP Statement of Works (SOW) and the data package contains all the deliverables including the information required under the contract.

2.1 Completeness

The data validator must check the entire data package to ensure that all deliverables required under the CLP contract are present and legible. In addition, copies of the Contract Compliance Screening (CCS) report, re-submittal from the laboratory, and Regional documentation should also be present in the data package. In Region 2, the data package completeness check is currently performed by the Regional Sample Control Coordinator (RSCC)for each Sample Delivery Group (SDG). The data package is not released to the data validator until all the required deliverables are received
from the laboratory.

2.2 Compliance

The data validator must check to ensure that all steps from sample receipt through sample preparation, analysis, data calculation and reporting are documented, and the information/data required under the contract is present in the appropriate reporting Forms and laboratory logs.

2.3 Contract Compliance Screening (CCS)

This screening step essentially checks the data package for the Completeness and Compliance requirements, and is performed by the Sample Management Office (SMO) currently operated by Computer Sciences Corporation (CSC), an EPA contractor. The CCS Report outlines the incomplete and non-compliant items as "Defects" in the data package, and is sent to the laboratory which is required to

provide additional or missing information/data required under the contract. The CCS Report for each SDG is transmitted electronically by the SMO to the Regional office. The CCS Report is intended to aid the data validator in locating any problems, both corrected and uncorrected. The incorrect original deliverable(s)of the data package must be replaced by the re-submittal(s)received from the laboratory in response to the CCS Report. The data validation should, however, be carried out even if the CCS Report is not available.

Web-based CCS is available for CLP laboratories to check their data prior to its delivery to EPA.

3.0 **Technical Review**

Technical review of the RAS data is carried out on the complete and compliant data to ensure its **validity** (i.e., data is of known quality and scientifically valid) and **usability** (i.e., data set is sufficiently complete and of sufficient quality to support a decision or an action described in the specific objectives of a data collection activity). The technical review process provides information on analytical limitations of data, if any, based on specific Quality Assurance/Quality Control (QA/QC) criteria. This is accomplished by performing an in-depth review of both the field deliverables which document the field sampling activities, and the laboratory analytical data deliverables which document the laboratory activities carried out to generate the reported data. Essentially, the validator shall first ensure that the data package is complete and compliant. The validator shall then evaluate data/information on all these deliverables (Final data sheets, Forms for QC analyses Chain-of-Custody/Traffic Report Forms, raw data, etc.) against the QA/QC acceptance criteria specified in the SOP “Checklist” (Appendix A.1). The validator must answer each question in the “ Checklist” and take an appropriate action as required under “Action” to qualify the data. As a result of the technical review, the data validator may qualify some of the data as **rejected** or as **estimated.** The data validator shall write a **Data Review Narrative** documenting the qualified data and the reason(s) for the qualification.

3.1 If the **raw data** necessary to support the reported results are not provided, the data validation must not be performed. The laboratory must be contacted to obtain missing raw data.

3.2 If batch quality control analyses are performed on samples other than **site specific samples**, data must not be validated
or at best be considered as estimated. The data user must be notified of this action.

3.3 **QA/QC Acceptance Criteria**

In order that reviews be consistent among reviewers, QA/QC protocol (stated in Appendix A.l) should be strictly adhered to. If a lab provides more than one set of QC analyses or more than one particular QC analysis for an SDG, the validator shall use the worst QC analysis to evaluate the SDG data. Professional judgement should only be used in the rare instances not addressed in the “Checklist”.

3.4 **Data Validation Flags**

Three types of data validation flags (J, R & U) are used in Region 2 to qualify the data.

3.4.1 **Flag "R" indicates Rejected Data**

Sample results determined to be unacceptable must preferably be lined over and flagged " R" with a red pencil only on the Inorganic Analysis Data Sheets (CLP Form I's). Data rejected on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user **must not** use the rejected data to make environmental decisions.

3.4.2 **Flag "J" indicates Estimated Data**

Sample results determined to be estimated must be flagged "J" with a red pencil only on the CLP Form I's. Data are flagged (J) when a QC analysis falls outside the primary acceptance limits. The qualified "J" data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result even though several associated QC analyses may fail. The "J" data may be biased high or low.

3.4.3 **Flg "U" indicates Non-Detects**

Sample results ≥ MDL associated with a contaminated blank are flagged "U" with a red pencil only on Form I's.

4.0 **Contractual Qualifiers**

The CLP laboratory applies contractual qualifiers on all Form I'S and the QC Forms when QC analyses are outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets with the exception of U and J. The contractual qualifiers and their meanings are as follows:

N : This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E : This qualifier indicates the presence of interference, and is applied when the ICP serial dilution analysis is outside the control limits.

***** : This qualifier indicates the lack of precision, and is applied to sample results on Form I's and Form VI when the Lab Duplicate analysis is outside the control limits.

U : This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analysis is indicated by the Contract Required Quantitation Limit (CRQL) of that analyze suffixed with "U".

J : This is a concentration qualifier that the laboratory applies to a positive result below the CRQL(i.e.,≥MDL but <CRQL).

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

4.0 Rounding Rule

The data reviewer must follow the standard practice to round off percent recoveries on the QC reporting forms.

5.0 Data Review Narrative (Appendix A.2)

The data review narrative should be written using the format of Appendix A.2. The narrative should indicate the QC analyses outside the acceptance limits and the actions taken to qualify the associated data. The narrative should be prepared on a Personal Computer or a typewriter. If hand-written, under no circumstances should a pencil be used to write the narrative. The Data Review Narrative should be written in four (4) Sections: (i)Data Case Description, (ii)Complete SDG File (CSF) Audit Section, (iii) Technical Review Section, and (iv) Contract-Problems/Non-Compliance Section.

5.1 Data Case Description Section

The data validator must briefly describe the data case in this Section, outlining important information such as the number of samples, their matrix, sampling date(s), analysis (TAL metals, mercury or cyanide), samples used for QC analyses, Field Blank(s), Field Duplicates, etc.

5.2 Complete SDG File (CSF) Audit Section

The data validator must perform an audit on each SDG in the data package to ensure that all SDG-specific documents (sampling, samples shipping and receiving, telephone contact logs, etc.) are present in the data case. The audit shall also discover any discrepancy in the deliverables. In Region 2, this audit is currently performed by the ESAT data validator and its findings reported under “Comments” on a CSF inventory checklist. The validator informs the CLP Project Officer (PO) of the missing or additional information/deliverable required for data validation. The PO then contacts the lab for the desired deliverable/information. The findings of the CSF audit are reported in the CSF Section of the Data Review Narrative (Appendix A.2).

5.3 Technical Review Section

The data validator shall report in this Section only the rejected (R) and estimated data (J) and the data rendered non-detects (U) as a result of technical review. It is imperative that the data reviewer highlights (i) QC analysis criteria applied to reject (R) or flag (J, U) the data, (ii) Samples rejected (R) or flagged (J, U), and (iii) the QC analysis out of control limits. The rest of the data that are not qualified (rejected or estimated) are not reported in this Section, and should be considered **fully useable.**

5.4 Contract-Problems/Non-Compliance Section

All the CLP non-compliant items detected during data review must be reported in this Section.

6.0 Computer-Aided Data Review and Evaluation (CADRE)

CADRE is a computer program that performs semi-automated Quality Assurance (QA) and Quality Control (QC) checks of results from the chemical analysis of soil and water samples according to the CLP protocols. After the CADRE data qualification is complete, a Lotus 1,2,3 spreadsheet or an XLS spreadsheet with data validation qualifiers (R,J,U) is generated for each SDG. Currently, Sample Management Office (SMO) performs this task using Data Assessment Tool (DAT), a software-driven process, and forwards to the Regions the customized electronic spreadsheets (Lotus 1,2,3 or XLS spreadsheet) and QC reports via the DART (Data Assessment Rapid Transmittal) system. Manual data validation is performed in conjunction with electronic data validation which can only be done by a trained and experienced data validator. The manual data review complements CADRE's findings to complete an assessment of data quality in a shorter time than by a solely manual process. The data validator must review the XLS or Lotus 1,2,3 spreadsheet against Form I's to ensure that the same results on Form I's and the Spreadsheet are qualified with the same data validation qualifiers. The spreadsheet for each SDG is provided with the Data Review Narrative.

7.0 Performance Evaluation Sample(PES)Based Data Validation Strategy

7.1 Scope and Summary

This strategy offers the use of Performance Evaluation Samples (PES) in the data validation process as a means of ensuring the quality of the CLP data while significantly reducing the validation time. The single blind PES provided by EPA (or any other reputable firm) is analyzed with samples of each matrix in a Sample Delivery Group (SDG). A software program (e.g.,PEAC TOOLS, SPS Web or equivalent)is used to determine whether or not the PES results fall within the previously statistically determined acceptance limits ("Action Low" and "Action High")for the Contaminants of Concern (**COC**). The PES results falling within the Action Limits are considered as acceptable results and may be designated as "Passed" analytes, and results of the analytes falling outside the Action Limits are considered as unacceptable and may be designated as "Failed" analytes. In either case ("Passed" Analytes or "Failed" analytes), the associated data is validated according to the Region 2 data validation SOP HW-2 in conjunction with the latest version of the WinCadre QC reports. The following strategy (procedure) is used:

7.2 "Passed" COC

If the COC in an SDG are within statistically generated Action Limits, the data validation is conducted according
to QC analyses indicated by check marks (√)in the "Review COC For" column of the Table I. The SDG samples are validated using the Region 2 data validation SOP in

conjunction with the latest version of the WinCADRE QC reports. The validation flags (J, R, U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS spreadsheet. Corrections, if needed, are
then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.3 **"Failed" COC**

If the COC in an SDG are not within the statistically generated Action Limits, the data validation is conducted according to the data validation SOP QC Criteria indicated by check marks (√)in the "Review COC For" column of Table II. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The data validation flags (J,R,U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS Spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.4 **COC "Not Evaluated"**

Acceptance limits for the analytes not present/spiked in the PE sample are not provided on the PES Scoring Evaluation Report. Such analytes will be marked as "Not Evaluated" in the PES Evaluation Column. These analytes will be validated much the same way as the "Failed Analytes".

The failed analytes and the analytes not present/spiked in the PE sample require data validation according to the QC criteria specified in Table II, and are identified by the TOPO in the TDF for the Case/SDG.

Table I

Passed PES - All Contaminants of Concern are within the limits
(Action Low ≤ PES Result ≤ Action High)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	
Post Digestion Spike	
Laboratory Duplicate	
Field Duplicates Comparison	√
Lab Control Sample	
ICP Serial Dilution	
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied. The Contract
- Compliance can be checked after the PES validation strategy is applied.

Table II
Failed PES - Contaminants of Concern are not within the limits
(PES Result ≤ Action Low, PES Result ≥ Action High **OR** The Limits Not Established)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	√
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	√
Post Digestion Spike	
Laboratory Duplicate	√
Field Duplicates Comparison	√
Lab Control Sample	√
ICP Serial Dilution	√
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	√
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied.

- The Contract Compliance can be checked after the PES validation strategy is applied.

8.0 **Sampling Trip Report**

The sampler prepares a Sampling Trip Report for each sampling event and sends it to the RSCC. The report provides details of all activities performed for each sampling event on the Superfund site. It also lists the field QC samples such as Field Duplicates, Field/Rinse Blanks, sampling time and date for each sample, and samples associated with each field/rinse blank. The validator must use this information to evaluate the Field Duplicate pairs as well as the samples associated with contaminated Field/Rinse Blanks.

9.0 **Telephone Record Log (Appendix A.3)**

A Telephone Record Log (Appendix A.3) must be written by the data validator when a deliverable is missing or a clarification is needed about a lab procedure. The data validator should outline a basic profile of the Case on the Telephone Record Log Form, clearly indicating the reason(s) for inquiry and forward this Form to CLP PO/TOPO who will contact the lab to receive the missing document or information. The original Telephone Record Log is kept in the data package and a copy attached to the Data Review Narrative.

10.0 **Request for Re-Analysis (Appendix A.6)**

Data validator must note all items of contract non-compliance in the Data Review Narrative. If holding times and sample storage times have not been exceeded, the Project Officer (PO) may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record" form (Appendix A.4).

11.0 **CLP Data Assessment Summary Form (Appendix A.7)**

Fill in the total number of analytes performed by different methods and the number of analytes rejected (R) or flagged (J) as estimated due to corresponding quality control criteria. Place an "X" in boxes wherever analyses were not performed, or criteria do not apply.

12.0 **Data Review Log:**

It is recommended that the data validator maintain a log of the reviews completed to document:

a. Case number
b. SDG # (s)
c. number of samples
d. matrix of samples
e. contract laboratory
f. site name
g. start-date of the data case review
h. completion-date of the data case review
i. actual hours spent
j. reviewer's signature

13.0 **Record of Communication -**

This is a Regional document prepared and provided by the RSCC for each data package. The ROC indicates the Case #, site name, samples and sample matrix and the laboratory name. The presence of a ROC in a data package is an indication that the package has been reviewed by the RSCC for completeness and is ready for data validation.

14.0 **Forwarded Paperwork**

Upon completion of review, the following are to be forwarded to EPA for final review:

a. Data package
b. Completed data assessment checklist (Appendix A.1,original)
c. Original and a copy of completed data review narrative Appendix A.2)
d. CLASS Contract Compliance Screening (CCS) report
e. Telephone Record Log (Appendix A.3)
f. Field Duplicates Form (Appendix A.4)
g. Total/Dissolved Concentrations Form (Appendix A.5)
h. CLP Re-analysis Request/Approval Record Form (Appendix A.6)
i. Data Assessment Summary Form (Appendix A.7)
j. CADRE Spreadsheet on a computer diskette.

ACRONYMS

AA Atomic Absorption
AOC Analytical Operations/Data Quality Center
CADRE Computer-Aided Data Review and Evaluation
CCB Continuing Calibration Blank
CCS Contract Compliance Screening
CCV Continuing Calibration Verification
CLP Contract Laboratory Program
CO Contracting Officer
COC Contaminants of Concern
CRI CRQL Check Standard
CRQL Contract Required Quantitation Limit
CSF Complete SDG File
CVAA Cold Vapor AA
DART Data Assessment Rapid Transmittal
DAT Data Assessment Tool
DF Dilution Factor
DQO Data Quality Objective
ICB Initial Calibration Blank
ICP Inductively Coupled Plasma
ICP-AES Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS Inductively Coupled Plasma - Mass Spectrometry
ICS Interference Check Sample
ICV Initial Calibration Verification
LCS Laboratory Control Sample
LRS Linear Range Sample
MDL Method Detection Limit
NIST National Institute of Standards and Technology
OERR Office of Emergency and Remedial Response
OSWER Office of Solid Waste and Emergency Response
PB Preparation Blank
PE Performance Evaluation
%D Percent Difference
%R Percent Recovery
%RI Percent Relative Intensity
%RSD Percent Relative Standard Deviation
%S Percent Solids
PO Project Officer
QA Quality Assurance
QAPP Quality Assurance Project Plan
QC Quality Control
RPD Relative Percent Difference
RSCC Regional Sample Control Center

SDG Sample Delivery Group
SMO Sample Management Office
SOP Standard Operating Procedure
SOW Statement of Work
TAL Target Analyze List
TR/COC Traffic Report/Chain of Custody Documentation

Inorganic Target Analyze List And Contract Required Quantitation Limits (CRQLs)

Analyze	CAS Number	ICP-AES CRQL Water Ug/L	ICP-AES CRQL Soil mg/kg	ICP-MS CRQL Water Ug/L
Aluminum	7429-90-5	200	20	---
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	-----
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	----
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	-----
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	---
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	-----
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	-----
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	----

Site:

Case #:

SDG #:

Samples: Soil Water

	YES	NO	N/A
A.l.l **Contract Compliance Screening Report**			
Present?	[___]	___	___
ACTION: If no, contact RSCC/PO.			
A.l.2 **Record of Communication (from RSCC)**			
Present?	[___]	___	___
ACTION: If no, request from the RSCC.			
A.1.3 **Sampling Trip Report**			
Present and complete?	[___]	___	___
ACTION: If no, contact RSCC/PO.			
A.l.4 **Chain of Custody/Sample Traffic Report**			
Present?	[___]	___	___
Legible?	[___]	___	___
Signature of sample custodian present?	[___]	___	___
ACTION: If no, contact RSCC/WAM/PO.			
A.l.5 **Cover Page**			
Present?	[___]	___	___
Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?	[___]	___	___
Do the sample identification numbers on the Cover Page agree with sample Identification numbers on:			
(a) Traffic Report Sheet?	[___]	___	___

	YES	NO	N/A
(b) Form I's?	[___]	___	___
Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 **SDG Narrative, DC-1 & DC-2 Form**

Is the SDG Narrative present?	[___]	___	___
Is Sample Log-In Sheet(Form DC-1) present and complete?	[___]	___	___
Is Complete SDG Inventory Sheet(Form DC-2) present and complete?	[___]	___	___

ACTION:
If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

A.1.7 **Form I to XV**

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?	[___]	___	___
Laboratory Code?	[___]	___	___
RAS/Non-RAS Case No.?	[___]	___	___
SDG No.?	[___]	___	___

		YES	NO	N/A
	Contract No.?	[___]	___	___

ACTION:
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2	After comparing values on Forms I-IX against the raw data, do any computation/ transcription errors exceed 10% of the reported values on the Forms for:			
	(a) all analytes analyzed by ICP-AES?	___	[_]	___
	(b) all analytes analyzed by ICP-MS?	_	[___]	_
	(c) Mercury?	__	[_]	_____
	(d) Cyanide?	_	[_]	_

ACTION:
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 Raw Data

Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 Digestion/Distillation Log

Digestion Log for ICP-AES (Form XII)present?	[___]	___	___
Digestion Log for ICP-MS (Form XII) present?	[___]	___	___
Digestion Log for mercury (Form XII) present?	[___]	___	___
Distillation Log for cyanide (Form XII) present?	[___]	___	___
Are pH values for metals and			

	YES	NO	N/A
cyanide reported for each aqueous sample?	[___]	__	__
Are percent solids calculations present for soils/sediments?	[__]	___	___
Are preparation dates present on the sample preparation logs/bench sheets?	[___]	__	__

NOTE:
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:

ICP-AES?	[___]	__	__
ICP-MS?	[___]	___	___
Mercury?	[__]	__	__
Cyanide?	[___]	___	___

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

Legible?	[__]	__	__
Properly labeled?	[__]	__	__

Are all field samples, QC samples and field QC samples present on:

Digestion/Distillation log?	[___]	___	___
Instrument Printouts?	[__]	__	__

ACTION:
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.

		YES	NO	N/A

A.1.9 **Technical Holding Times**: (Aqueous and soil samples)

(Examine sample Traffic Reports and digestion/distillation logs to determine the holding time from the sample collection date to the sample preparation date.)

A.1.9.1	Cyanide distillation(14 days)exceeded?	__	[__]	__
	Mercury analysis(28 days) exceeded?	__	[___]	__
	Other Metals analysis(180 days)exceeded? ___	___	[__]	__

ACTION:
If yes, reject (R) and red-line non-detects and flag as estimated (J)results ≥ MDL even if sample(s) was preserved properly.

NOTE:
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must be prepared. Report for each sample the number of days that were exceeded. (Subtract the sample collection date from the sample preparation date). Attach this list to the data review narrative.

A.1.9.2	Is pH of aqueous samples for:			
	Metals Analysis ≤ 2?	[___]	___	___
	Cyanide Analysis ≥ 12?	[__]	__	__

ACTION:
If no for any of the above, flag non-detects as “R” and detects as “J”.

A.1.9.3	Is the cooler temperature ≤ 10 C°?	[__]	__	__

ACTION:
If cooler temperature is >10 ^{0}C , flag non-detects as “UJ” and detects as “J”.

A.1.10 **Final Data Correctness - Form I**

A.1.10.1 Are Form I's for all samples

	YES	NO	N/A
present and complete?	[___]	___	___

ACTION:
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result?	[___]	__	__
Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)?	[___]	___	___
Are results on Form I'S reported by correct significant figures?	[___]	___	__
Are soil sample results on Form I's corrected for percent solids?	[___]	___	___
Are all "less than MDL" values reported by the CRQLs and coded with "U"?	[___]	___	___
Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"?	[___]	___	___
Are appropriate contractual quality control and Method qualifiers used?	[___]	____	___

ACTION:
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [___] ___ ___

Was a brief physical description

		YES	NO	N/A
	of the samples before and after digestion given on the Form I's?	[___]	___	__
	Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?	[___]	___	___
	ACTION: If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.			

A.1.11 **Initial Calibration**

A.1.11.1	Is a record of at least 2 point (A blank and a standard)calibration present for ICP-AES analysis?	[___]	___	___
	Is a record of at least 2 point (a blank and a standard)calibration present for ICP-MS analysis?	[___]	___	___
	Is a record of at least 5 point calibration (a blank & 4 standards)present for Hg analysis?	[___]	___	___
	Is a record of at least 4 point calibration (a blank & 4 standards)present for cyanide?	[___]	___	___
	ACTION: If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).			
	Is one initial calibration standard at the CRQL level for cyanide and mercury?	[___]	___	___
	ACTION: If no, write in the Contract Problem/ Non-Compliance Section of the Data Review Narrative.			
A.1.11.2	Is the curve correlation coefficient $\geq$ 0.995 for:			

		YES	NO	N/A
	Mercury Analysis?	[___]	___	___
	Cyanide Analysis?	[___]	___	___
	ICP-AES(more than 2 point Calib.)?	[___]	___	___
	ICP-MS (more than 2 point calib.)?	[___]	___	___

ACTION:
If no, qualify the associated sample results ≥ MDL as estimated "J" and non-detects as "UJ".

NOTE:
The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

A.1.12 **Initial and Continuing Calibration Verification- Form IIA**

A.1.12.1	Present and complete for every metal and cyanide?	[___]	___	___
	Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?	[___]	___	___

ACTION:
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

A.1.12.2	Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?	[___]	___	___

ACTION:
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.12.3	Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?	[___]	___	___

	YES	NO	N/A

ACTION:
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results ≥ MDL as estimated (J).

A.1.12.2 Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:

Metals - 90-110%R?	[___]	___	___
Hg - 80-120%R?	[___]	___	___
Cyanide - 85-115%R?	[___]	___	___

ACTION:
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results(≥ MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg;116-130% for CN). Reject (R) and red-line only
detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects)if the recovery is less than 75%(65% for Hg;70% for CN).

NOTE:
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3 Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)? [___] ___ ___

ACTION:
If no, Qualify all cyanide results ≥ MDL as "J".

A.1.13 **CRQL Standard Analysis - Form IIB**

A.1.13.1 For each ICP-AES run, was a CRI

	YES	NO	N/A
(CRQL or MDL when MDL > CRQL) standard analyzed? **(Note:CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)**	[___]	___	___
For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?	[___]	___	___ ___
For each mercury run, was a CRQL standard analyzed?	[___]	___	___
For each cyanide run, was a CRQL standard analyzed?	[___]	___	___

ACTION:
If no for any of the above, write this deficiency in the Contract Problems/ Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL)as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value ± CRQL
ICP-MS Analysis - *True Value ± CRQL
Mercury Analysis - *True Value ± CRQL
Cyanide Analysis - *True Value ± CRQL
*** True value of the CRQL Standard**

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis? [___] ___ ___

ACTION:
If no, write in the Contract Problem/ Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.

	YES	NO	N/A
Is the CRQL standard within control limits for:			
Metals(ICP-AES/ICP-MS)- 70 - 130%?	[___]	___	___
Mercury- 70 - 130%?	[___]	___	___
Cyanide - 70 - 130%?	[___]	___	___

ACTION:
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 150%, reject(R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J)detects ≥ 2xCRQL but < ICV/CCV if the recovery is > 180%.

NOTE:
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard
2. Flag (J) or reject (R) only the final sample results on Form I's when **Sample raw data** are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

A.1.14 Initial and Continuing Calibration Blanks - Form III

		YES	NO	N/A
A.1.14.1	Present and complete for all the instruments used for the metals and cyanide analyses?	[___]	___	___
	Was an initial Calibration Blank analyzed after ICV?	[___]	___	___
	Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?	[___]	___	___
	Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by			

		YES	NO	N/A
	using MDLs from direct analysis(Preparation Method "NP1")? (Check Form III against the raw data)	[___]	___	___

ACTION:
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

≥ MDL but ≤ CRQL

> CRQL

A.1.14.2.1	When MDL < CRQL, is any Calib. Blank value ≥ MDL but ≤ CRQL?	___	[___]	___

ACTION:
If yes, change sample results ≥ MDL but ≤ CRQL to the CRQL with a "U". Do not qualify non-detects.

A.1.14.2.2	When MDL < CRQL, is any Calib. Blank value > CRQL?	___	[___]	___

ACTION:
If yes, reject (R) and red line the associated sample results > CRQL but <ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results ≥ MDL but ≤ the CRQL to CRQL with a "U".

A.1.14.2.3	Is any Calibration Blank value below the negative CRQL?	___	[___]	___

ACTION:
If yes, flag (J) as estimated all associated sample results ≥ CRQL but <10xCRQL.

NOTE:

1. For ICB that does not meet the technical QC Criteria, apply the action to all samples

	YES	NO	N/A

reported from the analytical run.

2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.,

A.1.15 **Preparation Blank - FORM III**

NOTE:The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

Each Sample Delivery Group (SDG)?	[___]	___	___
Each batch of the SDG samples digested/distilled?	[___]	___	___
Each matrix type?	[___]	___	___
All instruments used for metals and cyanide analyses?	[___]	___	___

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated(J),but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

$\geq$ MDL but $\leq$ CRQL, and

> CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value $\geq$ MDL but $\leq$ CRQL? ___ [___] ___

ACTION:
If yes, change sample result $\geq$ MDL

		YES	NO	N/A
	but ≤ CRQL to CRQL with a "U".			
A.1.15.2.2	When the MDL ≤ CRQL, is any Preparation Blank value greater than its CRQL?	___	[___]	___
	If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples?	___	[___]	___
	If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value?	___	[___]	___

ACTION:
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep.Blank value. Flag as "J" detects > Prep. Blank value but <10xPrep.Blank. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

NOTE:
Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.

A.1.15.2.3	Is the Prep. Blank concentration below the negative CRQL?	___	[___]	___

ACTION:
If yes, flag (J) all associated sample results less than 10xCRQL. Qualify non-detects as estimated (UJ).

A.1.15.2.4	When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?	___	[___]	___

ACTION:

		YES	NO	N/A
	If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.			

A.1.16 **ICP-AES/ICP-MS Interference Check Sample (ICS)- Form IV**

NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1	Present and complete?	[___]	___	___
	Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples?	[___]	___	___
	Was ICS analyzed at the beginning of the ICP-MS analytical run?	[___]	___	___

ACTION:
If no, flag as estimated (J) all sample results.

A.1.16.2 **ICP-AES Method**

A.1.16.2.1	**ICSA Solution:** For ICP-AES, are the ICSA "Found" analyte values within the control limits ± of CRQL of the true/established mean value?	[___]	___	___
	If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV?	[___]	___	___

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results ≥MDL

		YES	NO	N/A
	for which the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag non-detects as "UJ" and detects as "J".			
A.1.16.2.3	**ICSAB Solution** For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?	[___]	___	___
	If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV?	[___]	___	___
	ACTION: If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run: Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as "J" and non-detects as "UJ". Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.			
A.1.16.3	**ICP-MS Method**			
A.1.16.3.1	**ICSA Solution:** For ICP-MS, are the ICSA "Found" analyte values within the control limits of ±CRQL of the true/established mean value?	[___]	___	___
	ACTION: If no, apply the following action to all samples reported from the analytical run: Flag (J) as estimated only sample results ≥ MDL if the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag the associated sample detects as "J" and non-detects as "UJ".			

		YES	NO	N/A
A.1.16.3.3	**ICSAB Solution** For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?	[___]	___	___

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%,reject (R) and red-line only detects (≥ MDL).

A.1.17 **Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A**
Note:Not required for Ca,Mg,K,and Na(both matrices);Al and Fe (soil only)

A.1.17.1	Was Matrix Spike analysis performed:			
	For each matrix type?	[___]	___	___
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each concentration range (i.e.,low, med., high)?	[___]	___	___
	For each analytical Method (ICP-AES,ICP-MS, Hg, CN)used?	[___]	___	___
	Was a spiked sample prepared and analyzed with the SDG samples?	[___]	___	___

ACTION:
If no for any of the above, flag as estimated(J)all the positive data for which a spiked sample was not analyzed.

NOTE:
If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.

		YES	NO	N/A
A.1.17.2	Was a field blank or PE sample used for the spiked sample analysis?	___	[___]	___
	ACTION: If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.			
A.1.17.3	Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.			
	Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?	[___]	___	___
	NOTE: Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.			
	Are results outside the control limits (75-125%)flagged with Lab Qualifier "N" on Form I's and Form VA?	[___]	___	___
	ACTION: If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.			
A.1.17.4	**Aqueous**			
	Are any spike recoveries:			
	(a) less than 30%?	___	[___]	___
	(b) between 30-74%?	___	[___]	___
	(c) between 126-150%?	___	[___]	___
	(d) greater than 150%?	___	[___]	___
	ACTION: If the matrix spike recovery is less than 30%,reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as “J” and non-detects			

		YES	NO	N/A
	as "UJ". If between 126-150%, flag (J) all data ≥ MDL as "J". If greater than 150%, reject (R) and red-line all associated data ≥ MDL.			
	(**NOTE:**Replace "N" with "J", "R" as appropriate.)			
A.1.17.5	**Soil/Sediment**			
	Are any spike recoveries:			
	(a) less than 10%?	___	[___]	___
	(b) between 10-74%?	___	[___]	___
	(c) between 126-200%?	___	[___]	___
	(d) greater than 200%?	___	[___]	___
	ACTION: If yes for any of the above, proceed as follows:			
	If the matrix spike recovery is less than 10%,reject (R) and red-line all associated data (detects & non-detects); if between 10-74%,qualify all associated data ≥ MDL as "J" and non-detects as "UJ"; if between 126-200%, flag (J) all associated data ≥ MDL as "J" If greater than 200%, reject (R) and red-line all associated data ≥ MDL. (**NOTE:**Replace "N" with "J" or "R" as appropriate.)			
A.1.18	**Lab Duplicates) - Form VI**			
A.1.18.1	Was the lab duplicate analysis performed:			
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each matrix type?	[___]	___	___
	For each concentration range (low or med.)?	[___]	___	___
	For each analytical Method (ICP-AES/ICP-MS,Hg,CN)Used?	[___]	___	___
	Was a lab duplicate prepared and analyzed with the SDG samples?	[___]	___	___

		YES	NO	N/A
	ACTION: If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.			
	NOTE: If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.			
A.1.18.2	Was a Field Blank or PE sample used for the Lab Duplicate analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.			
A.1.18.3	Circle on each Form VI all values that are:			
	RPD > 20%, or			
	Absolute Difference > CRQL			
	Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)?	[___]	___	___
	If no, are all results outside the control limits flagged with an "*" (Lab Qualifier)on Form VI and on all Form I's?	[___]	___	___
	ACTION: If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.			
	NOTE: The laboratory is not required to report on Form VI the RPD when both values are non-detects.			
A.1.18.4	**Aqueous**			
A.1.18.4.1	When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),			

	YES	NO	N/A
is any RPD > 20% but < 100%?	___	[___]	___
is any RPD ≥ 100%?	___	[___]	___

ACTION:
If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

(**NOTE:**Replace **"*"** with "J" or "R" as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?	___	[___]	___
> ± 2xCRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.
NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?	___	[___]	___
is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample

		YES	NO	N/A
	data ≥ CRQL. If the RPD is ≥ 120%, reject (R)and red-line the associated sample data ≥ CRQL.			
A.1.18.5.2	When the sample and/or duplicate value <5xCRQL(substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:			
	> ± 2 x CRQL?	___	[___]	___
	> ± 4 x CRQL	___	[___]	___

ACTION:
If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.19 Field Duplicates

Aqueous Field Duplicates

A.1.19.1	Was an aqueous Field Duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	____

ACTION:
If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I's. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the

		YES	NO	N/A

QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD $\geq$ 20% or

Difference > $\pm$ CRQL

When sample and duplicate values are both $\geq$5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD $\geq$ 20%?	___	[___]	___
is any RPD $\geq$ 100%?	___	[___]	___

ACTION:
If the RPD is >20% but < 100%, flag (J) only the associated sample and its Field Duplicate results $\geq$ CRQL. If the RPD is $\geq$ 100%, reject(R) and red-line only the associated sample and its Field Duplicate result $\geq$ CRQL.

A.1.19.3 When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate:

> $\pm$ CRQL?	___	[___]	___
> $\pm$ 2 x CRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag detects $\geq$ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the difference is > 2xCRQL,reject (R) and red-line non-detects

		YES	NO	N/A
	and results ≥ MDL but <5xCRQL of the sample and its Field Duplicate.			

Soil/Sediment Field Duplicates

A.1.19.4	Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	___

ACTION:
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate)is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5	Circle on each Appendix A.4 all values that have: RPD ≥ 35%, or Difference > ± 2xCRQL When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),			
	is any RPD ≥ 35% but < 120%?	___	[___]	___
	is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% but < 120%,

		YES	NO	N/A
	flag only the associated sample and its Field Duplicate results ≥ CRQL as "J". If the RPD is ≥ 120%, reject (R) and red-line only the sample and its Field Duplicate results ≥ CRQL.			
A.1.19.6	When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and Field Duplicate:			
	> ± 2 x CRQL?	___	[___]	___
	> ± 4 x CRQL?	___	[___]	___
	ACTION: If the absolute difference is > 2xCRQL, flag Sample and its Field Duplicate resuts ≥ MDL but <5xCRQL as "J" and non-detects as "UJ". If the difference is >4xCRQL, reject(R) and red-line non-detects and detects ≥ MDL but <5xCRQL of the sample and its Field Duplicate.			
A.1.20	**Laboratory Control Sample (LCS)- Form VII**			
A.1.20.1	Was one LCS prepared and analyzed for:			
	Each SDG?	[___]	___	___
	Each matrix type?	[___]	___	___
	Each batch samples digested/distilled?	[___]	___	___
	For each Method(ICP-AES,ICP-MS,Hg,CN) used?	[___]	___	___
	Was an LCS prepared and analyzed with the samples?	[___]	___	___
	ACTION: If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.			
	NOTE: If only one LCS was analyzed for			

	YES	NO	N/A

more than 20 samples, then the first 20 samples analyzed are not flagged(J), but all additional samples must be qualified (J).

A.1.20.2 **Aqueous LCS**

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

NOTE: 1.Use digested ICV as LCS for aqueous mercury
2.Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?	___	[___]	___
Between 50% and 79%?	___	[___]	___
Between 121% and 150%?	___	[___]	___
Greater than 150%?	___	[___]	___

ACTION:
If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as “J” all non-detects as “UJ”. if the LCS recovery is between 121-150%, flag only detects as “J”. if the recovery is greater than 150%, reject (R) and red-line all detects.

A.1.20.3 **Solid LCS**

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII?	___	[___]	___

ACTION:

		YES	NO	N/A
	If yes, flag (J) all the associated detects ≥ MDL as estimated (J).			
	Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?	___	[___]	___
	ACTION: If yes, flag detects as "J" and non-dectes as "UJ".			
A.1.21	**ICP-AES/ICP-MS Serial Dilution - Form VIII** **NOTE**:Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.			
A.1.21.1	Was a Serial Dilution analysis performed:			
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each matrix type?	[___]	___	___
	For each concentration range (low or med.)?	[___]	___	___
	Was a Serial Dilution sample analyzed with the SDG samples?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.			
A.1.21.2	Was a Field Blank or PE sample used for the Serial Dilution Analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples			
A.1.21.3	Circle on Form VIII the Percent Differences (%D) between sample results and its dilution results that are outside the control limits ± 10%			

		YES	NO	N/A
	when initial concentrations ≥ 50 x MDLs.			
	Are results outside the control limits flagged with an "E"(Lab Qualifier) on Form VIII and all Form I's?	[___]	___	___
	ACTION: If no, write in the Contract-Problem/ Non-Compliance Section of the Data Review Narrative.			
A.1.21.4	Are any %D values:			
	> 10%?	___	[___]	___
	≥ 100%?	___	[___]	___
	ACTION: If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose **raw data** ≥ MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with **raw data** ≥ MDL.			
	(**NOTE:**Replace "E" with "J" or "R" as appropriate.)			
A.1.22	**Total/Dissolved or Inorganic/Total Analytes**			
A.1.22.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?	___	[___]	___
	Were any analyses performed for inorganic as well as total analytes on the same sample(s)?	___	[___]	___
	ACTION: If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic)and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:			
	(1) The dissolved(or inorganic)concentration is greater than total concentration, and (2) greater than or equal to 5xMDL.			
A.1.22.2	Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?	___	[___]	___

		YES	NO	N/A
A.1.22.3	Is any dissolved(or inorganic) concentration greater than its total concentration by more than 50%?	___	[___]	___

ACTION:
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 **Field Blank - Form I**
NOTE: **Designate "Field Blank" as such on Form I**

A.1.23.1	Was a Field/Rinsate Bank collected and analyzed with the SDG samples?	[___]	___	___
	If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL(or 2xMDL when MDL>CRQL)?	___	[___]	___
	If yes, circle the Field Blank value on Form I that is greater than the CRQL,(or 2 x MDL when MDL > CRQL).			
	Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?	___	[___]	___
	If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?	[___]	___	___

ACTION:
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results)greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result $\geq$ MDL but $\leq$ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the

		YES	NO	N/A
	Prep.Blank value, do not qualify the sample results due to the Field Blank criteria.			

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24 **Verification of Instrumental Parameters - Form IX, XA, XB, XI**

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX-Annually)?	[___]	___	___
ICP-AES Interelement Correction Factors (Form XA & XB -Quarterly)?	[___]	___	___
ICP-AES & ICP-MS Linear Ranges (Form XI-Quarterly)?	[___]	___	___

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 **Method Detection Limits - Form IX**

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes?	[___]	___	___
All the instruments used?	[___]	___	___
Digested and undigested samples and Calib.Blanks?	[___]	___	___
ICP-AES and ICP-MS when both instruments are used for the same analyte?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/ Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ½ CRQL.

		YES	NO	N/A
A.1.24.2.2	Is MDL greater than the CRQL for any analyte?	___	[___]	___
	If yes,is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?	[___]	___	___

ACTION:
If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.

A.1.24.3 **Linear Ranges - Form XI**

A.1.24.3.1	Was any sample result higher than the high linear range for ICP-AES or ICP-MS?	___	[___]	___
	Was any sample result higher than the highest calibration standard for mercury or cyanide?	___	[___]	___
	If yes for any of the above, was the sample diluted to obtain the result reported on Form I?	[___]	___	___

ACTION:
If no, flag (J) as estimated the affected detects ($\geq$ MDL) reported on Form I.

A.1.25 **ICP-MS Tune Analysis - Form XIV**

A.1.25.1	Was the ICP-MS instrument tuned prior to calibration?	[___]	___	___

ACTION:
If no, reject (R) and red-line all sample data for which tuning was not performed.

A.1.25.2	Was the tuning solution analyzed or scanned at least five times consecutively?	[___]	___	___
	Were all the required isotopes spanning the analytical range present in the tuning solution?	[___]	___	___
	Was the mass resolution within			

		YES	NO	N/A
0.1 amu for each isotope in the tuning solution?		[___]	___	___
	Was %RSD less than 5% for each isotope of each analyte in the tuning solution?	[___]	___	___
	ACTION: If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated "J", and all non-detects associated with that Tune as "UJ".			
A.1.26	**ICP-MS Internal Standards - Form XV**			
A.1.26.1	Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)?	[___]	___	___
	Were all the target analyte masses bracketed by the masses of the five internal standards?	[___]	___	___
	ACTION: If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.			
A.1.26.2	Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank?	[___]	___	___
	If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed?	[___]	___	___
	Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)?	[___]	___	___
	ACTION: If no for any of the above, flag detects as "J" and non-detects "UJ" of all the analytes with atomic masses between the atomic mass of the internal standard lighter			

than the affected internal standard, and the atomic mass of the internal standard heavier than the affected internal standard.

A.1.27 **Percent Solids of Sediments**

A.1.27.1 Are percent solids in sediment(s):

< 50%? ___ [___] ___

ACTION:
If yes, qualify as estimated (J) all detects and non-detects of a sample that has percent solids less than 50%(i.e.,moisture content greater than 50%).

NOTE:
Flag(J) only the sample results that were not previously flagged due to other QC criteria.

Inorganic Data Review Narrative

Case# _____ **Site:** _____ **Matrix: Soil** ____

SDG# _____ **Lab:** _____ **Water** ____

Sampling Team: _____ **Reviewer:** _____ **Other** _____

A.2.1 **Data Validation Flags**:
The following flags may have been applied in red by the data validator and must be considered by the data user.

J - This flag indicates the result qualified as **estimated**

R and Red-Line - A red-line drawn through a sample result indicates **unusable** value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.

U - This data validation qualifier is applied to sample results ≥ MDL when associated blank is contaminated

Fully Usable Data - The results that do not carry "J" or "red-line" are fully **usable**.

A.2.2 **Laboratory Qualifiers**:
The CLP laboratory applies a contractual qualifier on all

Form I'S and the QC Form when a QC analysis is outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets. These qualifiers and their meanings are as follows:

N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E: This qualifier indicates the the presence of interference, and is applied when the ICP serial dilution is outside the control limits.

*****: This qualifier indicate the lack of precision , and is pplied on Fom I'S and Form VI when the Lab Duplicate analysis is outside the control limits.

U: This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analyte is indicated by the Contract Required Quantitation Limit (CRQL) of that analyte suffixed with "U".

J: This is also a concentration qualifier that laboratory applies to a positive result below the CRQL.

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

A.2.3.1 **Data Case Description**:

A.2.3.2 **CSF Audit**:

A.2.3.3 **Technical Review:**

A.2.3.4 **Contract-Problem/Non-Compliance:**

HWSS Reviewer: _____ Date:_____
Signature

Contractor Reviewer: _____ Date:_____
Signature

Verified by: _____ Date:_____
Signature

CASE #

SDG #

Contract Laboratory Program
REGION II/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: _____

ESAT Reviewer/Date: _____

Type of Analysis: ___**Inorganic**_____

Laboratory Name: _____

Lab Contact: _____

Call Initiated By: ___**Laboratory** _X_**Region II**

Inquiry made in reference to data for the following sample number(s):

Summary of Questions/Issues Discussed:

Summary of Resolution:

_____

Signature **Date:** _____

FIELD DUPLICATES

Sample No. **Field Duplicate No.** **Sample Matrix:**

Lab Code: **Case No. :** **SDG No.:**

% Solids Sample: **% Solids Duplicate:**

Concentration Units (ug/l or mg/kg dry weight):

	Action Limit	Sample Concentration	C	Duplicate Concentration	C	RPD	Difference	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									

					YES	NO	N/A		
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Total/Dissolved Concentrations

Lab Code **Case No.** **SDG No.** **Sample Matrix: Water**

Concentration: ug/L

ANALYTE	TOTAL	C	DISSOLVED	C	DIFFERENCE	Q	M
ALUMINUM							
ARSENIC							
BARIUM							
BERYLLIUM							
CADMIUM							
CALCIUM							
CHROMIUM							
COBALT							
COPPER							
IRON							
LEAD							
MAGNESIUM							
MAGNESE							
MERCURY							
NICKEL							

POTASSIUM							
SELENIUM							
SILVER							
SODIUM							
THALLIUM							
VANADIUM							
ZINC							
CYANIDE							

CONTRACT LABORATORY PROGRAM
CLP RAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SECTION A (TO BE COMPLETED BY REGIONAL SENDING OFFICIAL)

Initiated By:

Name, Affiliation, Phone Number

_____

Case Number:_____
! OLM
! OLC
! ILM

Details of Re-Analysis Request:

! Laboratory Name /Contract Number: _____

! Affected Sample Number(s) and Fraction(s): _____

! Reason for Re-Analysis: _____

! Contract Statement of Work Citation*: _____
_____

! Comments:_____

_____

! * PROVIDE SOW CITATION THAT SUPPORTS THIS REQUEST

RE-ANALYSIS Billable ( ) Not Billable ( )

! Approved By: _____ Date: _____

Authorized Regional Sending CLP PO Signature

SECTION B (TO BE COMPLETED BY SMO)

_____

Name of SMO Contact _____ Date:_____

Date of Laboratory Notification (Verbal): _____

Re-analysis Start Date: _____ Data Due Date: _____

_____

Return completed form to:

Sample Management Office (SMO)

Distribtion: (1) CLP PO Copy (2) Regional Sending Official Copy (3) SMO File Copy (4) Laboratory Copy
Final 9/3/99

CLP DATA ASSESSMENT SUMMARY FORM (INORGANICS)

Type of Review: _____ Date:_____ Case# _____SDG#___

Site: _____ Lab Name: _____

Reviewer's Initials:_____ Number of Samples:_____

Analytes Rejected (R) Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

Analytes Flagged (J) as Estimated Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

Polychlorinated Dibenzodioxins/ Polychlorinated Dibenzofurans SW-846 Method 8280 DATA Validation

Prepared by: George Karras Date: 12/8/06
George Karras, Chemist HWSS

Peer Reviewed by: Russell Arnone Date: 12-08-06
Russell Arnone, Chemist HWSS

Concurred by: Linda Mauel Date: 12/8/06
Linda Mauel, Chief HWSS

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief, HWSB

Annual Review

Reviewed by:_____ Date:_____

Reviewed by:_____ Date:_____

1.0 Introduction

1.1 The attached Standard Operating Procedure (SOP) is applicable to polychlorinated dibenzodioxin and polychlorinated dibenzofuran (PCDD/PCDF) data. Its scope is to facilitate the data validation process of the data reported by the contracting laboratory and also to ensure that the data is being reviewed in a uniform manner.

1.2 The SOP is based upon the quality control and quality assurance requirements specified in the analytical method PCDD/PCDF Protocol, Statement Of Work 9/91 (DFLM01.1) and its ensuing revision.

2.0 Responsibilities

2.1 The reviewer must be knowledgeable of the analytical method and its QC Criteria.

2.2 The reviewer must complete and/or file the following:

2.2.1 Data Assessment Checklist - The data reviewer must read each item carefully and must check yes if there is compliance, no if there is non compliance and N/A if the question is not applicable to the data.

2.2.2 Data Assessment Narrative - The data reviewer must present professional judgement and must express concerns and comments on the validity of the overall data package. The reviewer must explain the reasons for rejecting and/or qualifying the data.

2.2.3 Rejection Summary Form - The reviewer must submit the completed form using a ratio format. The numerator indicates the number of dioxins/furans data rejected; the denominator indicates the number of dioxins/furans fractions containing rejected compounds.

2.2.4 Organic Regional Data Assessment Summary - The data reviewer is also required to submit the completed Organic Regional Data Assessment Form.

2.2.5 Telephone Record Log - All phone conversations must be initiated by the technical project officer through SMO. If a phone call has been made, the reviewer must transcribe the conversation. After the data review has been completed, the white copy of the telephone log is mailed to the laboratory and the pink copy to SMO. The yellow copy is filed in the appropriate folder.

A photocopy of the Telephone Record Log is attached to the Data Assessment Narrative.

2.2.6 Forwarded Paperwork - Upon completion of the review the following are to be forwarded to the Regional Sample Control Center (RSCC):

a. data package
b. completed data assessment checklist and narrative (original)

The reviewer will forward one copy of the completed Data Assessment and one copy of the Organic Regional Data Assessment to the appropriate Regional TPO.

2.2.7 Filed Paperwork - The following are to be submitted to the Monitoring Management Branch (MMB) files:

a. a photocopy of the Data Assessment Narrative
b. a photocopy of the Regional Data Assessment Summary
c. Telephone record Log (copy)
d. Rejection Summary Form

2.3 Rejection of Data - All values determined to be unacceptable on the Organic Analysis Data Sheet (Form I) must be flagged with an "R". The qualifier R means that due to significant QA/QC problems the analysis is invalid and it provides no information as to whether the compound is present or not. Once the data are flagged with R any further review or consideration is unnecessary.

- The qualifier "J" is used to indicate that due to QA/QC problems the results are considered to be estimated.

- The qualifier "NJ" indicates that there is presumptive evidence for the presence of the compound at an estimated value.

- The data reviewer must explain in the data assessment narrative why the data was qualified. He or she must also indicate all items of contract non-compliance.

- When 2,3,7,8- substituted TCDD, TCDF, PnCDD and PnCDF data are rejected (flagged "R") or qualified "J" the project officer must be notified promptly. If holding times have not been exceeded reanalysis of the affected samples may be requested.

- All qualifications and corrections to reviewed data must be made in red pencil.

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____

LAB: _____

SITE: _____

l.0 Data Completeness and Deliverables

1.1 Are the Traffic Report Forms present for all samples?

1.2 Is the Narrative or Cover letter present? [___] ___ ___
___ ___

1.3 Are the Case Number and/or SAS numbers contained in the case narrative? [___] ___ ___
___ ___

1.4 Do the Traffic Reports or Lab Case Narrative indicate problems with sample receipt, sample condition, analytical problems, or other comments affecting the quality of the data?

ACTION: Use professional judgement to evaluate the effect of the noted problems on the quality of the data.

2.0 Reporting Requirements and Deliverables

2.1 All deliverables must be clearly labeled with the SMO number and the associated sample/traffic number. Missing or illegible or incorrectly labeled items must be identified. The contractor must immediately be contacted and requested to submit the missing or incorrect items.

2.2 Are the following forms present?

a. Sample Data Summary (Form I PCDD-1)

b. PCDD/PCDF Toxicity Equivalency Factor (Form I, PCDD-2)

__ ___

c. Second Column Confirmation Summary (Form I, PCDD-3)
[___] ___ ___

YES NO N/A

d. Total Homologue Concentration Summary (Form II PCDD)
_ ___

e. PCDD/PCDF Spiked Sample Summary (Form III PCDD-1)
[___] ___ ___

f. PCDD/PCDF Duplicate Sample Summary (Form III PCDD-2)

g. PCDD/PCDF Method Blank Summary (Form IV-PCDD)

h. PCDD/PCDF Window Defining Mix Summary (Form V-PCDD-1)
___ ___

i. Chromatographic Resolution Summary (Form V PCDD-2)

j. PCDD/PCDF Analytical Sequence Summary (Form V PCDD-3)

k. Initial Calibration (Form VI, PCDD-1, PCDD-2)

l. Continuing Calibration (Form VII,PCDD-1, Form VII,PDD-2)

2.3 GC/MS Displays

a. Standard and sample SIM chromatograms. SIM and TIC
_ chromatograms must list date and time of analysis; the file name; sample number; and instrument I.D. number.

b. Percent peak resolution valley

c. PCDD/PCDF window defining mix raw data

d. SIM mass chromatograms must display quantitation ion, confirmation ion, daughter ion (M-COC1) and polychlorinated diphenylether ion where applicable.

e. Integrated area and peak height must be listed for all peaks 2.5 times above background.

f. All peaks must show retention time at the maximum height.

2.4 Chain of Custody Records and in-house Laboratory Control Documents

a. EPA Chain of Custody Records

b. SMO Sample Shipment Records

YES NO N/A

c. Sample log-in sheets

d. GC/MS Standard and Sample Run Log in chronological order

___ ___

e. Sample Extraction Log

___ ___

2.5 The Sample Package Data must be paginated.

ACTION: If deliverables are missing call the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the reviewers narrative.

3.0 Holding Times

3.1 Have any holding times been exceeded?

a. For aqueous samples 30 days from sample collection to extraction.

b. For soil/sediment samples 30 days from sample collection to extraction.

c. For all samples 40 days from time of extraction to time of analysis.

ACTION: If holding times are exceeded, flag all data as estimated ("J"). Holding time criteria do not apply to PE samples.

4.0 Instrument Performance

4.1 Mass Calibration - Mass calibration of the MS is recommended prior to analyzing calibration solutions, blanks, samples, and QC samples. The lab is not required to submit mass calibration data.

4.2 Window Defining Mixture/Column Performance Mixture

4.2.1 The Window Defining Mixture and the Column Performance Mixture must be analyzed prior to the initial calibration. It must also be analyzed whenever the retention time of either recovery standard in any analysis varies by more than 10 seconds from the most recent

continuing calibration standard.

4.2.2 The window defining mix must contain the first and the last isomers of each homologue PCDD/PCDF, (the internal and recovery standards are optional).

4.2.3 All peaks must be labeled and identified on the SICPs.

ACTION: 1. If the window defining mix was not analyzed at the required frequency use professional judgement to determine the effect on the quality of the data.

4.3 Chromatographic Resolution

4.3.1 For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of the CC3 Standard Solution during the initial and continuing calibration.

4.3.2 For analyses on a SP-2331 (or equivalent) GC column the chromatographic resolution is evaluated before the analysis of initial calibration by the analysis of the column performance mixture. This commercially available solution contains the 2378-TCDD and the isomers eluting immediately prior and after the 2378-TCDD on SP-2331 or equivalent.

4.3.3 For SP-2331 or equivalent, the peak separation between the unlabeled 2378-TCDD and the peaks of 1468-TCDD and the 1237/1238-TCDD isomer pair shall be resolved with a valley of < 25 percent.

Valley = (x/y) x (100)

Y = The peak height of 2,3,7,8-TCDD isomer or any TCDD isomer

X = The distance from the baseline to the bottom of the valley between the adjacent peaks.

ACTION: If the percent valley criteria are not met, qualify all positive data J. Do not qualify non-detects.

YES NO N/A

5.0 Initial 5-Point Calibration - The initial calibration standard solutions (CC1-CC5) must be analyzed prior to any sample analysis. They do not have to be analyzed daily provided the continuing calibration standard met all criteria. However, initial calibration should be analyzed at least once every week and/or whenever the continuing calibration standard does not meet all criteria.

The calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze the window defining mix. The CC3 solution must contain the supplemental calibration solution (see analytical method - Table 3).

5.1 The following MS/DS conditions must be used:

5.1.1 Scanning time was < 1 second. __

5.1.2 SIM data were acquired for each of the ions listed in Table 5 including interfering ions (see analytical method) ___

5.2 The following GC criteria must be met:

5.2.1 The chromatographic resolution between the $^{13}C_{12}$2378-TCDD and $^{13}C_{12}$1234-TCDD isomers must be resolved with a valley of < 25 percent method.

5.2.2 In the CC3 solution, the chromatographic peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD shall be resolved with a valley of $\leq$ 50 percent.

5.2.3 For all calibration solutions the retention times of the isomers must fall within the retention time windows established by the window defining mix. In addition the absolute retention time of recovery standards, $^{13}C_{12}$1234-TCDD and $^{13}C_{12}$-123789HxCDD shall not change by more than 10 seconds between the initial CC3 analysis and the analysis of any other standard.

5.2.4 The three SIM ions for each homolog must maximize simultaneously and within 3 seconds of the corresponding labeled isomer ions.

5.2.5 The relative ion abundance criteria for PCDDs/PCDFs listed in table 6 (see analytical method) must be met.

5.2.6 The relative ion abundance criteria for the labeled internal and recovery standards listed in table 6 must

be met.

5.2.7 For all calibration solutions, including CC3, the signal to noise ration (S/N) for all ions of the unlabeled PCDDs/PCDFs must be greater than 2.5. _

5.2.8 For the internal and recovery standards, the signal to noise ratio for all ions must be greater than 10.

5.2.9 The percent relative standard deviation (% RSD) of the five RRFs (CC1-CC5) for the unlabeled PCDDs/PCDFs and the internal standards must not be greater than 15 percent.

ACTION:

1. If the 25% percent valley for TCDD and 50% valley for HxCDD requirement is not met, quality positive data J. Do not qualify non-detects. The tetra, pentas and hexas (dioxins and furans) are affected. Heptas and Octas are not affected.

2. If the %RSD for each isomer exceeds 20% percent, flag the associated sample positive results for that specific isomer as estimated ("J"). No effect on the non-detect data.

3. If the ion abundance ratio for an analyte is outside the limits flag the results for that analyte R (reject).

4. If the ion abundance ratio for an internal or recovery standard falls outside the QC limits flag the associated positive hits with J. No effect on the non-detects.

5. If the signal to noise ratio (S/N) is below control limits, use professional judgement to determine quality of the data.

6. If the selected monitoring ions specified in Table 5 were not used for data acquisition, the lab must be asked for an explanation. If an incorrect ion was used, reject all the associated data.

5.2.10 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled PCDDs/PCDFs and internal standards were used.

In addition verify that the appropriate internal standard was used for each isomer.

To recalculate the response factor use the equation:

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

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

Where:

A_n^1 and A_n^2 = integrated areas of the two quantitation ions of isomer of interest (Table 5).

A_{is}^1 and A_{is}^2 = integrated areas of the two quantitation ions of the appropriate internal standard (Table 5).

A_{rs}^1 and A_{rs}^2 = integrated areas of the two quantitation ions of the appropriate recovery standard (Table 5).

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected (ng)

Q_{is} = quantity of the appropriate internal standard injected (ng)

Q_{rs} = quantity of the appropriate recovery standard injected (ng)

6.0 Continuing Calibration - The continuing calibration consists of two parts: evaluation of the chromatographic resolution and verification of the RRF values to be used for quantitation.

6.l Chromatographic Resolution - At the beginning of each 12 hour period the chromatographic resolution is verified in a similiar fashion as in the initial calibration: through the analysis of CC3 Standard Solution on the DB-5 (or equivalent) column or through the analysis of the column performance solution on the SP2331 (or equivalent) column.

YES NO N/A

6.1.2 Was the continuing calibration and the column performance solution (when applicable) run at the required frequency?

6.1.3 Was the chromatographic peak separation on DB-5 (or equivalent) column between $^{13}C_{12}$-2378TCDD and $^{13}C_{12}$ 1234-TCDD isomers resolved with a valley of <25 percent?

6.1.4 Was the chromatographic peak separation on the SP-2331 (or equivalent) column between the unlabeled 2378-TCDD and the adjacent TCDD isomers resolved with a valley of <25 percent?

___ ___
In addition, was the chromatographic peak separation between the 123478-HxCDD and the 123678-HxCDD in the CC3 solution resolved with a valley of <50 percent?

___ ___

ACTION 1. If the continuing calibration standard was not analyzed at the required frequency, reject all the data. Contact TPO to initiate reanalysis.

2. If the 25 percent valley and 50 percent valley criteria are not met qualify all positive data with J. Do not qualify non-detects. Note: The tetras, pentas and hexas (dioxins and furans) are affected. Heptas and octas are not affected. If the percent valley is >75 percent and 2378-TCDD is non-detect but 1234-TCDD or an adjacent TCDD isomer is present, the data is questionable. The sample must be reanalyzed. Contact TPO.
If the valley criteria for HxCDD are not met but the valley criteria for TCDD are met or vice-versa, use professional judgement to determine which data must be qualified.

6.2 Continuing Calibration (CC3).
The CC3 shall be analyzed at the beginning of a 12 hour period.

6.2.1 The following MS/DS conditions were used:

6.2.2 Scanning time was < 1 second.

___ ___

6.2.2.1 SIM data were acquired for each of the ions listed in Table 5 including diphenylether interfering ions (see analytical method).

6.2.3 The following GC criteria must be met:

YES NO N/A

6.2.3.1 For all calibration solutions the retention time of the isomers must fall within the retention time windows established by the window defining mix.

___ ___

6.2.3.2 The absolute retention time of the recovery standards $^{13}C_{12}$1234-TCDD and $^{13}C_{12}$123679-HxCDD shall not change by more than 10 seconds between the initial CC3 and ending CC1 standard analyses.

___ ___

6.2.3.3 The three SIM ions for each homolog must maximize simultaneously (± 2 sec) and within 3 seconds of the corresponding ions of the labeled isomers.

___ ___

6.2.3.4 For the CC3 standard solution, the signal to noise ratio (S/N) for the unlabeled PCDD/PCDF ion shall be greater than 2.5.

6.2.3.5 For the internal standards and the recovery standards, the signal to noise ratio (S/N) shall be greater than 10.

6.2.3.6 The relative ion abundance criteria (Table 6 - analytical method) for all PCDD/PCDF shall be met.

6.2.3.7 The relative ion abundance criteria for all internal and recovery standards (Table 6 - analytical method) must be met.

6.2.3.8 The measured RRF of each analyte and internal standard in the CC3 solution must be within ± 30 percent of the mean RRF established during the initial calibration and within ± 30 percent of the single point RRFs obtained during initial calibration for the supplemental calibration standards.

Spot check response factor calculations and ion ratios. Verify that the appropriate quantitation ions for the unlabeled PCDD/PCDFs and internal standards were used.

6.2.3.9 Was the same internal standard used to calculate RRF for each PCDD/PCDF homolog in the initial calibration?

ACTION: 1. If any of the requirements listed in sections 6.2.2, 6.2.2.1, 6.2.3.1, 6.2.3.2,

YES NO N/A

and 6.2.3.9 are not met, use professional judgement to determine the validity of the data.

2. If any requirements listed in sections 6.2.3.3, 6.2.3.4, 6.2.3.5, 6.2.3.6, and 6.2.3.7 are not met reject all data (flag R) directly affected by each specific problem.

3. When the %D of the RRF is in between 30% and 50% all the data for the outlier congeners are flagged J.
Data with %D above 50% are rejected (R).

6.2.3.10 To recalculate RRFs for the unlabeled target analytes, and the RRFs for the five labeled internal standards, use the following equations:

$$RRFn = \frac{(An^1 + An^2) \times Qis}{(Ais^1 + Ais^2) \times Qn}$$

$$RRFis = \frac{(Ais^1 + Ais^2) \times Qrs}{(Ars^1 + Ars^2) \times Qis}$$

An^1, An^2, Ais^1, Ais^2, Ars^1, Ars^2, Qn, Qis and Qrs are defined in Section 5.2.10.

To calculate percent difference use the following equation:

$$\% \text{ Difference} = \frac{(RRFi - RRFc) \times 100}{RRFi}$$

Where:

RRFi = Relative response factor established during initial calibration
RRFc = Relative response factor established during ontinuing calibration

6.3 Instrument Sensitivity - In order to demonstrate that the GC/MS system has retained adequate sensitivity, during the course of sample analysis, the lowest of the initial calibration standards (CC1) is analyzed at the end of each 12-hour period.

6.3.1 Did all analytes in the CC1 solution meet ion abundance criteria?

YES NO N/A

6.3.2 Did the retention time of the two recovery standards $^{13}C_{12}$1234-TCDD and $^{13}C_{12}$123678HxCDD change by more than +/- 10 seconds?

6.3.3 For CC1 was the S/N ratio for all unlabeled PCDD/PCDF ions greater than 2.5 and greater than 10 for the labeled internal and recovery standards?

ACTION: If the CC1 standard did not meet criteria examine the samples which were analyzed prior to this standard and use professional judgement to determine if data qualification is necessary.
(See Recovery Standard areas - Section 9.0)

7.0 Sample Data

7.l The following MS/DS conditions were used:

7.1.1 Scanning time was < 1 second. __

7.1.2 SIM data were acquired for each of the ions listed in Table 5 (see analytical method) including diphenylether interfering ions.

7.2 Identification Criteria

7.2.1 For the 2378 substituted isomers found present and for which an isotopically labeled internal standard is present in the sample extract, the absolute retention time at the maximum peak height of the analyte must be within 3 seconds of the retention time of the corresponding labeled standard.

7.2.2 For the 2378 substituted isomer reported present, and for which a labeled standard does not exist, the relative retention time (RRT) of the analyte must be within ±.05 RRT units of the RRT established by the continuing calibration standard (CC3). __

7.2.3 For non-2378 substituted compounds (tetra through hepta) found present, the retention time must be within the window established by the window defining mix for the corresponding homologue.

7.2.4 All specified ions listed in Table 5 (analytical method) for each PCDD/PCDF isomer found present and the labeled standards must be present in the SICP. The three SIM _

YES NO N/A

ions for the analyte, the internal standards and recovery standards must maximize simultaneously (±2 seconds).

7.2.5 The integrated ion current for each characteristic ion of the analyte identified as positive must be at least 2.5 times background noise and must have not saturated the detector.

If the M-[COC1]+ ion does not meet the 2.5 times S/N requirement but meets all other criteria, the reviewer must use professional judgement to determine whether the compound is present.

___ ___ 7.2.6 The integrated ion current for the internal standard characteristic ions must be at least 10 times background noise.

7.2.7 The relative ion abundance criteria (Table 6 - analytical method) for all PCDDs/PCDFs found present must be met.

7.2.8 The relative ion abundance criteria for the internal standards must be met (Table 6 - analytical method).

ACTION:

1. Reject (flag R)all positive data for the analytes which do not meet criteria listed in Sections 7.2.1, 7.2.2, 7.2.3, and 7.2.4.

2. If the criteria listed in section 7.2.5 are not met but all other criteria are met, qualify all positive data of the specific analyte with J.

3. If the requirements listed in section 7.2.6 are not met but all other requirements are met qualify the positive data of the corresponding analytes with "J".

4. If the analytes reported positive do not meet ion abundance criteria, section 7.2.7, reject (R) all positive data for these analytes. Change the positive values to EMPC (estimated maximum possible concentration).

5. If the internal standards and recovery standards do not meet ion abundance criteria

(Table 6 - analytical method) but they meet all other criteria flag all corresponding data with "J".

6. If PCDF is detected but an interfering PCDPE is also detected reject the PCDF data (R). The reported value of PCDF is changed to EMPC.

7. If the lab did not monitor for PCDE's qualify all positive furan data N.

7.2.9 Spot check calculations for positive data and verify that the same internal standards used to calculate RRFs were used to calculate concentration and EMPC. Ensure that the proper PCDDs/PCDFs and internal standards were used.

To recalculate the concentration of individual PCDD/PCDF isomers in the sample use the following equation:

ALL MATRICES OTHER THAN WATER

$$Cn\ (ug/kg) = \frac{Qis \times (An^1 + An^2)}{W \times (Ais^1 + Ais^2) \times RRFn}$$

WATER

$$Cn\ (ng/L) = \frac{Qis \times (An^1 + An^2)}{V \times (Ais^1 + Ais^2) \times RRFn}$$

Where:

An^1 and An^2 = integrated ion abundances (peak areas) of the quantitation ions of the isomer of interest (Table 5).

Ais^1 and Ais^2 = integrated ion abundances (peak areas) of the quantitation ions of the appropriate internal standard (Table 5).

W= Weight (g) of sample extracted

V= Volume (ml) of sample extracted

Qis= Quantity (ng) of the appropriate

YES NO N/A

internal standard added to the sample prior to extraction
RRFn= Calculated relative response factor from continuing calibration (see Section 7.3).

Note: See SOW, Section 15.3 for calculations when any internal standard in a diluted sample is less than 10% of the internal standard area in the continuing calibration tandard.

7.3 Estimated Detection Limits (EDL)

7.3.1 Was an EDL calculated for each 2,3,7,8-substituted isomer that was not identified regardless of whether other non-2378 substituted isomers were present?

___ ___

7.3.2 Use the equation below to check EDL calculations:

ALL MATRICES OTHER THAN WATER

$$EDL\ (ug/kg) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{W \times (His^1 + His^2) \times RRFn}$$

WATER

$$EDL\ (ng/L) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{V \times (His^1 + His^2) \times RRFn}$$

Where:

Hx^1 and Hx^2 = peak heights of the noise for both quantitation ions of the 2,3,7,8-substituted isomer of interest.

His^1 and His^2 = peak heights of both the quantitation ions of the appropriate internal standards.

D = dilution factor (see Paragraph 10.4.3).

Qis, RRFn, W and V are defined in Section 5.2.10

NOTE: The validator should check the EDL data to verify that peak heights and not areas were used for this calculation. If the area algorithm was used, the validator should contact the laboratory for recalculation. The TPO must be notified.

7.4 Estimated Maximum Possible Concentration (EMPC)

YES NO N/A

7.4.1 Was an EMPC calculated for 2378-substituted isomers that had S/N ratio for the quantitation and confirmation ions greater than 2.5, but did not meet all the identification criteria?

7.4.2 Use the equation below to check EMPC calculations:

ALL MATRICES OTHER THAN WATER

$$EMPC\ (ug/L) = \frac{(Ax^1 + Ax^2) \times Qis \times D}{(Ais^1 + Ais^2) \times RRFn \times W}$$

WATER

$$EMPC\ (ng/L) = \frac{(Ax^1 + Ax^2) \times Qis \times D}{(Ais^1 + Ais^2) \times RRFn \times V}$$

Where:

Ax^1 and Ax^2 = areas of both quantitation ions.

Ais^1, Ais^2, Qis, RRF, D, W, and V are defined in Paragraph 7.3.3 and 10.4.3 and Section 15.1.

Action: 1. If EDL or EMPC of an analyte which was not reported as present is missing, contact the laboratory for correction.
2. If the spot check calculations yielded EDLs or EMPCs different from those reported in Form I, contact the laboratory for an explanation.
3. If EDLs or EMPCs for the most toxic analytes (TEF$\geq$ 0.05) are above CRQLs contact TPO for sample reanalysis.

7.5 Method Blanks

7.5.1 Has a method blank per matrix been extracted and analyzed with each batch of 20 samples?

7.5.2 If samples of some matrix were analyzed in different events (i.e. different shifts or days) has one blank for each matrix been extracted and analyzed for each event?

7.5.3 Acceptable method blanks must not contain any signal

of 2378-TCDD, or 2378-TCDF, equivalent to a concentration of > 0.1 ppb for soils or 1 ppt for water samples.

7.5.4 For other 2378- substituted PCDD/PCDF isomers of each homologue, the allowable concentration in the method blank is less than 1/10 of the CRQL listed in the SOW or the area must be less than 2% of the area of the nearest internal standard.

7.5.5 For the peak which does not meet identification criteria as PCDD/PCDF in the method blank, the area must be less than 5% of the area of the nearest Internal Standard.

ACTION:
1. If the proper number of method blanks were not analyzed, notify the contractor If they are unavailable, reject all positive sample data. However, the reviewer may also use professionaljudgement to accept or reject positive sample data if no blank was run.

2. If the method blank is contaminated with 2378-TCDD, 2378-TCDF, 12378PeCDD, 12378PeCDF or 23478 PeCDF at a concentration higher than the CRQL listed in the SOW, reject all contaminant compound positive data for the associated samples (flag R) and contact the technical project officer to initiate reanalysis if it is deemed necessary.

3. If the method blank is contaminated with any of the above isomers at a concentration of less than the CRQL or of any other 2378-substituted isomer at any concentration and the concentration in the sample is less than five times the concentration in the blank, transfer the sample results to the EMPC/EDL column and cross-out the value in the concentration column. If the concentration in the sample is higher than five times the concentration in the blank, do not take any action.

7.6 Rinsate Blank

7.6.1 One rinsate blank must be collected for each batch of 24 soil samples or one per day whichever is more frequent.

7.6.2 Do any rinsate blanks show the presence of 2378-TCDD, 2378-TCDF, and 12378PeCDD at amounts > .5 ug/L or any other analyte at levels > 1μg/L?

7.6.3 ACTION
If any rinsate blank was found to be contaminated with any of the PCDDs/PCDFs notify the technical project officer to discuss what proper action must be taken.

7.7 Field Blanks

7.7.1 The field blanks are PEM samples (blind blanks) supplied by EPA from EMSL-LV at the frequency of one field blank per 24 samples or less collected over a period of one week whichever comes first. A typical "field blank" will consist of uncontaminated soil. The field blanks are used to monitor possible cross contamination of samples in the field and in the laboratory.

7.7.2 Acceptable field blanks must not contain any signal of 2378-TCDD and 2378-TCDF equivalent to a concentration of > 0.1 ppb.

7.7.3 For other 2378-substituted PCDD/PCDF isomers of each homologue the allowable concentration in the field blank is less than 1/10 the CRQL listed in the SOW.

ACTION: When the field blank is found to be contaminated with target compounds apply the same action as described for the method blank (section 7.5).

NOTE: Contact EPA EMSL/LV to verify that the PEM blank (field blank) did not contain any PCDD/PCDF isomers and ask their assistance in the evaluation of the PE field blank.

8.0 Internal Standard Recoveries (Form I)

8.3.1 Were the samples spiked with all the internal standards as specified in the method?

8.3.2 Were internal standard recoveries within the required limits?

8.3.3 If not, were samples reanalyzed?

ACTION: 1. If the internal standard recovery was below 25 percent, reject (R) all associated non-detect data (EMPC/EDL) and flag with "J" all positive data.

YES NO N/A

2. If the internal standard recovery is above the upper limit (150 percent) flag all associated data (positive and non-detect data) with "J".
3. If the internal standard recovery is less than 10% qualify all associated data R (Reject). When highly toxic isomers (TEF≥ 0.05) are affected, notify TPO to initiate reanalysis.

Calculate the percent recovery of internal standard (Ris) in the sample extract using the following equation.

Recalculate the percent recovery for each internal standard in the sample extract, Ris, using the formula:

$$Ris = \frac{(Ais^1 + Ais^2 \times Qrs \times 100\%)}{(Ars^1 + Ars^2 \times RRFis \times Qis)}$$

Ais^1, Ais^2, Ars^1, Ars_2, Qis, Qrs and RRFis are defined, previously.

9.0 Recovery Standards

There are no contractual criteria for the Recovery Standard area. However, because it is very critical in determining instrument sensitivity, the Recovery Standard area must be checked for every sample.

9.1 Are the recovery standard areas for every sample and blank within the upper and lower limits of each associated continuing calibration?

Area upper limit= +100% of recovery standard area.
Area lower limit= -50% of recovery standard area.

9.2 Is the retention time of each recovery standard within 10 seconds of the associated daily calibration standard?

ACTION:
1. If the recovery standard area is outside the upper or lower limits flag all related positive and non-detect data (EMPC/EDL) with "J" regardless whether the internal standard recoveries met specifications or not.
2. If extremely low area counts (<25%) are reported flag all associated non-detect data as unusable (R) and the positive data J.
3. If the retention time of the recovery

YES NO N/A

standard differs by more than 10 seconds from the daily calibration use professional judgement to determine the effect on the results. A time shift of more than 10 seconds may cause certain analytes to elute outside the retention time window established by the window defining mix.

10.0 Matrix Spikes (PEM Blanks)

10.1 One known blank usually an interference fortified soil/sediment sample, supplied by EPA, EMSL-LV, is designated by the sampling team for the laboratory for spiking. The frequency of this QC sample is one per group of 24 environmental samples or less collected over a period of one week whichever is first. The sample is spiked by the laboratory with the appropriate volume of the matrix spiking solution specified in the analytical protocol (SOW) and then extracted and analyzed with the other samples.

10.2 Was a fortified PEM blank analyzed at the frequency described above? _

10.3 Was the percent recovery of 2378-TCDD and other 2378-substituted compounds within the 50 to 150 percent [control limit?

ACTION: 1. If the recovery of a 2,3,7,8-substituted isomer falls outside the 50-150 percent control limit, flag all positive and non-detect date of the same and related isomers in the same homolog series with J. However, if the recovery is below 20 percent qualify all associated non-detects R. Notify the Technical Project Officer. Reanalysis may be initiated.

2. If no fortified PEM blank was analyzed use professional judgement to assess data validity.

11.0 Matrix Spike (Field Sample)

11.1 Was a matrix spike analyzed at the frequency of one per SDG samples per matrix?

11.2 Was the percent recovery of 2378-TCDD and other 2378-substituted PCDDs/PCDFs within the same 50 to 150 percent?

ACTION: If problems such as interferences are observed, use professional judgement to assess the quality of the data. The 50-150% limits of the matrix spike data may be used to flag data of the spiked sample only. The matrix spike data of the PE blank sample are more important and must be used primarily in data validation.

12.0 Environmental Duplicate Samples

12.1 For every batch of 24 samples or less collected over a period of one week whichever comes first there must be a sample designated as duplicate. Results of the duplicate samples must agree within 50% relative difference.

ACTION: The duplicate results must be used in conjunction of other QC data. If no hits are reported, precision may be assessed from the internal standard recoveries.

13.0 Performance Evaluation Samples

13.1 Included among the samples are sets of performance evaluation samples containing known amounts of unlabeled 2378-TCDD or a mixture of 2378-TCDD and other PCDD/PCDF isomers. The PE samples are provided by the Region, and must be analyzed at the frequency of one set per batch of 24 samples or less collected over a period of one week whichever occurs first.

13.2 The analytical results must be within the EPA 99% acceptance criteria.

ACTION:
1. The PE samples must be validated as if they were environmental samples. There is no holding time for PE samples.

2. PE samples containing only 2378-TCDD
When 2378-TCDD was not qualitatively identified, or if the reported concentration is outside the 99% acceptance window all positive and negative (EMPC/EDL) data for all associated samples are rejected.

3. PE samples containing a mixture of PCDD/PCDF isomers
When the reported concentration of any

YES NO N/A

analyte is outside the EPA 99% confidence interval, all positive and negative (EMPC/EDL) data of the 2378 substituted isomers within the same homologue for all associated samples are rejected.

4. When PCDD/PCDF data are rejected because of PE results, the EPA technical project officer must be notified. Reanalysis may be initiated.

5. For PE blind blanks see 7.7 (Field Blanks)

14.0 Second Column Confirmation

14.1 Was a second column confirmation performed?

14.2 Was the sample extract reanalyzed on a 60m SP-2330 or SP-2331 GC column for better GC resolution and better identification of the individual 2378-substituted isomers?

14.3 Did the second column meet the calibration and linearity specification in the SOW (See sections 5.0 and 6.0).

14.4 Was the % D of the quantitation results of the two columns less than 50?

ACTION: Use professional judgement to decide which quantitation data to use. The two quantitation data should not be combined.

NOTE: If the sample extract was analyzed on a single GC column capable of resolving all 2378-substituted isomers, confirmation is not necessary.

15.0 Sample Reanalysis

15.1 The Region II TPO will evaluate the need for reanalyzing the samples with qualified data based on site-specific Regional Data Quality Objectives. The rerun may be billable or non billable as specified in the SOW. SMO should be notified of all reruns.

15.2 Due to a variety of situations that may occur during sample analysis the laboratory is required to reanalyze or reextract and reanalyze certain samples. If a reanalysis was required but as not performed, contact TPO to initiate reanalysis.

YES NO N/A

List below all reextractions and reanalyses and identify the PCDD/PCDF sample data summaries (Form I) which must be used by the data user (when more than one is submitted).

16.0 Isomer Specificity and Toxicity Equivalency Factor (TEF) - When calculating the 2378-TCDD Toxicity Equivalency of a sample only those 2378 substituted isomers that were positively identified in the sample must be included in the calculations. The sum of the TEF adjusted concentration is used to determine when a second column confirmation is required to achieve isomer specificity.

16.1 The lab did not include EMPC or EDL values in the toxicity equivalency calculations.

16.2 All samples whose toxicity equivalency exceeded the required values were reanalyzed on a confirmation column to establish isomer specificity.

ACTION: 1. If the toxicity equivalency calculations were not performed properly notify TPO.
2. If the toxicity equivalency exceeded the required limits (0.7 ppb for soil/sediment, 7ppt for aqueous and 7ppb for chemical waste samples), and the lab failed to reanalyze the samples on a specific secondary column, notify TPO.

PCDFs/PCDDs Data Assessment

CASE NO._____ LABORATORY_____
Site_____

SAMPLE
NO._____
_____

DATA ASSESSMENT:

All data are valid and acceptable except those values which have been qualified R (rejected) or qualified "J" (estimated). Rejected data does not imply the analyte is not present. It means that due to

significant QC problems the analysis is invalid and it provides no information as to whether the compound is present or not.

All action is detailed below and on the attached sheets.

Reviewer's Signature: _____
Date:____/____/20____

Verified By: _____
Date:____/____/20____

YES NO N/A

Case#_____

Site:_____

Lab:_____

Overall Assessment

YES NO N/A

Case#_____

Site:_____

Lab:_____

Contract Problems/Non-Compliance

SOP HW-13
Revision 3
Date: September 2006

Organic Data Review for Low Concentration Water
CLP/SOW, OLC03.2

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Robert Runyon, Chief, HWSB

Annual Review

Reviewed by:_____ Date:_____

Reviewed by:_____ Date:_____

OLC03.2

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the methods in the "USEPA Contract Laboratory Program Statement of Work Pages for Organics Analysis Low Concentration Water OLC0**3.2**," December 2000. The validation methods and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," June 2001. This document attempts to cover technical as well as contractual problems specific to each fraction; however, situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements are also covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are as follows:

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Lab Qualifiers:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

P - Pesticide/Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications:

Data reviewers must possess a working knowledge of the USEPA Statement of Work OLC03.2 and National Functional Guidelines mentioned above.

. . . . YES NO N/A

. . . .

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:________________________ **LAB**:__________________________________

SITE NAME:__________________________ **SDG No(s).**:___________________________

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ] ___ ___

ACTION: If no contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples and all fractions? [ ] ___ ___

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? ___ [ ] ___

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract Problems/Non-compliance section of the Data Assessment.

2.2 Was CLASS CCS checklist included with the package? [ ] ___ ___

. . . . YES NO N/A

. . . .

		YES	NO	N/A
2.3	Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report and Sample Tags?	___	[ ]	___

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 Cover Letter SDG Narrative

		YES	NO	N/A
3.1	Is the SDG Narrative or Cover Letter Present?	[ ]	___	___
3.2	Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?	[ ]	___	___
3.3	Does the Narrative contain the following information (see SOW, page B-12, section 2.5.1):			
	VOA: description or trap and column(s) used during sample analyses?	[ ]	___	___
	BNA: description of column(s) used during sample analyses?	[ ]	___	___
	PEST: description of columns used during sample analyses?	[ ]	___	___

NOTE: As stated in the SOW, page D-11/PEST, section 6.10.1.3.7, packed columns cannot be used.

		YES	NO	N/A
3.4	Does the narrative, VOA and BNA sections, contain a list of all TICs identified as alkanes and their estimated concentrations?	[ ]	___	___

. . . . YES NO N/A

. . . .

		YES	NO	N/A
3.5	Is the temperature indicator bottle present in the cooler? If not, did the Laboratory document in the SDG Narrative the alternative technique used to determine the cooler temperature?(Exhibit A/ p. A-7 sec. 4.2.1.2.3.3)	[ ]	___	___
3.6	Does the narrative contain a list of the pH values determined for each water sample submitted for volatiles analysis (SOW, page B-13, section 2.5.1.2)?	[ ]	___	___
3.7	Does the Case Narrative contain the "verbatim" statement as required on page B-12, section 2.5.1 of the SOW?	[ ]	___	___

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If the information is unavailable, document under the Contract Problems/Non-Compliance section of the Data Assessment.

4.0 <u>Data Validation Checklist</u>

4.1 Check the package for the following (see SOW reporting requirements, section 2.1, page B-10):

		YES	NO	N/A
a.	Is the package paginated in ascending order starting from the SDG narrative?	[ ]	___	___
b.	Are all forms and copies legible?	[ ]	___	___
c.	Is each fraction assembled in the order set forth in the SOW?	[ ]	___	___

The following checklist is divided into three parts. Part A is filled out if the data package contains any Low Concentration Volatile analyses, Part B for any Low Concentration Semivolatile analyses and Part C for Low Concentration Pesticide/Aroclors.

. . . .

	YES	NO	N/A
. . . .			
Does this package contain:			
Low Concentration Volatiles Data?	___	___	
Low Concentration Semivolatiles Data?	___	___	
Low Concentration Pesticides/Aroclors data?	___	___	

ACTION: Complete corresponding parts of checklist.

. . . . YES NO N/A

. . . .

PART A: VOA ANALYSES

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

ACTION: If both VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

2.0 Holding Times

2.1 Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

Technical Holding Times: The technical holding time criterion for water samples is 14 days from sample collection provided that samples are acid-preserved to pH 2 or below, and that they are stored in 4°C ± 2°C. If uncertain about preservation, notify the TOPO to contact the sampler and determine whether or not samples were preserved.

ACTION: List sampling, VTSR, analysis dates and preservation for samples which missed holding time in the table below.

. . . . YES NO N/A

. . . .

Table of Holding Time Violations
(See Chain-of-Custody Records)

Sample ID	Was Sample Preserved?	Date Sampled	Date Lab Received	Date Analyzed
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________

ACTION: Qualify sample results using preservation and technical holding time information as follows:

a. If there is no evidence that the samples were properly preserved, but were analyzed within the technical holding time (14 days from sample collection), qualify all positive results for non-halogenated compounds (including ketones and aromatics) with “J” and non-detects “R”.

b. If there is no evidence that the samples were properly preserved, but were analyzed within 14 days from sample collection, qualify all positive results for halogenated compounds with “J” and non-detects “UJ”.

c. If there is no evidence that the samples were properly preserved, and the samples were analyzed beyond 14 days from sample collection, qualify positive results for all volatile compounds with “J” and non-detects “R”.

d. If the samples were properly preserved, but were analyzed outside of the technical holding time (14 days from sample collection), qualify positive results for all volatile compounds with “J” and non-detects “R”.

. . . . YES NO N/A

. . . .

NOTE: Contractual Holding Times: Sample must be analyzed within 10 days from validated time of sample receipt (VTSR) at the laboratory.

3.0 Deuterated Monitoring Compound (DMC) Recovery (Form II LCV)

		YES	NO	N/A
3.1	Are the Volatile SMC Recovery Summaries (Form II LCV-1 and LCV-2) present?	[ ]	___	___

ACTION: Call the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

		YES	NO	N/A
3.2	Were outliers marked correctly with an asterisk?	[ ]	___	___

ACTION: Circle all outliers in red.

		YES	NO	N/A
3.3	Were more than three of the fourteen (14) Deuterated Monitoring Compounds (DMC's) recoveries outside their corresponding limits?	___	[ ]	___
	If yes, were samples re-analyzed?	[ ]	___	___
	Were method blanks re-analyzed?	[ ]	___	___

ACTION: If any DMC is outside the required limits (see Table below), qualify their associated target compounds (See Table below) as follows:

VOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

Chloroethane-d5 Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon Disulfide	**1,2-Dichloropropane-d6** Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	**1,2-Dichlorobenzene-d4** Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
Bromoform-d Dibromochloromethane 1,2-Dibromoethane Bromoform	**trans-1,3-Dichloropropene-d4** cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	**Chloroform-d** 1,1-Dichloroethane Bromochloromethane Chloroform
2-Butanone-d5 Acetone 2-butanone	**1,1-dichloroethene-d2** trans-1,2-Dichloroethene cis-1,2-Dichloroethene	**2-Hexanone-d5** 4-Methyl-2-pentanone 2-Hexanone
Vinyl Chloride-d3 Vinyl Chloride	**Benzene-d6** Benzene	**1,1,2,2-Tetrachloroethane-d2** 1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane

1,2-Dichloroethane-d4	**Toluene-d8**	
Trichlorofluoromethane 1,1-Dichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl Acetate Methylene Chloride Methyl tert-Butyl Ether Carbon Tetrachloride 1,2-Dichloroethane 1,1,1-Trichloroethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene Xylenes (total) Styrene Isopropylbenzene	

VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
Vinyl Chloride-d3	49-138	1,2-Dichloropropane-d6	84-123
Chloroethane-d5	60-126	Toluene-d8	77-120
DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
1,1-Dichloroethene-d2	65-130	trans-1,3-Dichloropropane-d4	80-128
2-Butanone-d5	42-171	2-Hexanone-d5	37-169
Chloroform-d	80-123	Bromoform-d	76-135
1,2-Dichloroethane-d4	78-129	1,1,2,2-Tetrachloroethane-d2	75-131

. . . . YES NO N/A

. . . .

Benzene-d6	78-121	1,2-Dichlorobenzene-d4	50-150

1. For any recovery greater than the upper limit:

   a. Qualify “J” all positive associated target compounds.
   b. Do not qualify associated non-detects.

2. For any recovery greater than or equal to 20%, but less than the lower limit:

   a. Qualify “J” all positive associated target compounds.
   b. Qualify “UJ” associated non-detects.

3. For any recovery less than 20%:

   a. Qualify “J” all positive associated target compounds.
   b. Qualify “R” all associated non-detects.

NOTE: Up tp three (3) DMC’s per sample may fail to meet the recovery limits. (SOW OLC03.2, sec. 11.4.4, p. D-41/VOA)
As per SOW, any sample which has more than 3 DMC’s outside the limits, it must be reanalyzed (sec. 11.5.1 p. d-42/VOA).

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis.

3.4 Are there any transcription/calculation errors between raw data and form II? ___ [ ] ___

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.

4.0 Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCV)

4.1 Is the MS/MSD Recovery Form (Form III LCV) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples, whichever is more frequent) for the Low Concentration VOA method? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data alone. However, Using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 Method Blanks (Form IV LCV)

5.1 Is the Volatile Method Blank Summary (Form IV LCV) present? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of Low Concentration VOA TCL compounds, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

5.3 Has a VOA method blank been analyzed at least once every twelve hours for each GC/MS system used? [ ] ___ ___

5.4 Was a VOA instrument blank analyzed after each sample/dilution which contained a target compound at a concentration > 25 µg/ℓ, and ketones > 125 µg/ℓ (see SOW, page D-44/VOA, section 12.1.1.3)? [ ] ___ ___

ACTION: If any method/instrument blank data are missing, notify the TOPO to obtain resubmittals or an

explanation from the lab. If method blank data are unavailable, the reviewer may use professional judgement, or substitute field blank or trip blank data for missing method blank data.

If an instrument blank was not analyzed after a sample containing > 25 µg/ℓ, (ketones > 125 µg/ℓ) inspect the sample chromatogram acquired immediately after this sample for possible carryover. Use professional judgement to determine if carryover occurred and qualify analyte(s) accordingly.

5.5 Was a storage blank analyzed once per SDG after all the samples were analyzed? [ ] ___ ___

ACTION: If storage blank data is missing, contact the TOPO to obtain any missing deliverables from the laboratory. If unavailable, note in the Contract Problems/Non-Compliance section of the Data Assessment.

5.6 The validator should verify that the correct identification scheme for EPA blanks was used. (See SOW page B-30, section 3.3.7.3 for more information.)

Was the correct identification scheme used for all Low Concentration VOA blanks? [ ] ___ ___

ACTION: Contact the TOPO to obtain corrections from the lab, or make the necessary corrections. Document in the "Contract Problems/Non-Compliance section of the Data Assessment all corrections made by the validator.

5.7 Chromatography: review the blank raw data - chromatograms (RICs), quant. reports, data system printouts and spectra.

Also compare the storage blank raw data with the method blank. Determine if contamination in the storage blank is also present in the method blank.

	YES	NO	N/A
Is the chromatographic performance (baseline stability) for each instrument acceptable for Low Concentration VOAs?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

	YES	NO	N/A
5.8 Are all detected hits for target compounds in method, instrument and storage blanks less than the CRQL for that analyte?	[ ]	___	___

Exception: Acetone and 2-butanone must be less than 2X times the CRQL, and Methylene Chloride and Cyclohexane must be less than 10X times its CRQL.

ACTION: If no, an explanation and laboratory's corrective actions must be addressed in the case narrative. If the narrative contains no explanation, then make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

	YES	NO	N/A
6.1 Does the storage blank contain positive results (TCL and/or TICs) for Low Concentration VOAs?	___	[ ]	___

ACTION: If the storage blank contains target compounds at a concentration greater than the CRQL, positive sample results for those compounds should be flagged "J". If gross contamination occurred positive sample results for that compound may be rejected (R).

6.2 Do any method/reagent/instrument blanks contain positive results (including TICs) for Low Concentration VOAs? When applied as described in

the table below, the contaminant concentration in these blanks are multiplied by the sample dilution factor. ___ [ ] ___

NOTE: Contaminated instrument blanks are unacceptable under this SOW (see page D-46/VOA, section 12.1.6.2).

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if a contaminated instrument blank was submitted.

ACTION: Sample analysis results after the high concentration sample must be evaluated for carryover. Sample must meet the maximum carryover criteria as listed in SOW sec. 11.4.9.2, p. D-42/VOA.("the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limit in the contaminated sample.")

6.3 Do any field/trip/rinse blanks have positive Low Concentration VOA results (including TICs)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Field blanks & trip blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

For:	Flag sample result with a "U" when:	Report CRQL & qualify "U" when:	No qualification is needed when:
Methylene Chloride **Cyclohexane**	Sample conc. is > CRQL, but < 10x blank value.	Sample conc. is < CRQL and < 10x blank value.	Sample conc. is > CRQL and > 10x blank value.
Acetone **2-Butanone**	Sample conc. is > CRQL, but < 2x blank value.	Sample conc. is < CRQL and < 2x blank value.	Sample conc. is > CRQL and > 2x blank value.
Other contami-nants	Sample conc. is > CRQL, but < 1x blank value.	Sample conc. is < CRQL and < 1x blank value.	Sample conc. is > CRQL and > 1x blank value.

NOTE: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V-LCV)

. . . . YES NO N/A

. . . .

7.1 Are the GC/MS Instrument Performance Check Forms (Form V-LCV) present for Bromofluorobenzene (BFB)? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List date, time, instrument ID and sample analyses for which associated GC/MS tuning data are missing.

DATE	TIME	INSTRUMENT ID	SAMPLE NUMBERS
________	________	________________	______________
________	________	________________	______________
________	________	________________	______________

ACTION: Notify the TOPO to obtain missing data from the lab. If the lab cannot provide missing data, reject (R) all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 95 (see SOW, page D-24/VOA)? [ ] ___ ___

NOTE: All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

	YES	NO	N/A
7.5 Have the ion abundance criteria been met for each instrument used?	[ ]	___	___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, professional Judgement may be applied to determine to what extent the data may be utilized.

	YES	NO	N/A
7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)	___	[ ]	___
7.7 Is the number of significant figures for the reported relative abundances consistent with the number given in the ion abundance criteria column on Form V LCV?	[ ]	___	___

ACTION: If large errors exist, take action as specified in section 3.1 above.

	YES	NO	N/A
7.8 Is the spectrum of the mass calibration compound acceptable?	[ ]	___	___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.0 Target Compound List (TCL) Analytes (Form I LCV)

8.1 Are the Organic Analysis Data Sheets (Form I LCV) present with required header information on each page, for each of the following:

	YES	NO	N/A
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control/MS/MSD samples?	[ ]	___	___
c. Blanks?	[ ]	___	___

	YES	NO	N/A
8.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control/MS/MSD samples?	[ ]	___	___
c. Blanks?	[ ]	___	___

ACTION: If any data are missing, take action specified in 3.1 above.

	YES	NO	N/A
8.**3** Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other: ________________________?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of the data.

	YES	NO	N/A
8.4 Are lab-generated standard mass spectra of the identified VOA compounds present for each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action as specified in 3.1 above. If lab does not generate their own standard spectra, make note under the "Contract Problems/Non-Compliance" section of the Data Assessment. If spectra are unavailable reject “R” the reported results.

	YES	NO	N/A
8.5 Is the RRT of each reported compound within ± 0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
8.7 Do sample and standard relative ion intensities agree to within ±20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R) flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in sections 8.4-8.7 above.

ACTION: When sample carry-over is suspected, use professional judgement to determine if instrument cross-contamination has affected positive compound identifications.

9.0 Tentatively Identified Compounds (TIC)

	YES	NO	N/A
9.1 Are all Tentatively Identified Compound Forms (Form I LCV-TIC) present? Do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	[ ]	___	___
9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Blanks?	[ ]	___	___

	YES	NO	N/A
b. Are Alkanes listed in/or part of the Case Narrative?	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.1 above.

ACTION: Add "JN" qualifier to all chemically named TICs if missing.

	YES	NO	N/A
9.3 Are any target compounds (from any fraction) listed as TICs? (Example: 1,2-dimethylbenzene is xylene - a VOA target analyte - and should not be reported as a TIC.)	___	[ ]	___

ACTION: Flag with "R" only target compound detected in another fraction. (Except blank contamination)

	YES	NO	N/A
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of TIC identifications. If it is determined that an incorrect identification was made, change its identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (I.e., common lab contaminants such as CO_2 - M/E 44, Siloxanes - M/E 73, hexane, Aldol condensation products, solvent preservatives, and related by-products. See the National Functional Guidelines June 2001, pp. 34-35 for further guidance.)

. . . . YES NO N/A

. . . .

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? (Check at least two positive values. Verify that the correct internal standards, quantitation ions, and RRFs were used to calculate Form I results.) ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions? [ ] ___ ___

ACTION: If errors are large, take action as specified in section 3.1 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs data from the diluted sample). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's not to be used, including any in the data summary package.

11.0 Standards Data (GC/MS)

11.1 Are the reconstructed ion chromatograms, and data system printouts (quant. reports) present for each initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in section 3.1 above.

12.0 GC/MS Initial Calibration (Form VI)

. . . . YES NO N/A

. . . .

12.1 Are the Initial Calibration Forms (Form VI LCV) present and complete for the volatile fraction at concentrations of 0.5, 1, 5, 10, and 25 µg/ℓ? [ ] ___ ___

ACTION: If any Initial Calibration forms are missing, take action as specified in section 3.1 above.

12.2 Are response factors stable for VOA's over the concentration range of the calibration (e.g., %RSD ≤ 30.0, ≤ 50 for poor performers)? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: There are fourteen (14) compounds (see Table below) which are poor performers. The RRF for these compounds must be greater than or equal to 0.01. The %RSD must be less than or equal to 50%.

VOLATILE COMPOUNDS WITH POOR RESPONSE

Volatile Compounds	
Acetone	1,2-Dichloropropane
2-Butanone	1,2-Dibromo-3-chloropropane
Carbon Disulfide	4-Methyl-2-pentanone
Chloroethane	2-Hexanone
Chloromethane	1,2-Dichloropropane-d6 (DMC)
Cyclohexane	2-Hexanone-d5 (DMC)
Chloroethane-d5 (DMC)	2-Butanone-d5 (DMC)

NOTE: Although 20 Low Conc. VOA compounds have no maximum %RSD and require only minimal $\overline{\text{RRF}}$ performance (see Table D-2, page D-53/VOA), the technical acceptance criteria are the same for all analytes.

. . . . YES NO N/A

. . . .

ACTION: If %RSD > 30.0%, or > 50.0% for the poor performers, qualify associated positive results for that analyte "J" (estimated) and non-detects using professional judgement. If %RSD is > 90, flag all non-detects for that analyte "R" (unusable) and positive hits "J".

NOTE: Analytes previously qualified "U" for blank contamination are still treated as "hits" when qualifying for initial calibration criteria.

12.3 Are any $\overline{RRF}$s < 0.05 or < 0.01 for poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If any $\overline{RRF}$ values are < 0.05 or < 0.01 for poor performers, qualify associated non-detects unusable (R) and associated positive results estimated (J).

NOTE: Contract Requirements: The SOW allows up to two of the required analytes (see compounds marked with a "*" on Form VI and Table D-2, page D-53/VOA) to fail contractual %RSD and RRF criteria, provided the %RSD is ≤ 40.0 and RRF ≥ 0.010.

ACTION: If more than two of the required analytes failed %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

12.4 Are there any transcription/calculation errors in the reporting of $\overline{RRF}$s, RRFs or %RSD values? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors in red.

ACTION: If errors are large, contact the TOPO to obtain an explanation/resubmittal from the lab, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 GC/MS Continuing Calibration (Form VII LCV)

	YES	NO	N/A
13.1 Are the Continuing Calibration Forms (Form VII LCV) present and complete for the volatile fraction?	[ ]	___	___
13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	[ ]	___	___

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, ask the TOPO to obtain explanation/resubmittal from the laboratory. If continuing calibration data are unavailable, flag all associated sample data as unusable (R).

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

____

____

	YES	NO	N/A
13.3 Do any volatile compounds have a % Difference (%D) between the initial $\overline{RRF}$ and continuing RRF which exceeds the ± 30% , or ± 50% for the poor performers criteria?	___	[ ]	___

ACTION: Circle all outliers in red.

NOTE: Although 20 Low Conc. VOA compounds have no maximum %D and require only minimal $\overline{RRF}$ performance (see Table D-2, page D-53/VOA), the technical acceptance criteria are the same for all analytes.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When % D is above 90%, reject all non-detects for that analyte as unusable (R) and qualify positive results “J”.

	YES	NO	N/A
13.4 Do any volatile compounds have a RRF < 0.05 or < 0.01 for the poor performers?	___	[ ]	___

YES NO N/A

ACTION: Circle all outliers in red.

ACTION: If the RRF < 0.05, or < 0.01 for poor performers qualify associated positive results as estimated (J) and associated non-detects unusable (R).

NOTE: Contract Requirements: The SOW allows up to two of the required analytes (see compounds marked with a "*" on Form VI, or Table D-2, page D-53/VOA) to fail %D or RRF criteria, provided %D is within ±40.0 and RRF ≥ 0.010.

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if more than two of the required analytes failed the above acceptance criteria.

13.5 Are there any transcription/calculation errors in the reporting of RRFs, or %D between initial RRFs and continuing RRFs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors with red pencil.

ACTION: If errors are large, notify the TOPO to obtain explanation/resubmittals from the lab. Document errors in the Contract Problems/Non-Compliance section of the Data Assessment.

14.0 Internal Standard (Form VIII LCV)

14.1 Are the internal standard areas (Form VIII LCV) of every sample and blank within the upper and lower limits (± 40%) for each continuing calibration? [ ] ___ ___

If no, was the sample reanalyzed? [ ] ___ ___

ACTION: 1. Circle all outliers with red pencil.

2. List all the outliers below.

Sample #	Int. Std.	Area	Lower Limit	Upper Limit
________	__________	_____________	_____________	_____________

. . . . YES NO N/A

. . . .

_________ ___________ _______________ _______________ _______________

_________ ___________ _______________ _______________ _______________

(Attach additional sheets if necessary, or attach copies of Form VIIIs.)

ACTION: 1. If the internal standard area count is outside the **upper** limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when associated IS area counts are > +40%.

3. If the IS area is less than the lower limit (-40%), qualify "J" all positive results quantitated with this Internal Standard. Qualify "R" all non-detects.

INTERNAL STANDARDS ACTIONS FOR VOLATILES

CRITERIA	ACTION	
	Detected Associated Compounds	Non-detected Associated Compounds
Area counts > 40% of 12-hour standard	"J"	No Action
Area counts < 40% of 12-hour	"J"	"R"

. . . . YES NO N/A

. . . .

14.2 Are the retention times of the internal standards within ±20 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 20 seconds.

NOTE: Contract Requirements: The SOW (section 11.5.1 page D-41/VOA) states that any sample which fails the acceptance criteria for IS response must be reanalyzed.

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance any sample(s) which failed the above IS acceptance criteria.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for Low Concentration VOA analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

. . . . YES NO N/A

. . . .

PART B: BNA ANALYSES

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody records or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ] ___

ACTION: If samples were not iced or the ice west melted upon arrival at the laboratory and the temperature of the cooler was > 10^{o} C, then flag all positive results with a “J” and all non-detects “UJ”.

2.0 Holding Times

2.1 Have any Low Concentration semivolatile technical holding times, determined from the date of collection to date of extraction, been exceeded? ___ [ ] ___

Technical Holding Time: Continuous liquid-liquid extraction of BNA samples must begin within seven days of the date of collection. Extracts must be analyzed within 40 days from the extraction date.

Table of Holding Time Violations
(See Chain-of-Custody records)

Sample ID	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________

	YES	NO	N/A

ACTION: If technical holding times were exceeded, flag all positive results as estimated (J) and sample quantitation limits as estimated (UJ), and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J" but the reviewer may determine that non-detect data are unusable (R). If holding times were exceeded by more than 28 days, qualify all non-detects unusable (R).

NOTE: Contractual Holding Times: Extraction of water samples must begin within 5 days VTSR. All laboratory extracts must be analyzed within 40 days of the VTSR.

ACTION: If contractual holding times were exceeded, document in the Data Assessment under Contract Problems/Non-Compliance.

NOTE: The data reviewer must note in the Data Assessment whether or not technical and contractual holding times were met.

3.0 Deuterated Monitoring Compound Recovery (Form II LCSV)

3.1 Are the Low Concentration Semivolatile Deuterated Monitoring Compound Recovery Summaries (Form II LCSV-1 and LCSV-2) present and complete for all samples? [ ] ___ ___

ACTION: Ask the TOPO to obtain explanations/resubmittals of any missing deliverables from the laboratory. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers in red.

3.3 Were more than four, two from each fraction, of the sixteen (16) Deuterated Monitoring Compounds (DMC's) recoveries outside their corresponding limits? ___ [ ] ___

If yes, were samples reanalyzed? [ ] ___ ___

Were method blanks reanalyzed? [ ] ___ ___

ACTION: If any DMC is outside the required limits(See Table below), qualify their associated target compounds (See Table below) as follows:

SEMIVOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

Phenol-d5 Benzaldehyde Phenol	**2-Chlorophenol-d4** 2-Chlorophenol	**2-Nitrophenol-d4** Isophorone 2-Nitrophenol
bis-(2-Chloroethyl)ether-d8 bis-(2-Chloroethyl)ether 2,2'-oxybis(1-Chloropropane) bis(2-Chloroethoxy)methane	**4-Methylphenol-d8** 2-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	**4-Chloroaniline-d4** 4-Chloroaniline Hexachlorocyclo-pentadiene 3,3'-Dichlorobenzidine

s)))

. . . . YES NO N/A

. . . .

Nitrobenzene-d5 Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine	**2,4-Dichlorophenol-d3** 2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol	**Dimethylphtalate-d6** Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate bis(2-Ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d10 Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether	**Anthracene-d10** Hexachlorobenzene Atrazine Phenanthrene Anthracene	**Pyrene-d10** Fluoranthene Pyrene Benzo(a)anthracene Chrysene
Acenaphthylene-d8 Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene Acenaphthylene Acenaphthene	**4-Nitrophenol-d4** 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-nitrophenol 4-Nitroaniline	**Benzo(a)pyrene-d12** Benzo(b)fluoranthene benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol-d2 4,6-Dinitro-2-methylphenol		

SEMIVOLATILE DEUTERATED MONITORING COMPOUND LIMITS

COMPOUND	% RECOVERY

Phenol-d5	10-110
bis-(2-Chloroethyl)ether-d8	41-94
2-Chlorophenol-d4	33-110
4-Methylphenol-d8	38-95
Nitrobenzene-d5	35-114
2-Nitrophenol-d4	40-106
2,4-Dichlorophenol-d3	42-98
4-Chloroaniline-d4	8-70
Dimethylphthalate-d6	62-102
Acenaphthylene-d8	49-98
4-Nitrophenol-d4	9-181
Fluorene-d10	50-97
4,6-Dinitro-2-methylphenol-d2	53-153
Anthracene-d10	55-116
Pyrene-d10	47-114
Benzo(a)pyrene-d12	54-120

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: .If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the Data Assessment.

ACTION: 1. For any recovery greater than the upper limit:

a. Qualify “J” all positive associated target compounds
b. Do not qualify associated non-detects.

2. For any recovery less than the lower limit:

a. Qualify “J” all positive associated target compounds

b. Qualify "UJ" all non-detects if recoveries are ≥ 10% except for 4-Chloroaniline-d4 and 4-Nitrophenol-d4.
c. Qualify "R" all non-detects if recoveries are < 10% except for 4-Chloroaniline-d4 and 4-Nitrophenol-d4.
d. For 4-Chloroaniline-d4 and 4-Nitrophenol-d4 qualify "R" all non-detects if recoveries are less than their lower limit.

NOTE: Up to four DMC's (two per fraction) per sample may fail to meet the recovery limits (SOW OLC03.2, sec. 11.6.4, p. D-34/SV). As per SOW, any sample that fails the technical criteria, must be reanalyzed (sec. 11.7.4 p. D-35/SV).

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if he Lab did not perform reanalysis.

4.0 <u>Laboratory MS/MSD (Form III LCSV)</u>

4.1 Is the Semivolatile MS/MSD Recovery Form (Form III LCSV) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in 3.1 above.

ACTION: No action is taken on MS/MSD <u>alone.</u> However, using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 <u>Blanks (Form IV LCSV)</u>

5.1 Is the Method Blank Summary Form (Form IV LCSV) present? [ ] ___ ___

5.2 <u>Frequency of Analysis</u>: For the analysis of Low Concentration semivolatile TCL compounds, has a method blank been analyzed and reported for each SDG, every 20 samples or each extraction batch, whichever is more frequent? [ ] ___ ___

5.3 Was a Low Concentration semivolatile method blank analyzed for each GC/MS system used? (See SOW page D-36/SV, section 12.1.2.2) [ ] ___ ___

ACTION: If any method blank data are missing, ask the TOPO to obtain an explanation/resubmittal from the laboratory. If method blank data is unavailable, reject (R) all associated positive results. However, the data reviewer may, based on professional judgement, substitute field blank data for missing method blank data.

5.4 The validator should verify that the correct identification scheme for EPA blanks was used. (See SOW page B-30, section 3.3.7.3 for more information.)

Was the correct identification scheme used for all Low Concentration Semivolatile blanks? [ ] ___ ___

ACTION: Contact the TOPO to obtain corrections from the lab, or make the necessary corrections. Document in the "Contract Problems/Non-Compliance section of the Data Assessment all corrections made by the validator.

5.5 Chromatography: Review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra. Is the chromatographic performance (baseline stability) acceptable for each instrument? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

5.6 Are all detected hits for target compounds less than the CRQL for that analyte in all method blanks? [ ] ___ ___

Exception: Phthalate esters must be less than five times (5X) the CRQL.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not

used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method blanks have positive results (TCL and/or TICs) for Low Concentration Semivolatiles? ___ [ ] ___

6.2 Do any field/rinse blanks have positive results for Low Concentration Semivolatiles (TCL and/or TIC)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, spectral, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

NOTE: When applied as described below, the contaminant concentration in these blanks is multiplied by the sample dilution factor.

For:	Flag sample result with a "U" when:	Report CRQL & qualify "U" when:	No qualification needed when:
Common Pthalate-Esters	Sample conc. is > CRQL, but < 5x blank value.	Sample conc. is < CRQL and < 5x blank value.	Sample conc. is > CRQL and > 5x blank value.
Other Conta-minants	Sample conc. is > CRQL, but < 1x blank value.	Sample conc. is < CRQL and < 1x blank value.	Sample conc. is > CRQL and > 1x blank value.

NOTE: Analytes qualified "U" for blank contamination are still treated as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R", unusable.

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in the Data Assessment that there is no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V LCSV)

7.1 Are the GC/MS Instrument Performance Check Forms (Form V LCSV) for Decafluorotriphenylphosphine (DFTPP) present? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analyses per instrument? [ ] ___ ___

ACTION: List samples, date, time and instrument ID for which no associated GC/MS tuning data are available.

SAMPLE ID	DATE	TIME	INSTRUMENT ID
___________	___________	______________	______________________
___________	___________	______________	______________________
___________	___________	______________	______________________

ACTION: If lab cannot provide missing data, reject (R) all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 198? [ ] ___ ___

NOTE: All ion abundance ratios must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may up to 110% that of m/z 198.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

7.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ ___

ACTION: If ion abundance criteria are not met, professional Judgement may be applied to determine to what extent the data may be utilized.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

7.7 Is the number of significant figures for the reported relative abundances consistent with the number given for each ion in the ion abundance criteria column on Form V LCSV? [ ] ___ ___

ACTION: If large errors exist, notify the TOPO to obtain an explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.8 Is the spectrum of the mass calibration compound acceptable? [ ] ___ ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified or rejected.

8.0 <u>Target Compound List (TCL) Analytes (Form I LCSV)</u>

	YES	NO	N/A
8.1 Are the Organic Analysis Data Sheets (Form I LCSV-1, 2) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control/MS/MSD Samples?	[ ]	___	___
c. Blanks?	[ ]	___	___
8.2 Are the Low Concentration Semivolatile reconstructed ion chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following:			
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control Sample(s) and MS/MSD?	[ ]	___	___
c. Blanks	[ ]	___	___
ACTION: If any data are missing, take action as specified in 3.1 above.			
8.3 Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other: _________________________?	[ ]	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
8.4 Are the lab-generated standard mass spectra of identified Low Concentration semivolatile compounds present for each sample?	[ ]	___	___

. . . . YES NO N/A

. . . .

ACTION: If any mass spectra are missing, take action specified in 3.1 above. If lab does not generate their own standard spectra, make note in "Contract Problems/Non-Compliance". If spectra are missing, reject the reported result(s).

8.5 Is the RRT of each reported compound within ± 0.06 RRT units of the standard RRT in the continuing calibration? [ ] ___ ___

8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

8.7 Do sample and standard relative ion intensities agree within ± 20%? [ ] ___ ___

ACTION: Use professional judgement to determine the acceptability of the data. If it is determined that incorrect identifications were made, all such data should be rejected (R) flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the qualitative identification criteria listed in SOW section 11.1, page D-29/SV.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.0 <u>Tentatively Identified Compounds (TIC)</u>

9.1 Are all Tentatively Identified Compound Forms (Form I LCSV-TIC) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? [ ] ___ ___

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

. . . . YES NO N/A

. . . .

	YES	NO	N/A
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Blanks?	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.1 above.

ACTION: Add "JN" qualifier to all chemically named TICs.

	YES	NO	N/A
9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2- dimethylbenzene is xylene a VOA TCL and should not be reported as a TIC)?	___	[ ]	___

ACTION: Flag "R" only TCL compound detected in another fraction. (Except blank contamination)

	YES	NO	N/A
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
9.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. In order to be positively identified, the data must comply with the criteria listed in SOW section 11.2, page D-30/SV.

Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). Common lab contaminants could be solvent preservatives, such as Cyclohexene. Related by-products include Cyclohexanone, Cyclohexanol, Chlorocyclohexene and Chlorocyclohexanol. Aldol reaction products of Acetone include 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten--2-one, and 5,5-dimethyl-2-(5H)-furanone.

. . . . YES NO N/A

. . . .

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions? [ ] ___ ___

ACTION: If errors are large, notify the TOPO to obtain an explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standards Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.1 above.

12.0 GC/MS Initial Calibration (Form VI LCSV)

. . . . YES NO N/A

. . . .

12.1 Are the Initial Calibration Forms (Form VI LCSV-1 & -2) present and complete for the Low Concentration Semivolatile fraction at concentrations of 5, 10, 20, 50 and 80 ug/l? [ ] ___ ___

NOTE: Seven compounds, 2,4-Dinitrophenol, 2,4,5-Trichlorophenol 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline 4-Nitrophenol, 4,6-Dinitro-2-methylphenol, require calibration at 20, 50, 80, 100 and 120 ug/l.

ACTION: If any calibration standard forms are missing, take action specified in 3.1 above.

NOTE: There are nineteen (19) semivolatile compounds (see Table below) which are poor performers. The RRF for these compounds must be greater than or equal to 0.01 The %RSD must be less than or equal to 50%. The %RSD must be less than or equal to 30% for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol, and less than or equal to 20.5% for all other compounds and DMC’s.

SEMIVOLATILE COMPOUNDS WITH POOR RESPONSE

SEMIVOLATILE COMPOUNDS	
2,2'oxybis(1-Chloropropane)	Benzaldehyde
4-Chloroaniline	Pentachlorophenol
Hexachlorobutadiene	4-Nitroaniline
Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol
2-nitroaniline	N-Nitrosodiphenylamine
3-nitroaniline	3,3'-Dichlorobenzidine
2,4-Dinitrophenol	4-Chloroaniline-d4 (DMC)
4-Nitrophenol	4,6-Dinitro-2-methylphenol-d2 (DMC)
Acetophenone	4-Nitrophenol-d4 (DMC)
Caprolactam	

12.2 Are response factors stable (%RSD ≤ 20.5, ≤ 50 for poor performers and ≤ 30 for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol) for Semivolatiles over the entire concentration range of the calibration? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: Although 24 Low Concentration semivolatile compounds have a minimum $\overline{RRF}$ and no maximum %RSD, the technical acceptance criteria are the same for all analytes.

ACTION: If the %RSD exceeds the above criteria, qualify positive results for that analyte "J" and non-detects using professional judgement. When %RSD > 90%, flag all non-detects for that analyte "R", and positive hits as “J”.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

12.3 Are any RRFs **<** 0.05, **<** 0.01 for poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If any RRF < 0.05, or < 0.01 for poor performers:

1. Flag "R" all non-detects.

2. Flag "J" all positive results.

12.4 Are there any transcription/calculation errors in the reporting of, $\overline{RRF}$s, RRFs or % RSD values? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: If errors are large, take action as specified in section 3.1 above.

NOTE: Contract Requirements: The SOW allows up to four (see sec. 9.3.5.4, p. D-21/SV) of the required analytes to fail contractual %RSD or RRF criteria, provided the %RSD is ≤ 40.0 and RRF is ≥ 0.010. (See Table D-4, page D-48, 49/SV

and analytes marked with a "*" on Form VI LCSV for a list of required analytes and contractual criteria.

ACTION: If more than four analytes fail %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 GC/MS Continuing Calibration (Form VII LCSV)

13.1 Are the Continuing Calibration Forms (Form VII LCSV-1 & -2) present and complete for the semivolatile fraction? [ ] ___ ___

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List below all sample analyses that were not within twelve hours of a continuing calibration analysis for each instrument used.

____

____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, notify the TOPO to obtain explanation/resubmittals. If continuing calibration data are not available, flag all associated sample data as unusable (R).

13.3 Do any semivolatile compounds have a %D between the initial $\overline{RRF}$ and continuing RRF which exceeds the ± 25.0% criteria? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is > 90%, reject all non-detects for that analyte (R) unusable and positive results “J”.

	YES	NO	N/A
13.4 Do any semivolatile compounds have a RRF < 0.05, <0.01 for the poor performers?	___	[ ]	___

ACTION: Circle all outliers with red pencil.

ACTION: If the RRF is < 0.05, < 0.01 for the poor performers, qualify associated positive results estimated (J) and non-detects unusable (R).

	YES	NO	N/A
13.5 Are there any transcription/calculation errors in the reporting of continuing RRFs or %D between initial $\overline{RRF}$s and continuing RRFs? (Check at least two values, but if errors are found check more.)	___	[ ]	___

ACTION: Circle errors with red pencil.

ACTION: If errors are large, notify the TOPO to obtain explanation/resubmittals, make any necessary corrections and document the effect in the data assessment.

14.0 Internal Standards (Form VIII LCSV)

	YES	NO	N/A
14.1 Are the Internal Standard Area and RT Summary Forms (Form VIII LCSV-1 & -2) present and complete for the semivolatile fraction?	[ ]	___	___
14.2 Are the internal standard areas for every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	[ ]	___	___

ACTION: Circle errors with red pencil.

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
________	____________	____________	____________	___________
________	____________	____________	____________	___________

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag all positive results and non-detects quantitated with this internal standard "J" and "UJ", respectively.

2. Do not qualify non-detects associated with IS areas > 100%.

3. If the IS area is < 50%, qualify all associated non-detects estimated “R”.

INTERNAL STANDARDS ACTIONS FOR SEMIVOLATILES

CRITERIA	ACTION	
	Detected Associated Compounds	Non-Detected Associated Compounds
Area counts > 100% of 12-hour standard	“J”	No Action
Area counts < 50% of 12-hour standard	“J”	“R”

14.3 Are the retention times of the internal standards within 20 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 20 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for Low Concentration semivolatile analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. If large

. . . . YES NO N/A

. . . .

differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

PART C: PESTICIDE/AROCLOR ANALYSIS

1.0 Sample Conditions/Problems

1.1 Do Traffic Reports/Chain-of-Custody records or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? [ ] ___ ___

ACTION: If samples were not iced, or the ice was melted upon arrival to the laboratory, and the temperature of the cooler was > 10^{o} C, flag all positive results "J" and all non-detects "UJ".

ACTION: Check extraction log for sample pH, if adjustment was needed, it should have been noted in the SDG Narrative. If more information is needed, notify the TOPO to contact the lab.

2.0 Holding Times

2.1 Have any Pest/Aroclor technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Technical Holding Times: Continuous liquid-liquid extraction of samples for Pesticide/Aroclor analysis must begin within seven days of collection. Extracts must be analyzed within 40 days of extraction.

Table of Holding Time Violations
(See Chain-of-Custody records)

Sample ID	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________

. . . . YES NO N/A

. . . .

________	________	________	________	________
________	________	________	________	________
________	________	________	________	________

ACTION: If technical holding times were exceeded, flag all positive results as estimated (J) and sample quantitation limits (UJ) and document in the Data Assessment that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable (R).

NOTE: Contractual Holding Times: Extraction of water samples must begin within 5 days VTSR. All laboratory extracts must be analyzed within 40 days of the VTSR.

ACTION: If contractual holding times were exceeded, document in the Data Assessment under Contract Problems/Non-Compliance.

3.0 Surrogate Recovery (Form II LCP)

3.1 Are the Low Concentration Semivolatile Surrogate Recovery Summaries (Form II LCSV) present and complete for all samples? [ ] ___ ___

ACTION: Notify the TOPO that explanation/resubmittals are required from the laboratory. If missing deliverables are unavailable, document effect in data assessments.

3.2 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with red pencil.

. . . . YES NO N/A

. . . .

3.3 Were surrogate recoveries of TCX or DCB in any sample or blank outside of the contractual limits of 30 - 150%? ___ [ ] ___

ACTION: If either surrogate spike recovery is outside the acceptance limits, the Validator must consider the existence of coelution and interference in the raw data and use professional judgement as described below, as surrogate recovery problems may not directly apply to target analytes.

. . . . YES NO N/A

. . . .

1. For any surrogate recovery greater than 150%:

   a. Qualify positive hits as estimated “J”.

   b. Do not qualify Non-detects.

2. For any surrogate recovery greater than or equal to 10%, but less than 30%.

   a. Qualify positive hits as estimated “J”.

   b. Qualify Non-detects as “UJ”.

3. For any surrogate recovery less than 10%, ignoring dilutions, and in the absence of interference

   a. Qualify positive hits as estimated “J”.

   b. Qualify Non-detects as unusable “R”.

Surrogate Actions for Pest/PCB Analyses

Criteria	**Action** *	
	Detected Associated Compounds	**Non-detected Associated Compounds**
%R > 150%	“J”	No qualification
10% ≤ %R < 30%	“J”	“UJ”
%R < 10% (ignore dil’s)	“J”	“R”
RT out of RT window	Professional Judgement	

***** Use professional judgement in qualifying data as surrogate recovery problems may not directly apply to target analytes.

Pesticides Surrogates and Associated Target Compounds

Tetrachloro-m-Xylene	**Decachlorobiphenyl**	
alpha-BHC	alpha-Chlordane	4,4'-DDE
beta-BHC	gamma-Chlordane	4,4'-DDT
gamma-BHC	Heptachlor epoxide	Endosulfan I
delta-BHC	Dieldrin	Endosulfan II
Heptachlor	Endrin	Endosulfan sulfate
Aldrin	Endrin Aldehyde	Methoxychlor
	Endrin ketone	Aroclors
	4,4'-DDD	Toxaphene

3.4 Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A (See Form VI LCP-1)? [ ] ___ ___

ACTION: If the RT limits are not met, positive results and non-detects may be qualified unusable (R) for that sample based on professional judgement.

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, notify the TOPO to obtain explanation/resubmittals. Make any necessary corrections and document effect in data assessments.

4.0 Laboratory Control Sample (LCS)

4.1 Is the Laboratory Control Sample (LCS) Recovery Form (Form III LCP-2) present? [ ] ___ ___

4.2 Was the LCS analyzed at the required frequency (once per SDG, or every 20 samples) for the Low Concentration Pest/Aroclor method? [ ] ___ ___

ACTION: If any LCS data are missing, take action as specified in 3.1 above.

4.3 How many PEST spike recoveries (see Table below) are outside QC limits listed in Table D-3, page D-61/PEST of the SOW?

Pesticides Laboratory Control Sample (LCS) spike compounds and limits.

LCS Spike Compound	Recovery Limits (%)	LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50-120	Endosulfan sulfate	50-120
Heptachlor epoxide	50-150	gamma-Chlordane	30-130
Dieldrin	30-130	TMX (Surrogate)	30-150
4,4'-DDE	50-150	DCB (Surrogate)	30-150
Endrin	50-120		

ACTION: Check calculations, surrogates, LCS solutions and instrument performance.

ACTION: Qualify only the <u>specific analytes</u> included in the LCS solution in the following two situations:

1. If the LCS recovery is greater than the upper QC limit, qualify positive results for the affected compound(s) estimated (J). Do not qualify non-detects.

2.If the LCS recovery is less than the lower QC limit, then qualify positive results for the affected compound(s) estimated (J) and non-detects unusable (R).

Qualify <u>all sample results</u> in the following situations

1. If 25% or more of the analyte recoveries are below QC limits qualify all associated positive results "J" and non-detects "R".

2. If two or more analytes exhibit < 10% recovery, qualify all associated positive results "J" and non-detects "R".

It should be noted in the Data assessment if a laboratory fails to analyze an LCS with each SDG, or consistently fails to generate acceptable LCS recoveries.

5.0 <u>Laboratory MS/MSD (Form III LCP-1)</u>

5.1 Is the Pest/PCB MS/MSD Recovery Form (Form III LCP-1) present? [ ] ___ ___

5.2 Was the MS/MSD analyzed at the required frequency (Once per SDG, or every 20 samples? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as Specified in 3.1 above.

ACTION: No action is taken on MS/MSD <u>alone.</u> However, using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

6.0 Blanks (Form IV LCP)

6.1 Is the Method Blank Summary (Form IV LCP) present? [ ] ___ ___

6.2 Frequency of Analysis: For the analysis of Pesticide/Aroclor TCL compounds, has a method blank been analyzed concurrently for each SDG, every 20 samples or each extraction batch, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified in section 3.1 above. If blank data is unavailable, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.3 A separate Form IV LCP should be present if just part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV LCP submitted when required? [ ] ___ ___

ACTION: If sulfur clean-up blank data and Form IV are missing, take action as specified in 3.1 above.

6.4 Has a Pest/Aroclor instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified in section 3.1 above.

6.5 Was the correct identification scheme used for all Pest/PCB blanks? (See SOW, page B-30, section 3.3.7.3 for further details.) [ ] ___ ___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the

. . . . YES NO N/A

. . . .

Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

6.6 Chromatography: Review the blank raw data - chromatograms, quant reports or data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable for Pest/PCBs?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/cleanup blanks have positive results for Pest/Aroclors?	___	[ ]	___
7.2 If any method, instrument and/or sulfur clean-up blanks contain "hits" for target compounds, are these hits greater than the CRQL for that analyte?	___	[ ]	___

ACTION: Note in the Data Assessment under Contract Problems/Non-Compliance if any method, instrument or sulfur clean-up blank(s) contain hit(s) at concentration(s) greater than the CRQL for that analyte.

7.3 Do any field/rinse blanks have positive Pest/Aroclor results?	___	[ ]	___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

NOTE: When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor.

Flag sample result with a "U":	**Report CRQL & qualify "U":**	**No qualification is needed:**
Sample conc. > CRQL, but < 1x blank.	Sample conc. < CRQL & is < 1x blank value.	Sample conc. > CRQL & > 1x blank value.

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

7.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in Data Assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 Calibration and GC Performance

8.1 Are the following gas chromatograms and data systems printouts for both columns present for all samples, blanks, and LCS:

a. Peak Resolution Check? [ ] ___ ___

b. PEM standards? [ ] ___ ___

c. Aroclor 1016/1260? [ ] ___ ___

d. Aroclors 1221, 1232, 1242, 1248, 1254? [ ] ___ ___

e. Toxaphene? [ ] ___ ___

	YES	NO	N/A
f. Low points Individual Mixtures A & B?	[ ]	___	___
g. Med points Individual Mixtures A & B?	[ ]	___	___
h. High points Individual Mixtures A & B?	[ ]	___	___
i. Instrument blanks?	[ ]	___	___
j. Were appropriate GC columns used (see SOW, page D-10/PEST, section 6.10.1.3)?	[ ]	___	___

ACTION: If no, take action as specified in 3.1 above.

	YES	NO	N/A
8.2 Do chromatograms for all initial calibration standards (Resolution Check Mixtures, Individual Standard Mixtures A & B and PEM) display single component peaks at > 10% but < 100% of full scale?	[ ]	___	___
Do chromatograms for multi-component standards display all peaks between 25% and 100% of full scale?	[ ]	___	___
Were chromatograms for at least one each of Standard Mixtures A & B replotted to display standard peaks between 50% and 100% of full scale?	[ ]	___	___
Have chromatograms for the above standards been replotted, when necessary, showing the scaling factor used to meet the above requirements?	[ ]	___	___

NOTE: All standard chromatograms must clearly display single component peaks at > 10% but < 100% of full scale, and multi-component peaks between 25% and 100% of full scale. At least one analysis each of Standard Mixtures A & B must display standard peaks between 50% and 100% of full scale. Chromatograms must be replotted, if necessary, to accommodate peaks not properly scaled initially. Both the initial and replotted chromatograms must be submitted with the data package. (See SOW, page D-25/PEST, section 9.2.5.10 for details.)

. . . . YES NO N/A

. . . .

ACTION: If all single component peaks in all standard chromatograms are not clearly displayed and properly scaled, notify the TOPO to obtain resubmittals of the necessary data.

8.3 Are Forms VI LCP-1 through VI LCP-7 present and complete for each column and each analytical sequence? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

8.4 Are there any transcription/calculation errors between raw data and Forms VI LCP? ___ [ ] ___

ACTION: If large errors exist, notify the TOPO to obtain explanation/resubmittals, make necessary corrections and document the effect in data assessments.

8.5 Do all standard retention times, for each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration sequence (see Form VI LCP-1)? [ ] ___ ___

ACTION: If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R). For Aroclors, the RT may be outside the RT window (Form VI LCP-3), but the Aroclor may still be identified from the individual pattern.

8.6 Have the linearity criteria been satisfied for the initial analyses of Individual Standard Mixtures A & B for both columns (Form VI LCP-2)? %RSD must be $\leq$ 25.0 for α- and δ-BHC, $\leq$ 30.0 for the two surrogates and $\leq$ 20.0 for all other analytes. [ ] ___ ___

NOTE: Contractual requirements allow up to two single-component analytes, except surrogates, to exceed the linearity criteria provided %RSD ≤ 30.0. (See SOW, section 9.2.5.7, page D-25/PEST.) The technical criteria, however, are the same for all analytes.

ACTION: If technical criteria were not met, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". If %RSD is > 90, flag all non-detects for that analyte unusable (R).

ACTION: Note in the Contract Problems/Non-Compliance section of the Data Assessment and the Organic Regional Data Assessment Summary if more than two analytes exceeded the 20.0 percent limit.

8.7 Is the resolution between each pair of adjacent peaks in the Resolution Check Mixture ≥ 60.0% on both columns (Form VI LCP-4)? [ ] ___ ___

ACTION: If no, qualify positive results for inadequately resolved compounds "J". Use professional judgement to determine if non-detects, which elute in areas affected by coeluting peaks, should be qualified "N" (presumptive evidence of presence) or "R" (unusable).

8.8 Is Form VI LCP-5 present and complete for each PEM standard used for <u>both initial and continuing</u> calibrations (see SOW page B-45, section 3.12.4)? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1 above.

8.9 For each PEM standard, was the resolution between each pair of adjacent peaks ≥ 90.0% on both columns? [ ] ___ ___

ACTION: Qualify positive results for compounds not adequately resolved estimated (J). Qualify non-detects based on professional judgement.

8.10 Have Forms VI LCP-6 & -7 been completed for all midpoint Individual Standards A and B used for initial calibration? [ ] ___ ___

For each standard, was the resolution between each pair of adjacent peaks $\geq$ 90.0% on both columns? [ ] ___ ___

ACTION: If no, qualify positive results for compounds that were not adequately resolved estimated (J). Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" (presumptive evidence of presence) or unusable (R).

8.11 Is Form VII Pest-1 present and complete for each PEM standard analyzed during the analytical sequence for both columns? [ ] ___ ___

Was the % breakdown of DDT and Endrin calculated using the equations given on page D-22/PEST, sec. 9.2.4.8 in the SOW? [ ] ___ ___

Were all pesticides and surrogates in each PEM standard within the RT windows established during the Initial Calibration? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1 above.

8.12 Has the individual % breakdown on either column exceeded 20.0% for:

4,4'-DDT? ___ [ ] ___

Endrin? ___ [ ] ___

Has the combined breakdown for 4,4'-DDT and Endrin exceeded 30.0% on either column (required for all PEM analyses)? ___ [ ] ___

ACTION: 1. If any % breakdown has failed the QC criteria in either PEM in steps 2 and 17 in the <u>initial calibration</u> sequence (SOW, page D-20/PEST, section 9.2.3.4) qualify all sample analyses in the entire analytical sequence as described below.

2. If any % breakdown has failed the QC criteria in a PEM Verification calibration, review data beginning

with the samples which followed the last in-control standard until the next acceptable PEM & qualify the data as described below.

a. 4,4'-DDT Breakdown: If 4,4'-DDT breakdown is greater than 20.0%:

i. Qualify all positive results for 4,4'-DDT "J".

ii. Qualify positive results for 4,4'-DDD and/or 4,4'-DDE "J".

iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE are detected qualify the quantitation limit for 4,4'-DDT as unusable "R", and qualify positive results for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity "JN".

b. Endrin Breakdown: If Endrin breakdown is greater than 20.0%:

i. Qualify all positive results for Endrin with "J".

ii. Qualify positive results for Endrin ketone and Endrin aldehyde as estimated "J".

iii. If Endrin was not detected, but Endrin Aldehyde and/or Endrin ketone are detected, qualify the quantitation limit for Endrin as unusable "R", and qualify positive results for Endrin Aldehyde and/or Endrin ketone as presumptively present at an approximate quantity "JN".

c. Combined Breakdown: If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0%:

i. The validator should consider the degree of individual breakdown of 4,4'-DDT and Endrin and apply qualifiers as described above.

8.13 Are the %D values for all PEM analytes ≥ -25.0% and ≤ +25.0% (Form VII LCP-1)? [ ] ___ ___

	YES	NO	N/A

ACTION: If no, qualify all associated positive results generated during the analytical sequence "J" and sample quantitation limits "UJ".

NOTE: If the failing PEM is part of the initial calibration, all samples are potentially affected. If the offending standard is a verification calibration, the associated samples are those which followed the last in-control standard until the next passing standard.

	YES	NO	N/A
8.14 Have all samples been injected within 12 hrs. of an acceptable instrument blank?	[ ]	___	___

ACTION: If no, use professional judgement to determine the severity to the effect on data reliability.

	YES	NO	N/A
8.15 Is Form VII LCP-2 present and complete for each INDA and INDB calibration verification analyzed?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1 above.

	YES	NO	N/A
8.16 Are there any transcription/calculation errors between raw data and Form VII LCP-2?	___	[ ]	___

ACTION: If large errors exists, notify the TOPO that explanation/resubmittals from the lab are required. Make any necessary corrections and document in the Data Assessment under Contract Problems/Non-Compliance.

	YES	NO	N/A
8.17 Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established during the initial calibration sequence?	[ ]	___	___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

8.18 Are all %D values for INDA and INDB calibration verification compounds ≥ -25.0% and ≤ +25.0%? [ ] ___ ___

ACTION: If the %D is outside the ±25.0% range for any compound(s), qualify associated positive results for that compound "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte(s) in question. If the %D is > 90%, flag all non-detects for that analyte "R" (unusable).

9.0 Analytical Sequence Check (Form VIII LCP)

9.1 Is Form VIII LCP present and complete for each column and each period of analyses? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

9.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses (see SOW pages D-39 & D-40/PEST)? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

9.3 Were all samples analyzed within a 12 hour time period beginning with the injection of an instrument blank and bracketed by acceptable analyses of the proper standards? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Document in the Data Assessment under Contract Problems/Non-Compliance.

9.4 If a multi-component analyte was detected in a sample, was a matching multi-component standard (Toxaphene or Aroclors) analyzed within 72 hours of the sample and within a valid 72-hr. sequence? [ ] ___ ___

NOTE: This standard is for identification purposes only. Positive results for Aroclors and Toxaphene are quantitated from the initial calibration.

ACTION: If no, document in the Contract Problems/Non-Compliance section of the Data Assessment and Organic Regional Data Assessment Summary.

10.0 Cleanup Efficiency Verification (Form IX LCP)

10.1 Is Form IX LCP present and complete for each lot of Florisil Cartridges used? (Florisil cleanup is required for all Pest/Aroclor extracts.) [ ] ___ ___

Are all samples listed on the Pesticide Florisil Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above. If the data suggests Florisil cleanup was not performed, note in the Data Assessment under Contract Problems/Non-Compliance.

10.2 Are percent recoveries (% REC) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedure within QC limits, 80 - 120%, for the Florisil cartridge check?

[ ] ___ ___

ACTION: If %REC of one or two TCL compounds is < 80%, qualify positive results "J" and non-detects "UJ" for these compounds.

If more than two compounds exhibited < 80% recovery, qualify all associated positive results "J" and non-detects "UJ".

If two or more have %REC < 10%, qualify all positive results "J", and non-detects "R". Use professional judgement to qualify positive results if recoveries are > 120%.

NOTE: Sample data should be evaluated for potential interferences if recovery of 2,4,5-Trichlorophenol was >

5% in the Florisil Cartridge Performance Check analysis. Note in Contract Problems/Non-Compliance section of reviewer narrative.

11.0 <u>Pesticide/Aroclor Identification (Forms X LCP-1 & -2)</u>

11.1 Are Forms X LCP complete for every sample in which a pesticide and/or Aroclor were detected? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

11.2 Are all sample chromatograms properly scaled, attenuated, etc. as required for proper identification of single and multi-component analytes? (See SOW, page D-46/PEST, sections 11.3.1 thru 11.3.9.8 for specific details.) ___ [ ] ___

NOTE: Proper verification of Pest/PCB results depends on clear, legible presentation of the raw data. Single component pesticides and all peaks chosen for quantitation of multi-component analytes must appear at less than 100% of full scale (see SOW). Toxaphene and PCB patterns must be clearly visible to enable comparison with standard chromatograms.

ACTION: If retention times or apex of peaks cannot be verified, or if multi-component peak patterns cannot be discerned, contact the TOPO to obtain rescaled chromatograms from the lab.

11.3 Are there any transcription/calculation errors between raw data and Forms 10LCA and 10LCB? [ ] ___ ___

ACTION: If large errors exists, notify the TOPO that explanation/resubmittals from the lab are required. Make any necessary corrections and document in the Data Assessment under Contract Problems/Non-Compliance and in the Organic Regional Data Assessment Summary.

11.4 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

ACTION: Use professional judgement to qualify positive results. Qualify as unusable (R) all positive results which were not confirmed on a second GC column. Also qualify as unusable (R) all positive results not within the RT window unless associated standards are similarly biased (see Functional Guidelines). Use professional judgement to assign an appropriate quantitation limit.

11.5 Is the percent %D calculated for positive sample results on the two columns > 25.0? ___ [ ] ___

NOTE: If %D is > 25.0, lab should have reported results with the "P" qualifier.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
0 - 25%	None
26 - 70%	"J"
71 - 100%	"JN"
> 100%	"R"
100 - 200% (Interference detected)*	"JN"
> 50% (Pesticide value is < CRQL)**	"U"

* When the reported %D is 100 - 200%, but interference is suspected on either column, qualify the data with "J".

** When the **reported** pesticide value is lower than the CRQL, and the %D is > 50%, raise the value to the CRQL and qualify "U", undetected.

NOTE: For Aroclors, if the %D is > 50%, but the pattern of GC peaks on both columns indicates a specific Aroclor is present, qualify that Aroclor "J".

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the Data Assessment.

11.6 Check chromatograms for false negatives (especially the multiple peak compounds Toxaphene and PCBs). Were there any false negatives? ___ [_] ___

ACTION: Use professional judgement to decide if the compound should be reported. If the appropriate Aroclor standards were not analyzed within 72 hrs. of the sample(s) in question, qualify the data unusable (R).

Also note in Data Assessment under Contract Problems/Non-Compliance if the lab failed to analyze Aroclor standards when required.

12.0 Target Compound List

12.1 Are the Organic Analysis Data Sheets (Form 1 LCP) present with required header information for each of the following:

a. Samples? [_] ___ ___

b. LCS analyses? [_] ___ ___

c. Method Blanks? [_] ___ ___

d. Instrument Blanks? [_] ___ ___

e. Matrix Spike/Matrix Spike Duplicate? [_] ___ ___

12.2 Are the chromatograms and quant. reports included in the sample data package for each of the following:

a. Samples? [_] ___ ___

b. LCS analyses? [_] ___ ___

c. Method Blanks? [_] ___ ___

d. Instrument Blanks? [_] ___ ___

e. Matrix Spike/Matrix Spike Duplicate? [_] ___ ___

ACTION: If any data are missing, take action as specified in section 3.1 above.

12.3 Is chromatographic performance acceptable with respect to:

a. Baseline stability? [ ] ___ ___

b. Resolution? [ ] ___ ___

c. Peak shape? [ ] ___ ___

d. Full-scale graph attenuation? [ ] ___ ___

e. Other: ________________________________? [ ] ___ ___

12.4 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

ACTION: Use professional judgement to determine the acceptability of the data. Address comments under System Performance section of the Data Assessment.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive results. Were any errors found? ___ [ ] ___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. Use professional judgement to decide whether a large discrepancy indicates the presence of an interfering compound. If an interfering compound is suspected, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity "JN". This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has interfered with the evaluation of the second column confirmation.

13.2 Are the CRQLs adjusted to reflect sample dilutions? [ ] ___ ___

YES NO N/A

ACTION: If large errors exist, take action as specified in section 3.1 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample). Replace concentrations which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer may offer an approximated quantitation limit (UJ) for each affected compound.

NOTE: If a sample required greater than a 10 times dilution, then a 10 times more concentrated analysis must also be performed and submitted (see SOW, page D-41/PEST, section 10.2.**3.5**).

ACTION: If a more concentrated analysis is unavailable, document in the Contract Problems/Non-Compliance section of the Data Assessment. Use professional judgement to qualify non-detects and positive hits below the CRQL.

14.0 Field Duplicates

14.1 Were any field duplicates submitted for Pest/Aroclor analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

Definitions

BFB - bromofluorobenzene
BHC - benzene hexachloride
BNA - base neutral acid
CADRE - Computer Aided Data Review and Evaluation
CARD - CLP Analytical Results Database
CCS - contract compliance screening
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
GC - gas chromatography
GC/EC - gas chromatography/electron capture detector
GC/MS - gas chromatography/mass spectroscopy
GPC - gel permeation chromatography
kg - kilogram
µg - microgram
MAGIC - Mainframe Access Graphical Interface with CARD
ℓ - liter
LCS - Laboratory Control Sample
LES - Laboratory Evaluation Sample
mℓ - milliliter
PCB - Polychlorinated Biphenyl
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
$\overline{RRF}$ - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX -tetrachloro-m-xylene
TIC - tentatively identified compound
TPO - technical project officer
VOA - volatile organic acid

VTSR - validated time of sample receipt
TOPO - Task Order Project Officer

References

SOW/CLP OLC03.2
National Functional Guidelines (June 2001)

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ SDG(s):_____

SITE:_____ LAB:_____

This Region II SOP document is based on Method TO-15: Determination of Volatile Organics Compounds (VOCs) in Air Collected in Specially-Prepared Canisters & Analyzed by Gas Chromatography/Mass Spectrometry, January 1999.

1.0 Data Completeness and Deliverables

1.1 Have any missing deliverables been received and added to the data package? [ ] ___ ___

ACTION: Contact lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect under "Contract Problems/ Non-Compliance" section of data assessment report.

2.0 Cover Letter, Narrative, and Data Reporting Forms

2.1 Is the Lab. Narrative and Cover Page present? [ ] ___ __

2.2 Is Case Number contained in the Narrative? [ ] ___ __

2.3 Are the following Data Reporting Forms present?

Analysis Data Sheet [Form I/Equivalent] [ ] ___ __

Tentatively Identified Compounds [Form I-TIC] [ ] ___ __

Blank Summary [Form IV/Equivalent] [ ] ___ __

Laboratory Control Sample Data Sheet [Form III/Equivalent] [ ] ___ __

GC/MS Instrument Performance Check and Mass Calibration [Form V/Equivalent] [ ] ___ __

Initial Calibration [Form VI/Equivalent] [ ] ___ __

Continuing Calibration [Form VII/Equivalent] [ ] ___ __

Internal Standard Area and RT Summary
[Form VIII/Equivalent] [ ] ___ __

Canister Certification [Form IX/Equivalent] [ ] ___ __

3.0 Canister Receipt/Log-in Sheet

Receipt of each canister is recorded in a laboratory notebook dedicated to this use. The sample receipt/log-in sheet must demonstrate that the information on custody records, traffic reports, and sample tags agree for each sample.

3.1 Do all info items agree with each sample ? [ ] ___ __

ACTION: If these documents are not consistent, contact Project officer or laboratory and attach a record of resolution.

4.0 Traffic Reports and Laboratory Narrative

4.1 Are the Traffic Report Forms present for all samples? [ ] ___ __

ACTION: If no, contact lab for replacement of missing or illegible copies.

5.0 Holding Times

5.1 Have any VOA technical holding times of 30 days, determined from the date of sample collection to the date of analysis, been exceeded? ___ [ ] __

NOTE: The contract requires that samples must be retained from verified time sample receipt (VTSR) until 45 days after delivery of a complete sample data package to the Agency.

VOA Table of Holding Time Violations

Sample ID	Sample Matrix	Date Lab Received	Date Analyzed

ACTION: If technical holding times have been exceeded, flag all results unusable ("R").

6.0 Leak Test Evaluation

6.1 All canisters are leak tested prior to each sampling use.
Form IX/Equivalent - summarizes the canister certification for each canister. The initial gauge pressure should be approximately 206 kPa (30 psi) with zero air.

Did the pressure test not vary by more than ± 13.8 kPa (± 2 psi) over the 24 hours period? ___ [ ] _

ACTION: If the canister does not meet the leak-tight criteria all results should be flagged "R".

7.0 Canister Certification Form IX/Equivalent

7.1 Blank Analysis

All canisters have to be checked after cleaning.

Were the target analytes < the required detection limits specified in the task order? [ ] ____ _

Note: Samples with large amount of non target analytes can be valid as long as this criterion is met for target analytes.

ACTION: If the lab failed to do so, it should be noted under contract non-compliance, and laboratory should be notified. Use Table 1 below to qualify samples with target compounds results also present in certification blanks.

Certification Contamination
TABLE 1

Certification Contamination	**Sample Result**	**Action for Sample**
≥ detect limit specified in task order	> 5X certification contamination	No qualification required
≥ detect limit specified in task order	< detect limit specified in task order	detection limit with U
≥ detect limit specified in task order	≥ detect limit and ≤ 5X certification contamination level	5X certification contamination with U
< detect limit specified in task order	≤ detection limit and ≥ detection limit	no qualification

7.2 Is the canister certification form provided, and the associated canister sample identification included? When contamination, included contamination detected

(all raw data),analyte and reference mass spectra. [ ] ___ ___

ACTION: If no, have EPA project officer/TOPO contact laboratory for missing documents.

8.0 Laboratory Control Samples

8.1 Is an LCS Data Sheet (Form III/Equivalent) present and complete for each LCS? [ ] ___ ___

8.2 Was an LCS prepared (10ppbv total scan) (0.1ppbv SIM) and analyzed at the required frequency (once per 24 hour analytical sequence, and concurrently with the samples in the SDG)? [ ] ___ ___

ACTION: Call lab for explanation/resubmittals. If missing deliverables or information is unavailable, document the effect in the data assessment.

8.3 Are there any transcription/calculation errors between the raw data and Form III/Equivalent? Check LCS target compound recoveries. ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effects in the data assessment.

8.4 Is the % recovery within 70-130 % for each LCS target compound reported on Form III/Equivalent? [ ] ___ ___

ACTION: Professional judgement should be used to qualify the impact on sample data, if the recoveries are outside the given limits.

8.5 Is the RT of each reported LCS compound within the windows established during the most recent valid calibration? [ ] ___ ___

If the most recent calibration is the initial calibration use mid level standard (10 ppbv).

ACTION: Professional judgement should be used to qualify sample data, if retention times differ by more than 20 seconds.

8.6 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2? [ ] ___ ___

ACTION: If not, see Sections 18.1 and 18.2.

ACTION: Circle outliers in red.

ACTION: Always use professional judgement. If qualification is necessary, follow the criteria below and in Table 2.

1. If any LCS compounds are outside the specified limits, the associated sample results for the outlying compounds should be qualified as indicated in Table 2 below.

2. If the absolute RT for any LCS compound is outside the established windows, then qualify positive results and non-detects in the associated environmental sample data for that LCS compound(s) (See Table 2). All non-LCS compounds should be qualified using professional judgement.

Laboratory Control Samples
TABLE 2

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R
% RECOVERY			
Detects	70 - 130%	< 70%, > 130%	
Non-detects	≥ 130%	50 - 69%	< 50%
ABSOLUTE RT OF LCS COMPOUNDS			

LCS Compounds in samples RT: (min)	± 0.33	> ± 0.33

9.0 GC/MS Instrument Performance Check

9.1 Are the GC/MS Instrument Performance Check

Forms (Form V/Equivalent) present for Bromofluorobenzene (BFB)? [ ] ___ __

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the 50 ng BFB provided for each twenty four hour shift? [ ] ___ __

9.3 Has the instrument performance compound been analyzed for every twenty four hours of sample analysis per instrument? [ ] ___ __

ACTION: List date, time, instrument ID, and sample analysis for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

9.4 Have the ion abundances been normalized to m/z 95? [ ] ___ __

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

9.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ __

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

9.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [ ] __

9.7 Have the appropriate number of significant figures (two) been reported? [ ] ___ __

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

9.8 Are the spectra of the mass calibration compound acceptable? [ ] ___ __

ACTION: Use professional judgement to determine whether associated data should be accepted, or qualified.

10.0 Performance Evaluation Sample (Optional)

10.1 The PE sample will assist the Agency in monitoring Contractor performance. The lab will not be informed as to which compounds are contained in the PE samples or the concentrations. Was a PE sample submitted from the Agency with each SDG? [ ] ___ __

10.2 PE samples must be validated like environmental samples. There is no holding time for PE samples. If the data results do not comply with the Agencies' spike results use professional judgement together with other QC criteria in order to determine usability of the other data in the SDG. If the associated data was rejected because of PE results, the EPA technical project officer must be notified.

10.3 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2? [ ] ___ __

ACTION: If not, see Sections 18.1 and 18.2.

11.0 Laboratory Method Blanks

11.1 Is an Analysis Data Sheet (Form IV/Equivalent) present and complete for each method blank? [ ] ___ __

11.2 Frequency of analysis:

Has a method blank analysis been reported per instrument for each 24-hour analytical sequence? [ ] ___ __

Has a method blank been analyzed after the initial calibration or a valid calibratio check standard, and before the LCS, prior to sample analysis? [ ] ___ __

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, reject ("R") all positive data.

11.3 Chromatography: review the blank raw data - chromatograms, quant reports and data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable? [ ] ___ __

ACTION: Use professional judgement to determine the effect on the data.

11.4 Were the area response of each Internal Standards (IS) in the blank within ± 40% of the mean area response of the IS of the most recent valid calibration? [ ] ___ __

Were the RT of each IS within ± 0.33 min (20 sec.) between blanks & most recent valid calibration [ ] ___ __

ACTION: If not, see section 18.1 and 18.2.

12.0 Blank Contamination

12.1 Do any method blanks have positive target and non-target VOA results ? ___ [ ] __

ACTION: Use Table 3 below to qualify samples with target compound results also present in the associated blank. Use the largest value from all the associated method blanks if more than one method blank was run.

VOA Laboratory Blanks
TABLE 3

Samples	Not Qualified	non detect U
Target Compounds	> 5X Blank value	≤ 5X Blank Level*

*** If sample result is also less than CRQL, report as not detected (U) at [CRQL].**
Note that the dilution factor has to be taken into account when calculating the Blank Level.

13.0 Target Compound Analytes

13.1 Are the Organic Analysis Data Sheets (Form I-, Equivalent), VOA chromatograms, and data system printouts present and complete with required header information for each of the following:

a. Samples? [ ] ___ ___
b. Method blanks? [ ] ___ ___
c. Laboratory Control Sample (LCS)? [ ] ___ ___
d. Performance Evaluation Sample (PES)? [ ] ___ ___

ACTION: If any data are missing, take action specified in 1.1 above.

13.2 Is chromatographic performance acceptable with respect to:

a. Baseline stability? [ ] ___ ___
b. Resolution? [ ] ___ ___
c. Peak shape? [ ] ___ ___
d. Full-scale graph (attenuation)? [ ] ___ ___
e. Other: [ ] ___ ___

13.3 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

ACTION: Use professional judgement to determine the acceptability of the data. Address comments under "System Performance" section of data assessment.

13.4 Is the sample component relative retention time (RRT) within ± 0.06 RRT units of the RRT of the standard component from the most recent continuing calibration? [ ] ___ ___

NOTE: If the most recent calibration is a calibration curve, the mean RRT (RRT) should be used for comparison.

ACTION: If the above criteria is not met, professional judgement should be used to qualify sample data.

13.5 Was Nafion dryer used? ___ [ ] ___

ACTION: In cases where Nafion tubing is used to dry the sample stream, polar target and non target compounds must not be reported.

ACTION: Reject all polar compounds if reported as non detects. Polar compounds reported as positive hits should be flagged "J".

14.0 Tentatively Identified Compounds (TIC)

14.1 Are all Tentatively Identified Compound Forms (Form I-TIC) present and are retention time, estimated concentration and "JN" qualifier listed corresponding to each TIC? [ ] ___ ___

14.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following?

a. Samples [ ] ___ ___

b. Blanks [ ] ___ ___

ACTION: If any TIC data are missing, take action specified in 1.1 above.

ACTION: Add "JN" qualifier if missing.

14.3 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

14.4 Do TIC and "best match" standard relative ion intensities agree within 20%? [ ] ___ ___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

Also, when a compound is not found in any blanks, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (e.g., Common Lab Contaminants: CO_2 (M/E 44), Siloxanes (M/E 73), Aldol Condensation Products, Solvent Preservatives, and related by products.

15.0 Initial Calibration and System Performance (Form VI/Equivalent)

15.1 Were each GC/MS system calibrated at 5 concentrations that span the monitoring range of interest in an initial calibration sequence to determine the sensitivity and the linearity of the GC/MS response for the target compounds**?** [ ] ___ ___

ACTION: If any calibration standard forms or raw data are missing, take action specified in section 1.1 above.

15.2 Was the same volume introduced into the trap consistently for all field and QC-sample analyses? [ ] ___ ___

15.3 Were the area response (Y) at each calibration level within $\pm$ 40% of the mean area response (mean Y) over the initial calibration range for each Internal Standard? [ ] ___ __

Did the laboratory tabulate the area response (Y) of the primary ions and the corresponding concen-

tration for each compound and Internal Standard? [ ] ___ __

ACTION: If the range exceeds ± 40% for particular compounds, flag these compounds "J" for positive and non-detects in the associated samples. If the %RSDs exceeds ± 90%, associated sample non-detect compounds should be rejected (R) and associated hits as estimate (J).

15.4 Are the relative retention times (RRT) for each of the target compounds at each calibration level within ± 0.06 RRT units of the mean relative retention time for the compound? [ ] ___ __

ACTION: If no, reject the associated sample compounds.

15.5 Are all individual RRF and average RRFs ≥ 0.050? [ ] ___ ___

NOTE: For the following compounds the individual RRF and average RRF must be ≥ 0.01.

2-Butanone
Carbon disufide
Chlorethane
Chlormethane
1,2-Dibromoethane
1,2-Dichloropropane
1,4-Dioxane
1,2-Dibromo-3-chloropropane
Methylene chloride

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 9 compounds in 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

15.6 Are response factors (RF) stable i.e. % Relative Standard Deviation (%RSD) ≤30.0% with at most two exceptions up to limit of ± 40%? [ ] ___ __

ACTION: Circle all outliers in red.

ACTION: If %RSD > 30.0%, qualify associated positive results for that analytes "J" and non-detects are not qualified. When RSD > 90%, flag all non-detects for that analytes R (unusable) and associate positive values as estimate (J).

NOTE: Analytes previously qualified "U" for blank contamination are still considered as "hits" when qualifying for initial calibration criteria.

15.7 Are there any transcription/calculation errors in the reporting of average response factors (RRFs) or %RSDs? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effects in data assessment.

15.8 Are the RT shift for each Internal Standard (IS) at each calibration level within 20s of the mean RT over the initial calibration range of each IS? [ ] ___ ___

16.0 Daily Calibration (Form VII/Equivalent)

16.1 Are the daily Calibration Forms (Form VII/Equivalent) present and complete for the volatile fraction? [ ] ___ ___

16.2 Has a daily calibration standard (10 ppbv total scan) (0.1ppb SIM)been analyzed for every twenty four hours of sample analysis per instrument after the BFB tuning analysis? [ ] ___ ___

ACTION: List below all sample analyses that were not within 24 hours of the daily calibration analysis.

_____

_____

_____

ACTION: If any forms are missing or no daily calibration standard has been analyzed within 24 hours of every sample analysis, call lab for explanation/resubmittal. If daily calibration data are not available, flag all associated sample data as unuable ("R").

16.3 Do any volatile compounds have a % Difference (% D) between the initial and daily RRFs which exceed the ± 30% criteria? ___ [ ] __

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When % D is above 90%, reject non-detects as R) unusable and associated positive values (J).

16.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %difference (%D) between initial and daily RRFs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Contract Non-Compliance".

17.0 <u>Compound Quantitation and Reported Detection Limits</u>

17.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct average RRF of the initial calibration was used to calculate Form I results. [ ] ___ ___

17.2 Are the reported detection limits adjusted to reflect sample dilutions? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under “Contract Non-Compliance" of the data assessment.

NOTE: When a sample is analyzed at more than one dilution, the lowest CRQLs are used

(unless a QC accedence dictates the use of the higher CRQL data from the diluted sample analysis). Cross out "E" from the original analysis. Replace the concentrations in the original analysis with the ones from the diluted sample. Specify which Form I is to be used. Draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

17.3 Have any target compound concentrations exceeded the calibration range of the GC? ___ [ ] ___

ACTION: If yes, flag as estimated ("J").

17.4 Was more than one method of quantitation used to calculate sample results within a batch or 24 hr. analytical sequence? ___ [ ] ___

17.5 Did the lab report the target compounds below CRQLs with the suffix "J"? [ ] ___ ___

ACTION: When appropriate, include suffix "J".

18.0 Internal Standard (Form VIII/Equivalent)

18.1 Are the 3 internal standard areas (Form VIII) of every sample, LCS, PE, and blank within the upper and lower limits (+40% to -40%) for each continuing calibration or 10 ppbv level of initial calibration? [ ] ___ ___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit

ACTION: 1. If the internal standard area count is outside the limit, flag all positive results quantitated with this internal standard with a "J."

2. Non-detects associated with IS area

counts > 40% are not qualified.

3. If IS area is below the lower limit (< 40%), qualify all associated non-detects (U values) "J". If extremely low area counts are reported, (< 25%), or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

18.2 Are the internal standard retention times in each sample, LCS, PE, and blank within 20 seconds of the corresponding retention times in the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify sample data if the internal standard retention times differ by more than 20 seconds.

19.0 Mass Spectral Interpretation/Identification

19.1 Are the Organic Analysis Data Sheets present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control Samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

19.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (quant. reports) included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control Samples [ ] ___ ___

c. Blanks? [ ] ___ ___

ACTION: If any data are missing, take action specified in 1.1 above.

19.3 Is chromatographic performance acceptable with respect to:

a. Baseline stability? [ ] ___ ___

b. Resolution? [ ] ___ ___

c. Peak shape? [ ] ___ ___

d. Full-scale graph (attenuation)? [ ] ___ ___

e. Other: _____? [ ] ___ ___

ACTION: Use professional judgement to determine the acceptability of the data.

19.4 Are the lab-generated standard mass spectra of the identified compounds present for each sample? [ ] ___ ___

ACTION: If any mass spectra are missing, take action as specified in 1.1 above. If the lab does not generate its own standard spectra, document in the Contract Problems/Non-compliance section of the Data Assessment.

19.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? [ ] ___ ___

19.6 Are all ions present in the reference standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

19.7 Do sample and reference standard relative ion intensities agree within ±20%? [ ] ___ ___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected "R", flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected "U" at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 19.5, 19.6, and 19.7

20.0 Field Duplicates

20.1 Were any field duplicates submitted for VOA analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Note the RPD value in the data assessment.

DATA ASSESSMENT

This Data Assessment is based on USEPA Region II SOP HW- : Volatile Organics Analysis of Ambient Air in Canisters by Method TO-15, May 2004.

Case No. _____ **SDG No**. _____ **LABORATORY:** _____

SITE : _____

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "U"(non-detects), "R" (unusable), or “N” (presumptive). All action is detailed on the following sheets.

The following facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error. In addition the “N” flag shows that the analysis indicates the presence of an analyte for which there is presumption evidence to make a “tentative identifiction.”

All actions are detailed below and on the attached sheets:

Overall Assessment:

Contract Non-Compliance:

Reviewer's
Signature:_____ Date:_____/____/20__

Verified By:_____ Date:_____/____/20__

Validating Chlorinated Herbicides
GC, SW-846, Method 8151A

Prepared by: George Karras Date: 12/05/06
George Karras, Chemist HWSS

Peer Reviewed by: Russell Arnone Date: 12/05/06
Russell Arnone, Chemist HWSS

Concurred by: Linda Mauel Date: 12/5/06
Linda Mauel Chief HWSS

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief HWSB

Annual Review

Reviewed by:_____ Date:_____

Reviewed by:_____ Date:_____

1.0 Traffic Reports and Laboratory Narrative

1.1 Are Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated (J). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced (4°C) upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Has the technical holding times,determined from date of sample receipt to date of extraction, been exceeded? ___ [ ] ___

Note: Samples may be analyzed for herbicide ester and acid. Check Laboratory SDG Narrative.

Note: Aqueous samples must be extracted within 7 days. Extracts must be analyzed within 40 days following extraction.
Soil/Concentrated Waste samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.

ACTION: If technical holding times are exceeded, flag all positive results and non-detects(U)as estimated ("J") and document in the narrative that holding times were exceeded.
Samples extracted more than 28 days from sample receipt, either on the first analysis or upon re-analysis, flag all positive results as

YES NO N/A

estimate ("J") and non-detects as unusable (R).

3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Are the Herbicide Surrogate Recovery Summaries (Form II/Equivalent) present for each of the following matrices?

a. Aqueous [ ] ___ ___

b. Soil [ ] ___ ___

3.2 Are all the samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?

a. Aqueous [ ] ___ ___

b. Soil/Concentrated Waste [ ] ___ ___

ACTION: Contact lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with red pencil.

Note: recommend surrogate is 2,4-Dichlorophenylacetic acid (DCAA)

3.4 Did the laboratory provide their developed in-house QC limits/recoveries? [ ] ___ _____

ACTION: If no, use 70 -130% recovery to qualify data

ACTION: No qualification is done if the surrogate is diluted out. If recovery for the surrogate is below the QC limit, but above 10%, flag all results for that sample "J". If recovery is < 10%, qualify postive results "J" and flag non-detects "R". If recovery is above the QC limits limit, qualify positive values "J".

Note: In-house QC limits must be examined for reasonableness, e.g. 10-170% may be appropriate for analytes not present in the sample.

YES NO N/A

Note: Matrix effect is indicated if the LCS data are within limits but surrogate data exceeds QC limits.

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point calibration analysis? [ ] ___ ___

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgement.

3.6 Are there any transcription/calculation errors between raw data and Form II/Equivalent? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III/Equivalent) present? [ ] ___ ___

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices?

Note: At a minimum, analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair with each batch of up to 20 samples.

a. Aqueous [ ] ___ ___

b. Soil/Concentrated Waste [ ] ___ ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 Did the laboratory provide their developed in-house QC limits/recoveries? [ ] ___ ___

ACTION: If no, use 70 -130% recovery to qualify data

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike results in conjunction with

YES NO N/A

other QC criteria (e.g. LCS) to determine the need for qualification of the data.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis: has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take the action specified above in 3.2. If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 Has a Herbicide instrument blank been analyzed at the beginning of every analytical sequence of 10 samples ? [ ] ___ ___

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

5.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Herbicides? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent/cleanup blanks have positive results for Herbicides? When applied as described in table below, the contaminant concentration in the method blank is multiplied by the sample dilution factor and corrected for % moisture when necessary. ___ [ ] ___

6.2 Do any field/rinse blanks have positive Herbicides results? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, calibration, or any QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Calibration and GC Performance

7.1 Are the Gas Chromatograms and Data Systems

	YES	NO	N/A
printouts for both columns present for all samples, blanks, QC Check references, and matrix spikes?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

	YES	NO	N/A
7.2 Are Form VI/Equivalent present and complete for each column and each analytical sequence?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

	YES	NO	N/A
7.3 Are there any transcription/calculation errors between raw data and Forms VI?	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

	YES	NO	N/A
7.4 Were the retention time windows calculated using the average absolute retention time (at least three measurements) ± three times the standard deviation of the absolute retention time, for each standard? (Refer to Method 8000A, section 7.5).	[ ]	___	___
7.5. Was a LCS check standard analyzed prior to environmental samples?	[ ]	___	___
7.5.1 If yes, was the surrogate recovery >50%?	[ ]	___	___
7.5.2 Was the LCS check standard re-extracted/re-analyzed, if surrogate recovery was <50%, or any one analyte was < 40%, or two analytes < 70% ?	[ ]	___	___

Action: If No/' to any of the above, then qualify positive hits as estimated "J" and non-detects as rejected "R" in the original analysis of all samples in the associated analytical sequence.

	YES	NO	N/A
7.6 Do all standard retention times, including each Herbicides in each level of Initial Calibration fall within the windows established during the initial calibration analytical sequence? (For Initial Calibration Standards, Form VI/Equivalent - Herbicides - 1).	[ ]	___	___

ACTION: If no, all samples in the entire analytical sequence are potentially

YES NO N/A

affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogate is visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.7 Are the linearity criteria for the Initial Calibration analyses within limits for both columns? (% RSD must be < 20.0% for all analytes). [ ] ___ ___

ACTION: If no, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When RSD >90%, flag all non-detect results for that analyte R (unusable).

7.8 Are there any transcription/calculation errors between raw data and Form VII - Herbicides-2? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

7.9 Is the resolution between any two adjacent peaks in the QC Reference Check Mixture > 60.0% for both columns? (Form VI-Herbicides- 4) [ ] ___ ___

ACTION: If no, positive results for compounds that were not adequately resolved should be qualified "J". Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable (R).

7.10 Is Form VII -Continuing Calibration present and complete for each analytical sequence for both columns? [ ] ___ ___

ACTION: If no, take action as specified in

YES NO N/A

3.2 above.

7.11 Have all samples been injected within a 24 hr. period beginning with the injection of the first standard? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly.

7.12 Do all analyte retention times for the Mid-concentration Check standard (Form VII Herb-2) fall within the windows established by the initial calibration sequence? [ ] ___ ___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present

and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.13 Are RPD values for all verification calibration standard compounds < 25.0% [ ] ___ ___

ACTION: The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria.

If %D is 25 -50% qualify as "J"
If %D is 51-100% qualify as "NJ"
If %D is >100% qualify as "R"
If %D is >100% with visible interferences/qualify as "JN"

8.0 Analytical Sequence Check (Form VIII)

8.1 Is Form VIII present and complete for each column

	YES	NO	N/A
and each period of analyses?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

		YES	NO	N/A
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses? (see SAS Client Request/section 8/paragraph 6)	[ ]	___	___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

9.0 Herbicides Identification

		YES	NO	N/A
9.1	Is Form X complete for every sample in which a Herbicide was detected?	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

		YES	NO	N/A
9.2	Are there any transcription/calculation errors between raw data and Form X.	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors in data assessment.

		YES	NO	N/A
9.3	Are retention times (RT) of sample compounds within the established RT windows for both columns?	[ ]	___	___
	Was GC/MS confirmation provided instead of confirmation by a second dissimilar column?	[ ]	___	___

Action: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis or by GC/MS. Also qualify as unusable (R) all positive results not meeting RT window unless associated standard compounds show a similar RT shift. The reviewer should use professional judgement to assign an appropriate quantitation limit.

9.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns

YES NO N/A

< 25.0%? [ ] ___ ___

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
25-50 %	J
50-90 %	JN
> 90 %	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

9.5 Check chromatograms for false negatives.
Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values.
Were any errors found? ___ [ ] ___

NOTE: The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity (NJ). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate the presence of interferences during the evaluation of the second column confirmation.

	YES	NO	N/A
10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	[ ]	___	___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

	YES	NO	N/A
10.3 Have all data (Forms and associated chromatograms and quantitation reports) been submitted for original, diluted or re-extraction/re-analysis samples?	[ ]	___	___

11.0 Chromatogram Quality

	YES	NO	N/A
11.1 Were baselines stable?	[ ]	___	___
11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen?	___	[ ]	___

ACTION: Address comments under System Performance of data assessment. Explain use of professional judgement where used to qualify data.

12.0 Field Duplicates

12.1 Were any field duplicates submitted for Herbicides analysis? [ ] ___ ___

Note: Check whether SAS Client Request required field duplicates.

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.