

Data Validation SOP

Congener PCB, Rev. 2

Congener PCBs

**Environmental Data Services
 Congener PCB (USEPA METHOD 1668A)
 Data Validation Checklist**

SITE:

DATE:

SDG:

Y N N/A

Data Completeness and Deliverables

Are the Field Chain of Custody Forms present for all samples?

Is the Narrative or Cover Letter present?

Do the Field Chain of Custody 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 judgment to evaluate the effect of the noted problems on the quality of the data.

Reporting Requirements and Deliverables

Are the following forms present?

Sample Data Summary?

Spiked Sample Summary?

Duplicate Sample Summary?

Method Blank Summary?

Analytical Sequence Summary?

Initial Calibration?

Continuing Calibration?

Y N N/A

GC/MS Displays

Standard and sample SICP's. SICP's must list date and time of analysis; the file name; sample number; and instrument I.D. number?

Percent peak resolution valley?

Integrated area and peak height must be listed for all peaks 2.5 time above background?

All peaks must show retention time at the maximum height?

Laboratory Records

GC/MS Standard and Sample Run Log in chronological order?

Sample Extraction Log?

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.

Holding Times

Have any holding times been exceeded for:

aqueous samples 30 days from sample collection to extraction?

soil/sediment samples 30 days from sample collection to extraction?

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.

If holding times from collection to extraction have been grossly exceeded, use professional judgment to determine whether non-detects shall be rejected.

Y N N/A

Column Performance

Column performance is assessed based on the evaluation of data obtained from the daily (12-hr. shift) diluted combined 209 congener solution.

The retention time for decachlorobiphenyl (PCB 209) must be greater than 55 minutes.

The column must uniquely resolve congeners 34 from 23 and 187 from 182, and congeners 156 and 157 must co elute within 2 seconds at peak maximum.

NOTE: Unique resolution means a valley height of less than 40% of the shorter of the two peaks.

ACTION: If column performance criteria were not met, a detailed discussion of the problems observed and potential effects on analytical data obtained should be included in the data assessment narrative.

Initial 5-Point Calibration

The initial calibration standard solutions (CS1-CS5) must be analyzed prior to any sample analysis. These calibration standards should include all CBs listed at the concentrations specified in Table 5 of Method 1668A. They do not have to be analyzed daily provided the continuing calibration standard met all criteria. The calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze samples.

SIM data were acquired for each of the ions listed in Table 7.

The following criteria must be met:

For IUPAC CBs (3, 4, 15, 19, 28, 37, 52, 77, 81, 138, 156/157, 167, 169, 178, 189, 194, 205, 206 & 208), the two ions for each CB must maximize simultaneously and within 3 seconds of the corresponding labeled isomer ions.

For the other CBs, the 2 ions for each of the CBs being quantified must maximize simultaneously.

The relative ion abundance criteria listed in table 8 must be met.

The relative ion abundance criteria for the labeled internal and recovery standards listed in table 8 must be met.

Y N N/A

For all calibration solutions, the signal to noise ratio (S/N) for all ions of the native compounds must be greater than 10.

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

The percent relative standard deviation (%RSD) of the five RRFs (CS1-CS5) for the CBs must not be greater than 20 percent.

Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled CBs and internal standards were used. In addition verify that the appropriate internal standard was used for each isomer.

ACTION: If required information were not supplied, notify the lab. If unavailable reject all associated sample data. If any calibration curve standards fail to meet any acceptance criteria, check for re-calibration and re-analysis

Calibration by Isotopic Dilution Procedure

(Native CBs included in the Toxics/LOC CBs list)

To recalculate the response factor for targets quantitated based on isotopic dilution, use the equation:

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

Where:

A_n^1 and A_n^2 = integrated areas of the two quantitation ions of isomer of interest (Table 7).

A_{is}^1 and A_{is}^2 = integrated areas of the two quantitation ions of the appropriate labeled compound (Table 7).

Q_n = quantity of the unlabeled CB analyte injected (ng)

Q_{is} = quantity of the appropriate labeled compound injected (ng)

Calibration by Internal Standard

(Native CBs not included in the Toxics/LOC CBs list)

Internal standard calibration is performed at a single point using the diluted combined 209-congener solution (Table 3).

To calculate the response factor for targets based on the internal standard method, use the equation:

$$RF = \frac{(A1_s + A2_s) C_{is}}{(A1_{is} + A2_{is}) C_s}$$

Where:

$A1_s$ and $A2_s$ = The areas of the primary and secondary m/z's for the PCB.

$A1_{is}$ and $A2_{is}$ = The areas of the primary and secondary m/z's for the internal standard.

C_{is} = The concentration of the internal standard (Table 5).

C_s = The concentration of the compound in the calibration standard (Table 5).

Continuing Calibration

At the beginning of each 12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native CBs and labeled compounds. For these tests, analysis of the Cs-3 calibration verification (VER) standard (Section 7.10.1 and Table 5) and the diluted compound 209-congener solution (Section 7.10.2.2 and Table 5) must be used to verify all performance criteria. Adjustment and/or recalibration (Section 10) must be performed until all performance criteria are met. Only after all performance criteria are met, may samples, blanks, IPRs and OPRs be analyzed.

Was the continuing calibration run at the required frequency? _____

ACTION: If the continuing calibration standard was not analyzed at the required frequency, reject all the data.

For native Toxics and LOC CBs, the two SIM ions for each PCB must maximize simultaneously and within 3 seconds of the corresponding ions of the labeled isomers. _____

For the other ions for each of the CBs being quantified, must maximize simultaneously.

	Y	N	N/A
For the continuing calibration standard solution, the signal to noise ratio (S/N) for the CBs ions shall be greater than 10.	_____	_____	_____
The absolute retention times of the labeled Toxics/LOC/window defining standard congeners in the verification test must be within ± 15 seconds of the respective retention times in the calibration. Has this criteria been met?	_____	_____	_____
The relative retention times of native CBs and labeled compounds in the verification test must be within their respective RRT limits in Table 2. Has the criteria been met?	_____	_____	_____
For the internal standards and the recovery standards, the signal to noise ratio (S/N) shall be greater than 10.	_____	_____	_____
The relative ion abundance criteria (Table 8) for all PCB's native shall be met.	_____	_____	_____
Evaluate the concentrations of the Toxics/LOC CBs in the CS-3 VER standard.			
For each compound, compare the concentration with the calibration verification limit in Table 6. If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. Was this criteria met?	_____	_____	_____
Spot-check continuing calibration concentrations and ion ratios. Verify that the appropriate quantitation ions for the unlabeled PCB's and internal standards were used.			

ACTION: If the required continuing calibration fail to meet any of the acceptance criteria, check for recalibration and reanalysis. If not performed reject all data associated with the analyte out of specification in the continuing calibration.

Y N N/A

Sample Data

SIM data were acquired for each of the ions listed in Table 7.

Identification Criteria

- A. For native Toxics and LOC CBs, the two ions for each of the PCB's being quantified must maximize simultaneously and within 3 seconds of the corresponding labeled standard.

For the remaining CBs, the two ions for each of the CBs being quantified must maximize simultaneously.

- B. 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.

- C. The integrated ion current for the internal and recovery standard characteristic ions must be at least 10 times background noise.

- D. The relative ion abundance criteria (Table 8) for all PCB's found present must be met or ion ratios must be within $\pm 15\%$ of the most recent CS-3 determination.

- E. The relative retention time of the peak for a positively identified CB must be within the RRT QC limits specified in Table 2, or, if an alternate column or column system is employed, within its respective RRT QC limits for the alternate column or column system.

ACTION: Reject (flag R) all positive data for the analytes which do not meet criteria listed in Sections A.

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

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

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

Y N N/A

Spot check calculations for positive data and verify that the same internal standards used to calculate RRFs were used to calculate concentration.

Compute the concentrations in the extract of the Native Toxics/LOC CBs, using the RRs from the calibration data and following equation:

$$C_{ex} \text{ (ng/mL)} = \frac{(A1_n + A2_n) C_i}{(A1_i + A2_i) RR}$$

Where:

C_{ex} = The concentration of the PCB in the extract, and the Other terms as defined in Section 10.5.1.

Internal Standard Quantitation and Labeled Compound Recovery

Compute the concentrations in the extract of the native compounds other than those in the Native Toxics/LOC standard, in the Labeled cleanup standard, and in the Labeled injection internal standard (except for Labeled CB178), using the response factors determined from the calibration data and the following equation:

$$C_{ex} \text{ (ng/mL)} = \frac{(A1_s + A2_s) C_{is}}{(A1_{is} + A2_{is}) RF}$$

Where:

C_{ex} = The concentration of the labeled compound in The extract. The other terms are defined in Section 10.6.1.

Using the concentration in the extract determined above, compute the percent recovery of the Labeled Toxics/LOC/window-defining CBs and the Labeled cleanup standard CBs using the following equation:

$$\text{Recovery (\%)} = \frac{\text{Concentration found (ug/mL)} \times 100}{\text{Concentration spiked (ug/mL)}}$$

Y N N/A

The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids as follows:

$$\text{Concentration in solid (ng/kg)} = \frac{(C_{ex} \times V_{ex})}{W_s}$$

Where:

C_{ex} = The concentration of the compound in the extract.

V_{ex} = The extract volume in mL.

W_s = The sample weight (dry weight) in kg.

The concentration of a native CB in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted as follows:

$$\text{Concentration in aqueous phase (pg/L)} = \frac{(C_{ex} \times V_{ex})}{V_s}$$

Where:

C_{ex} = The concentration of the compound in the extract.

V_{ex} = The extract volume in mL.

V_s = The sample volume in liters.

Y N N/A

Method Blanks

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

ACTION: 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 professional judgment to accept or reject positive sample data if no blank was run.

Were any PCB's found at greater than the method specified minimum level (Table 2) in the associated method blank?

ACTION: All associated samples, which have that PCB reported as detectable at a concentration less than 10 times the blank value, the reported value is changed to non-detectable with an EMPC equal to the reported value of the affected analyte.

Rinsate Blank

One rinsate blank must be collected for each day?

Do any rinsate blanks show the presence of PCB's above the method specified minimum level (Table 2)?

ACTION: If any rinsate blank was found to be contaminated with any of the PCB's, document in narrative notes.

Labeled Compound Recoveries

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

Were internal standard recoveries within the method required limits (25-150%)?

If not, were samples reanalyzed?

ACTION: If the internal standard recovery was outside (25 -150%) flag with "J" all positive data.

Y N N/A

Labeled Injection Internal Standards

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

Internal standard areas for every sample and blank within the upper and lower limits of each associated continuing calibration? _____

Area upper limit = +100% of internal standard area.

Area lower limit = -50% of internal standard area.

Is the retention time of each internal standard within 10 seconds of the associated daily calibrations standard? _____

ACTION: If the internal standard area is outside the upper or lower limits flag all related positive and non-detect data with "J" regardless whether the labeled standard recoveries met specifications or not.

If extremely low area counts (<25%) are reported, flag all associated non-detect data as unusable (R) and the positive data "J."

If the retention time of the internal standard differs by more than 10 seconds from the daily calibration, use professional judgment 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.

Y N N/A

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

For every sample delivery group of 20 or fewer environmental samples there should be one MS/MSD pair analyzed. In order for the MS/MSD to be appropriate for use in qualifying or rejecting associated sample data, the spike level must be within the range of 25% to 400% of the reported sample concentration.

If recovery of the MS is out of the 60 to 140% acceptance range, qualify the value for the analyte in the sample used for the MS/MSD as estimated "J"; use professional judgment in association with signal to noise ratios and internal standard recoveries for the associated sample data to determine the effect on the quality of the associated sample data.

If the precision of the MS/MSD analyses is greater than 50% RPD, then qualify the value for that analyte in the sample used for the MS/MSD as estimated "J"; use professional judgment in association with signal to noise ratios and internal standard recoveries for the associated sample data to determine the effect on the quality of the associated sample data.

Field Duplicate Samples

For every batch of 20 samples or less collected there must be a sample designated as duplicate.

Results of the duplicate samples must agree within 70% 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.

Ongoing Precision and Recovery (OPR)

For every batch of 20 samples or less processed by the laboratory, there must be an OPR processed as well. Has this requirement been met?

Have the calculated percent recoveries for all CBs in the associated OPR met the acceptance limits provided in Table 6?

ACTION: If the laboratory failed to process an OPR sample with the associated field samples, qualify all target analyte results as estimated.

ACTION: If any CB OPR percent recoveries failed to meet the acceptance criteria listed in Table 6, qualify effected sample results as estimated.

As part of the data validation process, the following validation qualifiers and their meanings will be used:

- "U" - Non-Detect - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- "J" - Estimated Value - The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
- "NJ" - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- "UJ" - Estimated Non-Detect - 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 significant QA/QC problems the analysis is invalid and provides no information as to whether the analyte is present or not. Once the data are flagged with "R", any further review or consideration is unnecessary.

If no determination of the overall bias of a result qualified as estimated can be made, the result will be flagged with "J." If the data reviewer can determine the overall bias for sample data qualified as estimated, the data reviewer will qualify the sample result as either an estimated minimum value (JL) or an estimated maximum value (JH).

Data Validation SOP

HW-25, Rev. 2

Dioxin/Furans

SOP NO. 25
REVISION 2
SEPTEMBER 1999

**USEPA REGION II DATA VALIDATION SOP FOR EPA METHOD 1613, REVISION A
Tetra- through Octa-chlorinated Dioxins and Furans by Isotope Dilution (HRGC/HRMS)**

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ATTACHMENT A
Data Assessment

ATTACHMENT B
Data Rejection Summary

1.0 INTRODUCTION

This method is designed to meet the survey requirement of the USEPA ITD. The method is used to detect the Tetra- through octa- chlorinated dibenzo-p-dioxins and dibenzofurans associated with the Clean Water Act (CWA, as amended 1987); the Resource Conservation and Recovery Act (RCRA, as amended 1986) and the Compensation and Liability Act (as amended in 1986) and other dioxins and furan compounds amenable to this method.

The dioxins and furans may be determined in waters, soils, sludges and other matrices using this method. The method is based on EPA, industry, and academic methods.

2.0 APPLICABILITY

The attached Standard Operating Procedure (SOP) is applicable to polychlorinated dibenzodioxin and polychlorinated dibenzofuran (PCDD/PCDF) data obtained using EPA Method 1613A, Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by Isotope Dilution using High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), April 1990. Its scope is to facilitate the data validation process of the data reported by the contracting laboratory and to ensure that the data is being reviewed in a uniform manner. This SOP is based upon the quality control and quality assurance requirements specified in Method 1613A, April 1990.

3.0 **Responsibilities/Scope**

- 3.1 The reviewer must be knowledgeable of the analytical method and its QC Criteria.
- 3.2 The reviewer must complete the following:
 - 3.2.1 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.
 - 3.2.2 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. Example of Data Assessment format is provided in Attachment A.
 - 3.2.3 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. Example of Data Rejection Form is provided in Attachment B.
 - 3.2.4 Telephone Record Log - All Laboratory phone conversations must be documented on the Telephone Record Log Sheet. A photocopy of the Telephone Record Log is attached to the Data Assessment package.
 - 3.2.5 Paperwork - Upon completion of the review the following are to be maintained with the data package and returned to the authorized person :
 - a. completed data assessment checklist and narrative (original)
 - b. Two copies of the data assessment narrative (attach copies of the Rejection Summary Form at end)
 - c. Telephone record Log (original and copy)
 - d. Rejection Summary Form (original)
- 3.3 Rejection of Data - All values determined to be unacceptable on the Dioxin/Furan 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, PeCDD and PeCDF 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 on the Analysis Data Sheet must be made in red pencil.

4.0 Definitions

CALIBRATION SOLUTION: solutions containing known amounts of selected analytes, internal standards and recovery standards that are analyzed prior to sample analysis. The solutions are used to determine the ratio of the instrument response of the analytes to that of the appropriate internal standard and the internal standards to that of the recovery standards.

CALIBRATION VERIFICATION (VER): a mixture of known amounts of analytes that is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance and establish the retention time window for each homologue.

CLEAN-UP STANDARD: only one labeled analyte (2,3,7,8-TCDD) is added to all samples extracts prior to any Clean-up procedure. This standard is used to differentiate between losses of analytes or internal standards during extraction and losses that occur during the various Clean-up procedures.

CONGENER: elements of the same group in the periodic table.

DEFLECTIONS: bend or broadening of a peak

ESTIMATED DETECTION LIMIT (EDL): the concentration of a analyte required to produce a signal with peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8 substituted isomer for which the response of the quantitation and confirmation ions is less than 2.5 times the background level.

ESTIMATED MAXIMUM POSSIBLE CONCENTRATION (EMPC): the concentration of a given analyte that would produce a signal with a given area peak. The EMPC is calculated for each 2,3,7,8 substituted isomer for which the response of the quantitation and/or confirmation ions has signal to noise in excess of 2.5 times the background level but does not meet identification criteria.

FIELD CHAIN OF CUSTODY: see Traffic Report

GEL PERMEATION CHROMATOGRAPHY (GPC): removes many high molecular weight interferences that cause GC column performance to degrade. It may be used for all soil and sediment extracts and may be used for water extracts that are expected to contain high molecular weight organic compounds.

HOMOLOGUE: a member or members of a particular homologous series that has the same molecular weight but not necessarily the same structural arrangement. For example, the 28 pentachlorinated dibenzofurans are homologues.

HRGC/HRMS: high resolution gas chromatography/ high resolution mass spectrometry.

HPLC: high performance liquid chromatography

INITIAL CALIBRATION STANDARD SOLUTION (CS1-CS5): analysis of analytical standards for a series of different specified concentrations. The initial calibration is used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INITIAL PRECISION AND RECOVERY (IPR): must be performed by the laboratory to establish the ability to generate acceptable precision and accuracy. The recoveries of the labeled analytes must be within 25 to 150 % recovery. The standard deviation (s) of the concentration and the average concentration (x) for each unlabeled analyte must be within range established by the Method (Table 7).

INTEGRATED ION CURRENT: electronic output to computer from instrument to provide a hard copy of area and height of a peak that

may or may not be an analyte of interest.

INTERNAL STANDARDS (IS): labeled analytes are added to every sample and are present at the same concentration in every blank, quality control sample, and calibration solution. The IS are added to the sample before extraction and are used to measure the concentration of the analytes. In Method 1613A, the ISs are $^{13}\text{C}_{12}$ -1,2,3,4-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD.

ION ABUNDANCE RATIO: mathematical comparison of selected pair of ions stipulated by the method for each target analyte. The ratio between each pair of ions must fall within established limits. These ions are needed for the identification and quantitation of target analytes.

ISOMER: chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.

LABELED ANALYTE (or analog): an analyte that has isotopically carbon added to its chemical structure. These compounds are used to establish identification (retention time) and used for quantitation of unlabeled analytes.

MASS/CHARGE: usually expressed as m/z.

METHOD BLANK (MB): an analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The MB is used to define the level of laboratory background contamination.

MAXIMUM CONCENTRATION LEVEL (MCL): Highest level of concentration for each analyte depending upon upper concentration of analyte. Usually used to determine upper level of the concentration range.

NON-CONGENER: elements not from the same group in the periodic table.

NON-2,3,7,8 SUBSTITUTED ANALYTES: analytes whose structure have positions other than 2,3,7,8.

ONGOING PRECISION AND RECOVERY (OPR): must be performed by the laboratory to establish the ability to maintain on a continuous basis, acceptable precision and accuracy. The recoveries of the labeled analytes must be within 25 to 150 % recovery. The standard deviation (s) of the concentration and the average concentration (x) for each unlabeled analyte must be within range established by the Method (Table 7).

PERCENT MOISTURE: an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at this degree including water. %M is determined from decanted samples and from samples that are not decanted.

PERCENT VALLEY: see Resolution

PERFLUOROKEROSENE (PFK): compound used to establish mass spectral instrument performance for dioxin/furan analysis.

PERFORMANCE EVALUATION MIXTURE (PEM): See Performance Evaluation (PE) Sample,

PERFORMANCE EVALUATION (PE) SAMPLE: a chemical waste, soil or water sample containing known amounts of unlabeled PCDDs/PCDFs used for Quality Assurance programs. There are 3 types of PE's available. PEM Blank which consists of uncontaminated soil and used to monitor possible crossover contamination of samples in the field and laboratory. PEM Interference Fortified Blank which is a soil containing matrix interference and spiked by the laboratory with target compounds. A PEM sample(s) is a soil sample containing known amounts of unlabeled TCDD or a mixture of TCDD and other PCDD/PCDF isomers. These PEMs are used to monitor the laboratory's performance.

POLYCHLORINATED DIBENZO-P-DIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs): compounds that contain from one to eight chlorine atoms.

PCDPE: Polychlorinated Diphenylether; isomers having the same SICP and ion ratios identical to furan isomers and are monitored for interference in furan qualitative and quantitative analysis.

PRECISION AND RECOVERY (PAR) Standard: this is a stock solution containing unlabeled analytes and diluted to prepare spiking solution used for Initial Precision and Recovery (IPR) and Ongoing Precision and Recovery (OPR). This Quality Assurance program must be performed by the laboratory to establish the ability to generate acceptable precision and accuracy. The recoveries of the labeled analytes must be within 25 to 150 % recovery. The standard deviation (s) of the concentration and the average concentration (x) for each unlabeled analyte must be within range established by the Method (Table 7).

RECOVERY: a determination of the accuracy of the analytical procedure made by comparing measured values from a fortified (spiked) sample against the known spiked values. Recovery is determined by the following equation:

$$\% \text{ Recovery} = \frac{\text{measured value}}{\text{known value}} \times 100\%$$

RELATIVE RETENTION TIME (RRT): ratio of the retention time of the analyte versus the retention time of the corresponding internal standard. RRT for each analyte must be within range established by the method.

RELATIVE RESPONSE (RR): the ratio of the area response of the mass spectrometer to a known amount of an analyte (unlabeled to labeled) versus a known concentration in standard solution, plotted using linear regression. The RR is used to determine instrument performance and is used in the quantitation calculations. RR are calculated using the following equation:

$$RR = \frac{(A_n^1 + A_n^2) C_l}{(A_l^1 + A_l^2) C_n}$$

$A_n^1 + A_n^2$ are the areas of the primary and secondary m/z's for the unlabeled compound.

$A_l^1 + A_l^2$ are the areas of the primary and secondary m/z's for the labeled compound.

C_l is the concentration of the labeled compound in the calibration standard.

C_n is the concentration of the unlabeled compound in the calibration standard.

RESPONSE FACTOR (RF): the ratio of the response of the mass spectrometer to a known amount of an analyte relative to that of a known amount of internal standard as measured in the initial and continuing calibrations. The RF is used to determine instrument performance using correlation coefficient and is used in the quantitation calculations. RF are calculated using the following equation:

$$RF = \frac{(A_l^1 + A_l^2) C_{is}}{(A_n^1 + A_n^2) C_s}$$

- $A_p^1 + A_p^2$ are the areas of the primary and secondary m/z's for the compound to be calibrated.
- $A_{is}^1 + A_{is}^2$ are the areas of the primary and secondary m/z's for the internal standard.
- C_s is the concentration of the compound in the calibration standard.
- C_{is} is the concentration of the internal standard.

RESOLUTION: the separation between peaks on a chromatogram. Resolution is calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RINSATE: a portion of the solvent that is used to rinse sampling equipment. The rinsate is later analyzed to demonstrate that samples were not contaminated during collection.

SAMPLE DELIVERY GROUP (SDG): a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer samples within a case, received over a period of time up to 14 calendar days. Data from all samples in a SDG are due concurrently. A SDG is defined by one of the following, whichever occurs first:

- Case; or
- each 20 samples within a case; or
- each 14 day calendar period during which samples in a case are received, beginning with receipt of the first sample in the case or SDG.

SELECTED ION MONITORING (SIM): a mass spectrometric technique whereby ions with predetermined mass/charge ratios (m/z) are monitored, as opposed to scanning MS procedures in which all m/z's between two limits are monitored.

SICP: a plot of ion abundance versus time for each ion which provides the retention time, peak area and height. This information is used for identification and quantitation of target analyte.

SIGNAL TO NOISE (S/N) RATIO: the ratio of analyte signal to random background signal. To determine the ratio, display each characteristic ion using a window 100 scans wide, and draw a base line from the lowest point in the 100 scan window. The noise is defined as the height of the largest signal (excluding signal due to PCDDs/PCDFs or other chemicals) within the 100 scan window. The signal is defined as the height of the PCDD/PCDF peak. If the data system determines the ratio, the Contractor shall demonstrate comparability between the above criteria and the automated S/N determination. Chemical noise is left to the judgement of the analyst.

2,3,7,8 SUBSTITUTED ANALYTES: analytes whose structure has other positions as well as the 2,3,7,8 positions.

TOXICITY EQUIVALENCY FACTOR (TEF): a method of converting concentrations of PCDDs/PCDFs to an equivalent concentration of 2,3,7,8-TCDD to obtain an estimation of the toxicity of the entire sample. The concentrations can be found on Form I PCDD-2 in the DFLM01.1 Statement of Work for Dioxin Analysis.

TRAFFIC REPORT (TR): (may also be called Field Chain of Custody), a sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and documents sample condition and receipt by the laboratory.

TWELVE HOUR TIME PERIOD: the 12 hour time period begins with the injection of the CC3 solution on the DB-5 (or equivalent) column or the injection of the column performance solution on the SP-2331 (or equivalent) column. The 12 hour period continues until 12:00 hours have elapsed according to the system clock. To be included in a given 12-hour time period, a sample or standard must be

injected with 12:00 hours of the CC3 solution or the column performance solution.

UNLABEL ANALYTE: target compound that has not been isotopically altered.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR): the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample traffic report.

WINDOW DEFINING MIXTURE (WDM): a mixture containing the first and last eluting isomer for each congener. The retention time for each first and last eluting isomer establishes the retention time window for each congener. All analytes in the standards (calibrations, internal standards, recovery standards, Clean-up standard) and identified analytes in samples must have a reported retention time within the established window. It is analyzed before any calibration standard, at the beginning of each 12 hour time period or when there is a shift greater than 10 seconds between retention time of recovery standards in standards or any analysis from retention time in recent calibration verification.

N/A

YES NO

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____

LAB: _____

SITE: _____

1.0 Data Completeness and Deliverables

- | | | | | |
|-----|---|--------------------------|--------------------------|-----|
| 1.1 | Does the Traffic Report or Field Chain of Custody list all samples? | ☐ | ___ | ___ |
| 1.2 | Is the Case Narrative present? | ☐ | ___ | ___ |
| 1.3 | Are the Case Number and SDG numbers contained in the case narrative? | ☐ | ___ | ___ |
| 1.4 | Do the Traffic Reports, Field Chain of Custody 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 Case number and the associated sample/traffic number. Missing or illegible or incorrectly labeled items must be identified. The Project Officer must immediately be contacted and requested to ask laboratory to submit the missing or incorrect items.
- 2.2 The following forms were taken from the CLP SOW, DFLM01.1 and should be specified in the Project Plan. Laboratories will not always use the exact CLP format for the forms. A comparison of CLP forms must be made against the Laboratory's version. Some information may not be found on the exact form as the CLP version but may be located on another form. As long as the information is present and accessible, it is not a problem. Are these forms (CLP or lab's version) 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) | ☐ | ___ | ___ |
| ___ | | | |
| 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) | ☐ | ___ | ___ |

		YES	NO
N/A			
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, PCDD-2)		☐	☐
ACTION:	If forms are missing, contact the Project Officer to confirm which forms if any were specified in the Project Plan. If the forms are required, inform the Project Officer or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.		
2.3	GC/MS Displays		
	Are the following GC/MS displays present?		
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.	Window Defining Mixture raw data	☐	☐
d.	SIM mass chromatograms must display quantitation ion, confirmation ion, and polychlorinated diphenylether ion, where applicable.	☐	☐
e.	Integrated area and peak height must be listed for all peaks 2.5 times above background	☐	☐
ACTION:	If deliverables are missing, contact the Project Officer to request explanation/resubmittals or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.		
2.4	Are the following Chain of Custody Records and in-house Laboratory Control Documents present?		
a.	Chain of Custody Records	☐	☐
b.	Sample Shipment Records	☐	☐
c.	Sample log-in sheets	☐	☐
d.	GC/MS Standard and Sample Run Log in chronological order	☐	☐

N/A

YES NO

- e. Sample Extraction Log ☐ ☐ ☐

ACTION: If deliverables are missing, contact the Project Officer to request explanation/resubmittals or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.

- 2.5 Was the sample data package paginated and one sided? ☐ ☐ ☐

ACTION: If no, document difficulties of reviewing data caused by lack of pagination in Data Assessment.

3.0 Holding Times

- 3.1 Have any of the following 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.

Note: All samples must be stored in dark at 4°C.

Note: **Extraction** holding times listed are recommendations. PCDDs and PCDFs are very stable in a variety of matrices. Holding times may be as high as a year for certain matrices. Sample extracts must be analyzed within 40 days of extraction.

4.0 Instrument Performance

- 4.1 Mass Calibration - Mass calibration of the MS must be performed prior to analyzing calibration solutions, blanks, samples, and QC samples. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12 hour period of operation. Include in the narrative, minimum required resolving power of 10000 was obtained for perfluorokerosene (PFK) ion 380.9760. This is done by first measuring peak width at 5% of the maximum. This should not exceed 100 ppm, i.e., it should not exceed 0.038, for ion 380.9760. Resolving power, then is calculated using the formula,

$$\text{Resolving Power} = m/\Delta m = 380.9760/0.038 = 10025.$$

NOTE: The mass calibration is generally not reported. Improper mass calibration may be detected by examining ion abundance ratios for initial and continuing calibration standards. If the mass calibration is not properly performed, the standards will not have ion abundance ratios within criteria.

N/A

YES NO

4.2 Window Defining Mixture/ Isomer Specificity Test Standards

The Window Defining Mixture must contain the first and the last isomers of each homologue PCDD/PCDF, (the labeled and internal standards are optional). The solution also should contain a series of other TCDD analytes for the purpose of documenting the chromatographic resolution.

- 4.2.1 For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of Isomer Specificity Test Standards at the beginning of every 12 hour period. Was this performed accordingly? ☐ ☐ ☐

ACTION: If the Isomer Specificity Test Standards was not analyzed at the required frequency, use professional judgement to determine the effect on the quality of the data. Document in Data Assessment under contract non-compliance.

- 4.2.2 Were all peaks labeled and identified on the Selected Ion Current Profiles (SICPs)? ☐ ☐ ☐

- 4.2.3 Did the absolute retention time of the internal standards ¹³C₁₂-1,2,3,4-TCDD exceed 25.0 minutes on the DB-5 column and 15.0 minutes on the DB-225 column? (Method 1613A, Section 7.2.4) ☐ ☐ ☐

- 4.2.4 Are the relative retention times of native and labeled PCDD's and PCDF's within the limits given in Table 2 of the method. (Method 1613A, Section 14.4.1.2) ☐ ☐ ☐

ACTION: If no for sections 4.2.2, 4.2.3 and 4.2.4, assess the effect on the validity of the data. Note in the Data Assessment.

- 4.2.5 For DB-5 or equivalent, (Method 1613A, Section 14.4.2.24) the peak separation between the unlabeled 2,3,7,8-TCDD and the peaks representing any other TCDD analyte shall be resolved with a valley of ≤ 25 percent. Was this criteria met? ☐ ☐ ☐

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

Y = The peak height of 2,3,7,8-TCDD analyte

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.

- 4.2.6 Is the last eluting tetra chlorinated congener (1,2,8,9-TCDD) and the first eluting penta chlorinated congener (1,3,4,6,8-PeCDF) separated properly, since they elute within 15 seconds of each other? ☐ ☐ ☐

ACTION: If one of the congener is missing, report that in the Data Assessment.

5.0 Initial 5-Point Calibration

N/A

YES NO

The initial calibration standard solutions (CS1-CS5) must be analyzed prior to any sample analysis. However, initial calibration should be analyzed when the CS3 Calibration Verification (VER) or Isomer Specificity Test Standard do not meet performance criteria. The initial calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze the Window Defining Mixture and the Isomer Specificity Test Standards.

Was the initial calibration performed at the frequency specified above?

☐ ☐ ☐

5.1 The method allows the Laboratory to perform quantitative analysis by isotope dilution and internal standard, or to combine calibration solutions.

1. Isotope Dilution: performed for the fifteen 2,3,7,8-substituted PCDDs and PCDFs unlabeled analytes with labeled analytes added to the samples prior to extraction and for 1,2,3,7,8,9- HXCDD and OCDF (see sections 5.2.8 and 5.2.9). The relative response (RR) is calculated and the percent coefficient of variation must be < 20% over the 5 point range to use the average response factor for quantitation, otherwise a calibration curve must be used.
2. Calibration by Internal Standard: performed for non-2,3,7,8 substituted compounds having no labeled analytes in this method and for measurement of labeled compounds for intra laboratory statistics. The response factor (RF) is calculated and the percent coefficient of variation must be <35% over the 5 point range to use the average response factor for quantitation, otherwise a calibration curve must be used.
3. Combined Calibration: performed by using solutions containing unlabeled, labeled compounds and internal standards. The requirements of each of the above methods are used. This method allows the laboratory to produce a single set of curves for isotope dilution and internal standard method.

5.1.1 The following MS/DS conditions must be used:

5.1.1.1 Mass calibration as per Section 4.1?

☐ ☐ ☐

5.1.1.2 Were SIM data acquired for each of the ions listed in Table 3, including interfering ions? (see analytical method)

☐ ☐ ☐

5.2 Were the following GC criteria met?

5.2.1 The chromatographic resolution between the 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of ≤ 25 percent on the primary analysis (DB-5) column (see sec. 14.4.2.2/ Pg. 29 of the method).

☐ ☐ ☐

5.2.2 The chromatographic resolution between the 2,3,7,8-TCDF and the peaks representing any other unlabeled TCDF isomers must be resolved with a valley of ≤ 25 percent on the confirmation (DB-225 or SP2330) analysis column.

☐ ☐ ☐

5.2.3 For all calibration solutions, the relative retention time of peaks representing an unlabeled 2,3,7,8- substituted PCDD or PCDF must be within the limits given in table 2 of the Method. The retention times of the peaks representing non-2,3,7,8- substituted PCDD or PCDF's must

		YES	NO
N/A	fall within the retention time windows established by the Window Defining Mixture. In addition, the absolute retention times of internal standards, $^{13}\text{C}_{12}$ 1,2,3,4-TCDD and $^{13}\text{C}_{12}$ 1,2,3,7,8,9-HxCDD shall not change by more than 15 seconds between the CS3 analysis and the analysis of any other standard.	☐	☐
5.2.4	The two SIM ions for each homolog must maximize simultaneously and within 2 seconds of the corresponding labeled analyte ions.	☐	☐
5.2.5	The relative ion abundance criteria for PCDDs/PCDFs listed in Table 3A (see analytical method) must be met.	☐	☐
5.2.6	For all calibration solutions the signal to noise ratio (S/N) for the GC signal present in every SSCP, including the ones for the labeled standards must be ≥ 10 .	☐	☐
5.2.7	The percent relative standard deviations (% RSD) for the mean response factors (RRF) from the 17 unlabeled standards must be $\leq 20\%$, and those for the 15 labeled reference compounds must be $\leq 35\%$.	☐	☐
5.2.8	Labeled analyte 1,2,3,7,8,9-HxCDD is used as an internal standard in this method, and can not be used to quantitate corresponding unlabeled analyte. The unlabeled 1,2,3,7,8,9-HxCDD must be quantitated using the average of the responses of the labeled analytes of 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD. The concentration of the unlabeled 1,2,3,7,8,9-HxCDD is corrected for the average recovery of the other HxCDD's. Was the unlabeled 1,2,3,7,8,9-HxCDD quantitated correctly?	☐	☐
5.2.9	The labeled analog of OCDF is not added to the sample because of a potential interference. Unlabeled OCDF is quantitated against the labeled OCDD. The concentration of the unlabeled OCDF is corrected for the recovery of the labeled OCDD. Was the unlabeled OCDF correctly quantitated against the labeled OCDD.	☐	☐

ACTION:

1. If mass calibration criteria as specified in Section 4.1 was not met, note in Data Assessment.
2. If the selected monitoring ions specified in Table 3 were not used for data acquisition, the lab must be contacted by the Project Officer for an explanation. If an incorrect ion was used, reject "R" all the associated data.
3. If the 25% percent valley for TCDD requirement was not met, quality positive data "J". Do not qualify non-detects. The tetra and penta (dioxins and furans) are affected. Heptas, Hexas and Octas are not affected.
4. If the ion abundance ratio for an analyte is outside the limits, flag the results for that analyte "R" (reject).
5. If the ion abundance ratio for an internal or labeled standard falls outside the QC limits flag the associated positive hits with "J". No effect on the non-detects.

N/A

YES NO

6. If the signal to noise ratio (S/N) is below control limits, use professional judgement to determine quality of the data.
7. If the %RSD for each unlabeled analyte exceeds 20%, or the %RSD for each labeled analyte exceeds 35%, flag the associated sample positive results for that specific analyte as estimated ("J"). No effect on the non-detect data.
8. If 1,2,3,7,8,9-HxCDD was not calculated using the correct HxCDD response (average) factor, either manually recalculate the values for all standards and samples or contact Project Officer to request resubmittals from the laboratory.
9. If OCDF was not calculated using the correct response factor (OCDD), either manually recalculate the values for all standards and data or contact Project Officer to request resubmittals from the laboratory.
10. Non compliance of any other criteria specified above should be evaluated using professional judgement.

5.2.10 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled PCDDs/PCDFs and labeled standards were used. In addition, verify that the appropriate labeled standard was used for each analyte.

To recalculate the response factor, use the equation:

For target compounds (unlabeled analytes with corresponding labeled analytes):

$$RR = \frac{(A_{n1} + A_{n2}) \times Q_i}{(A_{i1} + A_{i2}) \times Q_n}$$

For labeled analytes, Internal standards and cleanup standard listed in Table 6 of method 1613:

$$RF = \frac{(A_{i1} + A_{i2}) \times Q_n}{(A_{is1} + A_{is2}) \times Q_i}$$

Note: There is only one m/z for ³⁷Cl₄2,3,7,8-TCDD.

A_{n1} + A_{n2} = integrated areas of the two quantitation ions of analytes of interest. (Target analyte, unlabeled compounds)

A_{i1} + A_{i2} = integrated areas of the two quantitation ions of the appropriate labeled analytes compound.

A_{is1} + A_{is2} = integrated areas of the two quantitation ions of the appropriate internal standard.

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected [pg]

Q_i = quantity of the appropriate labeled analytes compound [pg]

Q_{is} = quantity of the appropriate internal standard injected [pg]

ACTION: 1. If calculations were not performed correctly, notify the Project Officer to initiate

N/A

YES NO

resubmittals from the laboratory.

6.0 System and Laboratory Performance
(Calibration Verification and Isomer Specificity Test Standard)

At the beginning of a 12 hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all unlabeled and labeled compounds. For these tests the calibration verification (VER) standard and the isomer specificity test standards shall be used to verify all performance criteria.

Only if the laboratory meets all performance criteria may samples, blanks, and precision and recovery standards be analyzed.

6.1 Calibration Verification

- 6.1.1 Was the relative ion abundance for PCDDs/PCDFs listed in Table 3A of the analytical method met? (Method 1613A, Section 14.3.2) ☐ ☐ ☐
- 6.1.2 Were the peaks representing each unlabeled and labeled compound in the verification standard present with signal to noise ratio (S/N) of ≥ 10 ? (Method 1613A, Section 14.3.3) ☐ ☐ ☐
- 6.1.3 For each compound, was the concentration within the limit in Table 7 of the method? (Method 1613A, Section 14.3.5) ☐ ☐ ☐
- 6.1.4 Were the absolute retention time of the internal standards $^{13}\text{C}_{12}$ -1,2,3,4- TCDD and $^{13}\text{C}_{12}$ 1,2,3,7,8,9- HxCDD within ± 15 seconds of the retention times obtained during calibration? (Method 1613A, Section 14.4.1.1) ☐ ☐ ☐
- 6.1.5 Were the relative retention times of the unlabeled and labeled PCDDs and PCDFs within the limits given by Table 2 of the method? (Method 1613A, Section 14.4.2.2) ☐ ☐ ☐

6.2 Isomer Specificity Test Standard

- 6.2.1 Was the chromatographic resolution between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers resolved with a valley of ≤ 25 percent on the primary analysis (DB-5) column? (Method 1613A, Section 14.4.2.2) ☐ ☐ ☐
- 6.2 Was the chromatographic resolution between 2,3,7,8- TCDF and the peaks representing any other unlabeled TCDF isomers resolved with a valley of ≤ 25 percent on the confirmation (DB-225 or SP2330) analysis ☐ ☐ ☐

ACTION:

1. If the ion abundance ratio for an analyte is outside the limits, flag the results for that analyte "R" (reject).
2. If the signal noise ratio (S/N) is below control limits, use professional judgement to determine the quality of the data.

N/A

YES NO

3. If an analyte concentration fell outside the acceptance criteria listed in Table 7 of the method.
 - A. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte exceeds the range, flag the associated sample positive results for that specific analyte as estimated ("J"). No effect on the non-detect data.
 - B. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte is below the range, flag the associated sample positive results as well as non-detects for that specific analyte as estimated ("J").
 - C. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte is excessively below, $\leq 10\%$ of the range, at the minimum, flag the associated sample positive results as well as non-detects for that specific analyte as estimated ("J"). However the validator may use professional judgement to accept or reject positive data and non-detects.
 4. If the 25 percent valley for TCDD and TCDF requirement was not met, qualify positive data "J". Do not qualify non-detects. The tetras and pentas (dioxin and furans) are affected. Heptas, Hexas and Octas are not affected.
 5. Non compliance of any other criteria specified above, in the method should be evaluated using professional judgement.
- 6.3 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled PCDDs/PCDFs and labeled standards were used. In addition, verify the appropriate labeled standard was used for each analyte.

7.0 Sample Data

NOTE: Any qualifications such as "J" applied to target compounds should be also applied to their associated total congeners concentration column.

7.1 Were the following MS/DS conditions used?

7.1.1 SIM data were acquired for each of the ions listed in Table 3 (see analytical method) including diphenylether interfering ions.

☐ ☐ ☐

7.2 Were the following identification criteria met?

7.2.1 For the 2,3,7,8 substituted analytes found present and the corresponding labeled compound or internal standard in the sample extract, must show relative retention times at the peak height within the limits given in Table 2. (Method 1613A, Section 15.4)

☐ ☐ ☐

7.2.2 For non-2,3,7,8 substituted compounds (tetra through octa) found present, the retention time must be within the window established by the Window Defining Mixture, for the corresponding homologue. (Method 1613A, Section 15.4)

☐ ☐ ☐

7.2.3 All specified ions listed in Table 3 for each isomer found present and the associated labeled compounds must be present in the SICP. The two SIM ions for the analyte, the labeled compound, and the internal standard must maximize simultaneously. (± 2 sec.)

		YES	NO
N/A	(Method 1613A, Section 15.1)	☐	☐
7.2.4	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 not have saturated the detector. (Method 1613A, Section 15.2)	☐	☐
7.2.5	The integrated ion current for the labeled compounds, internal standards, and cleanup standard characteristic ions must be at least 10 times background noise. (Method 1613A, Section 15.2)	☐	☐
7.2.6	The relative ion abundance criteria for all PCDDs/PCDFs found present must be met. (Table 3A - Method 1613A, Section 15.3)	☐	☐
7.2.7	The relative retention time of the unlabeled 2,3,7,8-substituted PCDD or PCDF must be within the limits given in Table 2 (Method 1613A).	☐	☐
7.2.8	The relative ion abundance criteria for the labeled compounds, cleanup, and internal standard must be met (Table 3A - Method 1613A).	☐	☐
7.2.9	The analyte concentration must be within the calibration range. If not, dilution should have been made to bring the concentration within the calibration range. Was this criterion met?	☐	☐
NOTE: The analytical method clearly states that samples containing analytes having concentrations higher than 10 times the upper MCLs should be analyzed using a less sensitive, high resolution GC/low resolution MS method.			
7.2.10	The identification of a GC peak as a PCDF can only be made if no signal having a S/N ≥ 2.5 is detected at the same time in the corresponding polychlorinated diphenylether (PCDPE) channel. Was the above condition met?	☐	☐
ACTION:			
1. If the selected monitoring ions specified in Table 3 were not used for data acquisition, the lab must be contacted by the Project Officer for an explanation. If an incorrect ion was used, reject "R" all the associated data.			
2. If the retention time of an analyte falls outside the retention time windows established by the associated Window Defining Mixture take the following action:			
A. If the analyte has a corresponding labeled analyte and is within 2 seconds of the labeled analyte, no action taken on positive data or non-detects.			
B. If the analyte has a corresponding labeled analyte and is outside 2 seconds of the labeled analyte, use professional judgement to determine qualifications for positive data or non-detects. At a minimum, "J" or "JN" positive data.			
C. If the analyte does not have a corresponding labeled analyte and is outside 2 seconds of the matching unlabeled analyte from the associated calibration, use professional judgement to determine qualifications for positive data or non-detects. At a minimum, "J" or "JN" positive data.			

N/A

YES NO

- D. If analyte meets identification criteria (7.2.2, 7.2.4, 7.2.5, 7.2.7) but does not meet ion abundance ratio criteria (7.2.8) and is not a labeled analog, the sample must be reanalyzed on a confirmation column. If confirmation analysis was not performed, reject the failing analyte.
3. If the criteria listed in section 7.2.4 and 7.2.5 are not met but all other criteria are met, qualify all positive data of the specific analyte with "J".
 4. If the analytes reported positive do not meet criteria for section 7.2.6, reject (R) all positive data for these analytes. Change the positive values to EMPC (Estimated Maximum Possible Concentration). Flag "J"
 5. If the labeled compounds, internal standards and cleanup standards do not meet ion abundance criteria section 7.2.6. and 7.2.7. (Table 3 - analytical method) but they meet all other criteria, flag all corresponding data with "J".
 6. If the lab reported values exceeding the calibration range flag those values with "J".
 7. If peak deflections >50% are visible qualify particular compound with "J".
 8. If PCDF was detected but an interfering PCDPE was also detected (see Section 7.2.9) and concentration not corrected for the interference, cross out the PCDF data. The reported value of PCDF is changed to EMPC.
 9. If the lab did not monitor for PCDPEs, qualify all positive furan data "JN".
- 7.2.10 Spot check calculations for positive data and verify that the same labeled compounds used to calculate RFs were used to calculate concentration and EMPC. Ensure that the proper PCDDs/PCDFs and labeled compounds were used.

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

All Matrices other than water

$$C_n (\text{pg/g}) = \frac{(A_{n1} + A_{n2}) \times Q_i}{W \times (A_{i1} + A_{i2}) \times RR}$$

Water

$$C_n (\text{pg/L}) = \frac{(A_{n1} + A_{n2}) \times Q_i}{V \times (A_{i1} + A_{i2}) \times RR}$$

Where:

$A_{n1} + A_{n2}$ = integrated areas of the two quantitation ions of analyte of interest. (Target analyte)

$A_{i1} + A_{i2}$ = integrated areas of the two quantitation ions of the appropriate labeled analyte compound.

W = Weight (g) of sample extracted

V = Volume (L) of sample extracted

N/A

YES NO

Q_i = Quantity (pg) of the appropriate labeled compound added to the sample prior to extraction.

RR = Calculated relative response from initial calibration. (see section 5.2.10)

ACTION: If the spot check calculations yielded positive hit concentrations with $\leq 15\%$ Difference from those reported in Form I, correct manually. If the difference between the validator's value and the form I's values are $> 15\%$ contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.

7.3 Clean-up procedures

Clean-up may not be necessary for relatively clean samples (drinking waters, ground waters etc). If the matrix required clean-up, the laboratory has 4 different procedures to choose from. Before using any clean-up procedure, the laboratory must demonstrate that the Initial Precision and Recovery requirements of the method can be met using the clean-up procedure.

A labeled clean-up standard $^{37}\text{Cl}_4,2,3,7,8\text{-TCDD}$ is added to the sample just before the back extraction with base and acid procedure. This occurs before any recommended clean-up procedures are initiated.

7.3.1 Was the percent recovery of the clean-up standard within the recommended range of 25-150% for each sample? ☐ ☐ ☐

ACTION: If no, and the recovery is less than 25%, qualify all data as estimated "J". If recovery is 0 %, qualify all positive data as estimated "J" and reject "R" all non-detects for that sample.

7.3.2 Check the chromatograms that clean-up procedure was needed for each sample. Were any clean-up procedures needed for either water or soil samples? ☐ ☐ ☐

ACTION: If yes, check extraction log to verify which clean-up procedures if any were performed. The laboratory is not limited to only one procedure.

1. If no clean-up was performed and the chromatograms indicated that some should have been performed. Use professional judgement to assess the effect on the interference on the validity of the data. Document lack of required clean-up for complex samples in Data Assessment.
2. If one type of clean-up was performed, but the chromatograms indicate that additional clean-up should have been utilized. Use professional judgement to assess the effect on the interference on the validity of the data. Document lack of additional clean-up for complex samples in Data Assessment.

7.3.3 If clean-up procedures were used, did the Laboratory perform clean-up procedures on the Initial Precision and Recovery samples as required by the method? ☐ ☐ ☐

ACTION: If no, Use professional judgement to assess the effect of the interference on the validity of the data. Document lack of IPR documentation for clean-up procedures in Data Assessment.

8.0 Estimated Detection Limits (EDL) If required for the project

8.1 Was an EDL calculated for each 2,3,7,8-substituted analyte that was not identified regardless

N/A

YES NO

of whether other non-2,3,7,8 substituted analytes were present?

☐ ☐ ☐

ACTION: 1. If EDL or EMPC of an analyte which was not reported as a positive hit is missing, correct manually or contact the Project Officer to request from the laboratory corrections.

8.2 Use the equation below to check EDL calculations:

ALL MATRICES OTHER THAN WATER

$$\text{EDL (pg/g)} = \frac{2.5 \times Q_{is} \times (H_{x^1} + H_{x^2}) \times D}{W \times (H_{is^1} + H_{is^2}) \times RR}$$

WATER

$$\text{EDL (pg/L)} = \frac{2.5 \times Q_{is} \times (H_{x^1} + H_{x^2}) \times D}{V \times (H_{is^1} + H_{is^2}) \times RR}$$

Where:

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

H_{is^1} and H_{is^2} = peak heights of both the quantitation ions of the appropriate internal standards.

D = dilution factor

Q_{is} , RR, W and V are previously defined.

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 Project Officer to request recalculations from the laboratory.

ACTION: If the spot check calculations yielded EDLs or EMPCs with $\leq 15\%$ Difference from those reported in Form I, correct manually. If the difference between the validator's value and the Form I's values are $> 15\%$ contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.

9.0 Estimated Maximum Possible Concentration (EMPC) If required for the project

9.1 Was an EMPC calculated for 2,3,7,8-substituted analytes that had S/N ratio for the quantitation and confirmation ions greater than 2.5, but did not meet all the identification criteria? ☐ ☐ ☐

9.2 Use the equation below to check EMPC calculations:

All Matrices other than water

$$\text{EMPC (pg/g)} = \frac{(A_{n1} + A_{n2}) \times Q \times D}{W}$$

N/A YES NO

Water: $W \times (A_{11} + A_{12}) \times RR$

$$EMPC \text{ (pg/L)} = \frac{(A_{n1} + A_{n2}) \times Q \times D}{V \times (A_{11} + A_{12}) \times RR}$$

- Action: 1. If EDL or EMPC of an analyte which was not reported as a positive hit is missing, correct manually or contact the Project Officer to request from the laboratory corrections.
2. If the spot check calculations yielded EDLs or EMPCs with $\leq 15\%$ Difference from those reported in Form I, correct manually. If the difference between the validator's value and the Form I's values are $> 15\%$ contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.
3. If EDLs or EMPCs for the most toxic analytes ($TEF \geq 0.05$) are above reporting limits, contact the project office to recommend sample reanalysis.

10.0 Method Blanks

- 10.1 Has a method blank per matrix been extracted and analyzed with each batch of 20 samples? [] _ _
- 10.2 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? [] _ _
- 10.3 Acceptable method blanks must not contain any signal of 2,3,7,8-TCDD, or 2,3,7,8-TCDF, equivalent to a minimum levels listed in Table 2 ($> 1 \text{ ng/Kg}$ for soils, and 10 pg/L for water). Was this criteria met? (Method 1613A, Section 8.5.2) [] _ _
- 10.4 For other 2,3,7,8- substituted PCDD/PCDF isomers of each homologue, the allowable concentration in the method blank is less than minimum level listed in Table 2 ($< 5 \text{ ng/Kg}$ for soils and 50 pg/L for waters). Was this criteria met? [] _ _

- ACTION: 1. If the proper number of method blanks were not analyzed, document in Data Assessment. If the validator feels that the validity of the data is seriously compromised and validation of data without the method blanks would be flawed then notify the Project Officer. If decision is made to proceed with the validation process, consider the following actions: no action taken on non-detected analytes. If an analyte has a reported concentration that is > 5 times the EDL, qualify "J" and all concentrations ≤ 5 times the EDL are qualified "R" due to possibility of contamination.
2. If the method blank is contaminated with 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDD, 1,2,3,7,8-PeCDF or 2,3,4,7,8-PeCDF at a concentration higher than the minimum levels in Table 2, reject all contaminant compound positive data for the associated samples "R" and notify the Project Officer to initiate reanalysis.
3. A. If the method blank is contaminated with any of the analytes mentioned in Action # 2 at a concentration of less than the minimum levels in Table 2 specified in the method or of any other 2,3,7,8-substituted analytes at any concentration and the concentration in the sample is less than five times the concentration in the blank,

N/A

YES NO

transfer the sample results to the EMPC/EDL column and cross-out the value in the concentration column in order to present the data as a non-detect.

- B. If the concentration in the sample is higher than five times the contamination concentration in the blank, no action is needed.

11.0 Labeled Compound Recoveries

- 11.1 Were the samples spiked with all the labeled compounds as specified in the method? ☐ ☐ ☐
- 11.2 Have labeled compounds' recoveries been within the required (25 - 150%) limits? ☐ ☐ ☐
- 11.3 If not, were samples reanalyzed? ☐ ☐ ☐

- ACTION: 1. If the labeled compound recovery was below 25 percent, reject "R" all associated non-detect data (EMPC/EDL) and flag with "J" the positive data for the associated compound.
2. If the labeled compound recovery is above the upper limit (150 percent) flag associated positive data with "J". No effect on non-detects.
3. If the labeled compound recovery is less than 10%, qualify positive hits and non-detects associated with the failed labeled compound "R" (Reject). When highly toxic analytes ($TEF \geq 0.05$) are affected, notify Project Officer to initiate reanalysis.

Recalculate the percent recovery for each labeled standard in the sample extract, Rec_p , using the formula:

$$\% Rec_i = \frac{(A_{i1} + A_{i2}) \times Q_{is} \times 100}{(A_{n1} + A_{n2}) \times RF \times Q_i}$$

$A_{i1} + A_{i2}$ = integrated areas of the two quantitation ions of the appropriate labeled compound.

$A_{n1} + A_{n2}$ = integrated areas of the two quantitation ions of the appropriate internal standard.

Q_i = quantity of the appropriate labeled compound

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

RF was defined, previously.

12.0 Internal Standard Area Response

There is no method criterion for the Internal Standard area response. However, because it is very critical in determining instrument sensitivity, the Internal Standard area response should be checked for every sample. The two standards $^{13}C_{12}1,2,3,4$ -TCDD and $^{13}C_{12}1,2,3,7,8,9$ -HxCDD are referred to as Internal Standards in this method. In other Dioxin methods, the two standards are called Recovery Standards.

- 12.1 Are the internal standard areas for every sample and blank within the upper and lower limits of each associated initial calibration CS3?

N/A YES NO

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

☐ ☐ ☐

12.2 Is the retention time of each internal standard within 15 seconds of the associated initial calibration CS3 standard?

☐ ☐ ☐

- ACTION: 1. If the internal standard area is outside the upper or lower limits, flag all related positive and non-detect data (EMPC/EDL) with "J" regardless whether the lab's labeled compound 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 internal standards differs by more than 15 seconds from the initial calibration CS3, use professional judgement to determine the effect on the results. A time shift of more than 15 seconds may cause certain analytes to elute outside the retention time window established by the GC window defining/column performance check solution. A constant shift could be also the result of a leak.

NOTE: Action 1 and 2 are recommendations only since this criterion is not a method requirement. These guidelines are based on other methods, previously validated data packages and Region II recommendations. If method blanks have low area responses as well as the samples, the validator should seriously consider qualifying the data for this criterion. Action 3 is a method requirement.

13.0 Second Column Confirmation

13.1 Any sample in which 2,3,7,8-TCDF is identified on a DB-5 column, must have a confirmation analysis (Method 1613A, section 15.2). Was a second column confirmation performed?

☐ ☐ ☐

13.2 Was the sample extract reanalyzed on a 30 m DB-225, fused silica capillary column, for 2,3,7,8-TCDF using the GC/MS conditions given in Section 7.9.7.1.2 of the analytical method?

☐ ☐ ☐

NOTE: The concentration of 2,3,7,8-TCDF obtained from the primary column (DB-5) should only be used for qualification, due to better QC data associated with the primary column. Also note that the confirmation and quantitation of 2,3,7,8-TCDF may be accomplished on a SP-2330 GC column.

ACTION: If confirmation is missing, use professional judgement, or contact the Project Officer for assistance.

13.3 Did the second column meet the calibration and linearity specification in Sections 5.0 and 6.0 above?

☐ ☐ ☐

ACTION: If no, refer to section 5.0 and 6.0 for appropriate action.

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

☐ ☐ ☐

ACTION: Note in data assessment the differences, use professional judgement to decide which column data to report for TCDF. No other action is needed since this is not a method requirement

N/A

YES NO

but a technical recommendation.

14.0 Sample Reanalysis

- 14.1 The Project Officer will evaluate the need for reanalyzing the samples with qualified data based on site-specific Data Quality Objectives.
- 14.2 Due to a variety of situations (see below) that may occur during sample analysis, the laboratory is required to reanalyze or re-extract and reanalyze certain samples. If a reanalysis was required but was not performed, contact the Project Officer to initiate reanalysis. List in data assessment all re-extractions and reanalyses and identify the PCDD/PCDF sample data summaries which must be used by the data user (when more than one analysis is submitted for a sample).

Lab must re-extract and/or re-analyzed samples when the following criteria are not met:

1. Contaminated method blank at concentrations above the minimum levels (Table 2)
2. Labeled compound recoveries outside acceptable range of 25-150%.
3. Exceedance of calibration range by an analyte (dilution or re-extract using a smaller aliquot).
4. Recovery of labeled compounds outside acceptable limits (25-150%) in a diluted sample (re-extracted using a smaller aliquot).

ACTION: For criteria 1, 2, or 3, notify the Project Officer to discuss possible re-analysis of sample by the laboratory.

For criteria 4, If the calibration was verified and the re-extracted sample still does not meet labeled recovery requirements (25-150%), then the method does not apply to the sample. The results are not reportable for regulatory purposes (Method 1613A, section 17.2). Notify the Project Officer of problem to initiate re-analysis of sample using a different method. Document in Data Assessment.

15.0 Precision and Recovery (PAR)

The laboratory is required to show initial demonstration of capability, to evaluate and document data quality. Laboratory performance is compared to established performance criteria to determine if results of analyses meet the performance characteristics of the method.

The laboratory must perform and submit data to establish the ability to generate acceptable precision and accuracy.

- 15.1 Did the laboratory analyzed an Initial Precision and Recovery (IPR) standard as outlined in section 8.2 required by the method?

☐ ☐ ☐

ACTION: If no, contact the Project Officer to request resubmittals from the laboratory.

If data is not available, discuss with the Project Officer the feasibility of continuing with validation. If a decision is made to proceed with validation, use professional judgement. All data at a minimum should be qualified as estimated "J". Technically according to the method, data and system performance is unacceptable for all compounds. Analyses should not have continued as per the

N/A

YES NO

method. Document under contract non-compliance in Data Assessment.

- 15.2 Did the IPR standard deviation (s) and average concentration (x) passed criteria as outlined in Table 7 of the method? ☐ ☐ ☐

ACTION: If no, refer to action from section 15.1.

NOTE: The concentration limits in Table 7 for labeled compounds are based on the requirements that the recovery of each labeled compound be in the range of 25-150%.

The laboratory must analyzed an Ongoing Precision and Recovery standard (OPR) periodically, at the beginning of 12 hour shift after the analysis of the CS3 calibration verification (VER), and before the analysis of any sample in each set.

- 15.3 Was the Ongoing Precision and Recovery (OPR) standard analyzed at the required frequency? ☐ ☐ ☐

- 15.4 Did the OPR standard passed the concentration criteria limits in Table 7 of the method? ☐ ☐ ☐

ACTION: If no, refer to action from section 15.1. All samples that do not have a passing OPR standard are potentially affected for that analyte.

The following sections may be incorporated in the validation process on a case by case basis depending upon the requirements of the Project Plan. Sometimes a laboratory will provide data for some of the following sections on a routine basis. If not a requirement of the Project Plan, then professional judgement is needed to qualify data based on additional information.

16.0 Isomer Specificity and Toxicity Equivalency Factor (TEF)

NOTE: The TEF value concentrations can be found in the DFLM01.1 Statement of Work for Dioxin Analysis Form I PCDD-2.

When calculating the 2,3,7,8-TCDD Toxicity Equivalency of a sample only those 2,3,7,8 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 analyte specificity.

- 16.1 Did the lab include EMPC or EDL values in the toxicity equivalency calculations? ☐ ☐ ☐

- 16.2 Were all samples, whose toxicity equivalency exceeded the required values were reanalyzed on a confirmation column to establish analyte specificity? ☐ ☐ ☐

ACTION: 1. If yes, the toxicity equivalency calculations were not calculated properly, notify the Project Officer to arrange for laboratory resubmittals.

2. If the toxicity equivalency exceeded the required limits (0.7 µg/Kg for soil/ sediment, 7 ng/L for aqueous and 7 µg/Kg for chemical waste samples), and the lab failed to reanalyze the samples on a specific secondary column, notify Project Officer. Reanalysis may be initiated.

NOTE: Any qualifications such as "J" applied to target compounds should be also applied to their associated total congeners concentration.

17.0 Rinsate Blank (Region 2 QA guidelines recommend rinse blanks for all projects)

N/A

YES NO

17.1 One rinsate blank should be collected for each batch of 20 soil samples or one per day whichever is more frequent. Were rinsate blanks collected at the above frequency? ☐ ☐ ☐

17.2 Do any rinsate blanks show the presence of 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 1,2,3,7,8-PeCDD at amounts $> .5 \mu\text{g/L}$ or any other analyte at levels $> 1 \mu\text{g/L}$? ☐ ☐ ☐

ACTION: If any rinsate blank was found to be contaminated with any of the PCDDs/PCDFs notify the Project Officer to discuss what proper action must be taken.

If any qualification is needed due to rinsate blank contamination, follow the guidelines outlined under Method Blanks, section 10, Actions 2 and 3.

18.0 Field Blanks

18.1 The field blanks are PEM samples (blind blanks) supplied to Laboratory at the frequency of one field blank per 20 samples or one per samples 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.

Were the following conditions met?

18.2 Acceptable field blanks must not contain any signal of 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDD and 1,2,3,7,8-PeCDF equivalent to a concentration of $> 20 \text{ ng/Kg}$. ☐ ☐ ☐

18.3 For other 2,3,7,8 substituted PCDD/PCDF analytes of each homologue the allowable concentration in the field blank is less than the upper MCLs listed in the method. ☐ ☐ ☐

ACTION: When the field blank is found to be contaminated with target compounds, apply the same action as described for the Method Blank, section 10, Actions 2 and 3.

NOTE: Ask Project Officer to verify that the PEM blank (field blank) did not contain any PCDD/PCDF analytes and ask their assistance in the evaluation of the PEM field blank.

19.0 PEM Interference Fortified Blanks

NOTE: This type of blank may not be available at this time. In many cases, laboratories will substitute matrix spike/matrix spike duplicate (MS/MSD). If a PEM Interference Fortified blank(s) were not analyzed but MS/MSD data were submitted, skip this section and go onto to section 21.

19.1 One known blank usually an interference fortified soil/sediment sample is supplied to the Laboratory. The frequency of this QC sample is one per group of 20 environmental samples or one per samples collected over one week period, whichever occurs first. The sample is spiked by the laboratory with the appropriate volume of the matrix spiking solution and then extracted and analyzed with other samples.

19.2 Was a fortified PEM blank analyzed at the frequency described above? ☐ ☐

N/A

YES NO

- 19.3 Was the percent recovery of 2,3,7,8-TCDD and other 2,3,7,8-substituted compounds within the 50 to 150 percent control limits? ☐ ☐ ☐

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

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

20.0 Matrix Spike (MS) Field Sample

Note: Matrix spike is not required by this method although Labs may routinely perform this analysis as part of internal QA/QC and submit this data as part of the package. Verify requirements with Project Officer.

- 20.1 Was a matrix spike analyzed at the frequency of one per SDG samples per matrix? ☐ ☐ ☐

- 20.2 Was the percent recovery of 2,3,7,8-TCDD and other 2,3,7,8-substituted PCDDs/PCDFs within 60 to 140 percent? ☐ ☐ ☐

ACTION: If problems such as interferences are observed, use professional judgement to assess the quality of the data. The 60-140% 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.

- 20.3 Was a matrix spike duplicate analyzed as per section 11.1 and 11.2? ☐ ☐ ☐

ACTION: No action required. A matrix spike duplicate is not required. Use professional judgement if there is a large difference in concentrations reported between MS and MSD. Qualifications if any, can only be performed on the sample that was used for this criteria.

21.0 Environmental Duplicate Samples (recommended in Region 2 for all Projects)

NOTE: Do not confuse an environmental duplicate with a matrix spike duplicate. An environmental duplicate is a sample that has been divided into 2 parts (extracted and analyzed as two different samples) or as 2 separate samples from the same location sent by the sampling crew. This sample is not spike with any additional compounds other than those compounds required by the method for analysis of all routine samples.

- 21.1 For every batch of 20 samples or samples collected over a period of one week, whichever is less, there must be a sample designated as duplicate. Were duplicate samples collected at the above frequency? ☐ ☐ ☐

- 21.2 Did results of the duplicate samples agree within 25% relative difference for 2,3,7,8-substituted analytes and 50% for the rest of the analytes? ☐ ☐ ☐

ACTION: The duplicate results can be used in conjunction of other QC data. Use professional judgement.

N/A

YES NO

22.0 REFERENCES

The following references are cited in Method 1613. They are important references for technical information and are submitted here as part of this method's documentation.

1. "Analytical Procedures and Quality Assurance Plan for the Determination of PCDD/PCDF in Fish", USEPA Environmental Research Laboratory, 6201 Congdon Boulevard, Duluth, NH 55804, April 1988.
2. Barnes, Donald G., Kutz, Frederick W., and Bottimore, David P., "Update of Toxicity Equivalency Factors (TEFs) for Estimating Risks Associated with Exposure to Mixtures of Chlorinated Dibenzo-p-dioxins and dibenzofurans (CDDs/CDFs)", Risk Assessment Forum, USEPA, Washington, DC 20460, February 1989.
3. Lamparski, L.L., and Nestrick, T.J., "Determination of Tetra-, Hexa-, Hepta-, and Octachlorodibenzo-p-dioxin Isomers in Particulate Samples at Parts per Trillion Levels", Analytical Chemistry, 52: 2045-2054, 1980.
4. "Measurment of 2,3,7,8-Tetrachlorinated Dibenzo-p-dioxin (TCDD) and 2,3,7,8-Tetrachlorinated Dibenzofurans (TCDF) in Pulp, Sludges, Process Samples and Waste-waters from Pulp and Paper Mills", Wright State University, Dayton, OH 45435, June 1988.
5. Method 1613-Revision A- Tetra through Octa- chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, USEPA, Washington, DC, 20460, October 1990
6. "Method 613--2,3,7,8-tetrachlorodibenzo-p-dioxin", 40 CFR 136 (49 FR 43234), October 26, 1984, Section 4.1.
7. "NCASI Procedures for the Preparation and Isomer Specific Analysis of Pulp and Paper Industry Samples for 2,3,7,8 TCDD and 2,3,7,8 TCDF", National Council of the Paper Industry for Air and Stream Improvement, 260 Madison Avenue, New York, NY 10016, Technical Bulletin No.551, Pre-release Copy, July 1988.
8. Provost, L.P., and Elder, R.S., "Interpretation of Percent Recovery Data", American Laboratory, 15: 56-83, 1983
9. Stanley, John S., and Sack, Thomas M., "Protocol for the Analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin by High Resolution Gas Chromatography/High Resolution Mass Spectrometry", USEPA EMSL, Las Vegas, Nevada 89114, EPA 600/4-86-004, January 1986.
10. Tondeur, Yves, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-dioxin and Dibenzofurans hy High Resolution Gas Chromatography/High Resolution Mass Spectrometry", USEPA ENSL, Las Vegas, Nevada, June 1987.
11. Tondeur, Yves, "Proposed GC/MS Methodology for the Analysis of PCDDs and PCDFs in Special Analytical Services Samples", Triangle Laboratories, Inc., 801-10 Capitola Dr., Research Triangle Park, NC 27713, January 1988, updated by personal communication September 1988.

ATTACHMENT A

PCDFs/PCDD DATA ASSESSMENT

SDG No.
LABORATORY:
SITE:

DATA ASSESSMENT

The current Functional Guidelines for evaluating dioxin/furans organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "N" (presumptive evidence for the presence of the material), "U"(non-detects), "R" (unusable), or "JN"(presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two 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 can not 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.

Reviewer's
Signature: _____ Date: __/__/199__

Verified By: _____ Date: __/__/199__

GENERAL COMMENTS:

HOLDING TIME:

BLANK CONTAMINATION:

WINDOW DEFINING MIXTURE:

ION ABUNDANCE:

CALIBRATIONS:

RESOLUTION:

LABELED STANDARDS PERFORMANCE:

INTERNAL STANDARDS:

PEAK IDENTIFICATION:

MATRIX SPIKE/ ENVIRONMENTAL DUPLICATE:

CONFIRMATIONS:

OTHER QC OUT OF SPECIFICATION:

SYSTEM PERFORMANCE AND OVERALL ASSESSMENT:

CONTRACT PROBLEMS NON-COMPLIANCE:

RE-EXTRACTION, REANALYSIS OR DILUTIONS:

DO NOT USE

USE

FIELD DOCUMENTS:

ATTACHMENT B

DATA REJECTION SUMMARY

Type of Review: Organic Date : September 21, 1999 Case/SAS No. : _____
Site Name: _____ Lab Name: _____
Reviewer's Initials : _____ Number of Samples: _____

Analytes Rejected Due To Exceeding Review Criteria For: Number of Compounds /Number of Fractions (Samples)

	Labeled Standards	Holding Times	Calibration	Contamination	ID	Internal Standard	Other	Total # Samples	Total # REJECTED/ Total Analytes in samples Percent
Dioxin (17)	0	0	0	0	0	0	0	0	??

Analytes Estimated Due To Exceeding Review Criteria For: Number of Compounds /Number of Fractions (Samples)

	Labeled Standards	Holding Times	Calibration	Contamination	ID (RT)	Internal Standard	Other	Total # Samples	Total # ESTIMATED/ Total Analytes in samples Percent
Dioxin (17)	0	0	0	0	0	0	0	0	??

Note: Asterisk (*) indicates additional Exceedances of Review Criteria

Data Validation SOP

HW-17, Rev. 1.3

Herbicides

YES NO N/A

This Region II SOP document is based on SW846
Method 8150A, Revision I, July, 1992

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 upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

- 2.1 Have any technical holding times, determined from date of collection to date of extraction, been exceeded?

Note: Water and soil samples for herbicide analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date extraction. However, the SAS Client Request takes precedence and the Holding Times specified in the SAS are the criteria used for validating data.

YES NO N/A

ACTION: If technical holding times are exceeded, flag all positive results as estimated (J) and sample quantitation limits (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 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).

3.0 Surrogate Recovery (Form II)

3.1 Are the Herbicide Surrogate Recovery Summaries (Form II) present for each of the following matrices?

a. Low Water ☐ ☐ ☐

b. Soil ☐ ☐ ☐

3.2 Are all the Herbicide samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?

a. Low Water ☐ ☐ ☐

b. Soil ☐ ☐ ☐

ACTION: Call 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 in red.

3.4 Were surrogate recoveries outside of the advisory limits for any sample or blank? (50-120%) ☐ ☐ ☐

YES NO N/A

ACTION: No qualification is done if the surrogate is diluted out. If recovery for the surrogate is below the contract limit, but above 10%, flag all results for that sample 'J'. If recovery is < 10%, qualify positive results 'J' and flag non-detects 'R'. If recovery is above the contract advisory limit qualify positive values 'J'.

- 3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point calibration analysis? (see Form VI Herb-1)

☐ ☐

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?

☐ ☐

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)

- 4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?

☐ ☐

- 4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices?
(1 MS/MSD must be performed for every 20 samples of similar matrix or concentration level)

a. Low Water

☐ ☐

b. Soil

☐ ☐

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

YES NO N/A

- 4.3 How many herbicide spike recoveries are outside QC limits (60-140%)?

Water

Soil

_____ out of _____

_____ out of _____

- 4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water

Soil

_____ out of _____

_____ out of _____

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

5.0 Blanks (Form IV)

- 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 ? [] _____

YES NO N/A

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 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 qualified because of contamination in another blank. Field blanks must be qualified for surrogate, calibration, or any QC problems.

YES NO N/A

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	

in the associated samples should be qualified
as unusable (R).

NOTE

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 Printouts for both columns present
for all samples, blanks, QC Check reference,
and MS/MSD?

☐

ACTION: If no, take action specified in 3.2 above.

7.2 Are Forms VI - Herbicides 1,2,4 present and complete
for each column and each analytical sequence?

☐

ACTION: If no, take action specified in 3.2
above.

7.3 Are there any transcription/calculation errors
between raw data and Forms VI?

 ☐

YES NO N/A

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.4 Were the retention time windows calculated using the average absolute retention time (at least three measurements) $\pm$ three times the standard deviation of the absolute retention time, for each standard? (Refer to Method 8000A, section 7.5). ☐ ☐ ☐

7.5.1 Was a QC check standard analyzed prior to environmental samples? ☐ ☐ ☐

7.5.2 If yes, was the surrogate recovery >50%? ☐ ☐ ☐

7.5.3 Was the QC 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.

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 - Herbicides - 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 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).

YES NO N/A

- 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 3.2 above.

- 7.11 Have all samples been injected within a 24 hr. period beginning with the injection of the first standard?

YES NO N/A

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 50-100% 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 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

- 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.

- 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.

- 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.

YES NO N/A

- 9.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns < 25.0%? 1

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

<u>% Difference</u>	<u>Qualifier</u>
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? 1

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? 1

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.

- 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

- 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.

YES NO N/A

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.

Data Validation SOP

HW-2, Rev. 11

Metals

Evaluation of Metals Data for the Contract Laboratory Program (CLP)

based on

SOW. 3/90

(SOP Revision XI)

PREPARED BY: _____ DATE: _____
Hanif Sheikh, Quality Assurance Chemist
Toxic and Hazardous Waste Section

APPROVED BY: _____ DATE: _____
Kevin Kubik, Chief
Toxic and Hazardous Waste Section

APPROVED BY: _____ DATE: _____
Robert Runyon, Chief
Monitoring Management Branch

STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program

Date: Jan. 1992
Number: HW-2
Revision: 11

1.0 **Scope**

1.1 This procedure is applicable to inorganic data obtained from contractor laboratories working for Hazardous Waste Site Contract Laboratory Program (CLP).

1.2 The data validation is based upon analytical and quality assurance requirements specified in Statement of Work (SOW) 3/90 .

2.0 **Responsibilities** - Data reviewers will complete the following tasks as assign the Data Review Coordinator:

2.1. For a **total review**:

2.1.1 **Data Assessment - "Total Review-Inorganics" Checklist Appendix (A.1).**
The reviewer must answer every question on the checklist.

2.1.2 **Data Assessment - Data Assessment Narrative (Appendix A.2)**
The answer on the checklist must match the action in the narrative (appendix A.2) and on Form I's. Do not use pencil to write the narrative.

2.1.3 **Contract Non-Compliance - SMO Report (Appendix A.3)**
This report is to be completed only when a serious contract violation is encountered, or upon the request of the Data Validation Task Monitor, or Tech Project Officer (TPO). Forward 5 copies: one each for internal files, appropriate Regional TPO, Sample Management Office (SMO) and last two address Mailing List for Data Reviewers (Appendix A.4). In other cases, all contract violations should be appended to the end of the Data Assessment Narrative (See A.2.2).

2.1.4 CLP Data Assessment Summary Forms

2.1.4.1 Appendix A.5

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

2.1.4.2 Appendix A.6

Data reviewer is also required to fill out Inorganic Regional Data Assessment form (Appendix A.7) provided by EPA Headquarters. Codes listed on the form will be used to describe the Data Assessment Summary.

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Title: Evaluation of Metals Data for the
Contract Laboratory Program

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2.1.5 Data Review Log: It is recommended that each data reviewer should maintain a the reviews completed to include: a. date of start of case

- b. date of completion of case review
- c. site
- d. case number
- e. contract laboratory
- f. number of samples
- g. matrix
- h. hours worked
- i. reviewer's initials

2.1.6 Telephone Record Log - the data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with CLP laboratory. After the case review has been completed, mail white copy of Telephone Record Log to the laboratory and pink copy to SMO. File yellow copy in the Telephone Record Log folder, and attach a xerox copy of the Telephone Record Log to the completed Data Assessment Narrative (Appendix A.2).

2.1.7 Forwarded Paperwork

- 2.1.7.1 Upon completion of review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch
- a. data package
 - b. completed data assessment checklist (Appendix A.1, original)
 - c. SMO Contract Compliance Screening (CCS)
 - d. Record of Communication (copy)
 - e. CLP Reanalysis Request/Approval Record (original + 3 copies)
 - f. Appendix A.6 (original).
- 2.1.7.2 Forward 2 copies of completed Data Assessment Narrative (Appendix A.2) along with 2 copies of the Inorganic Data Assessment Form (Appendix A.6) and Telephone Record Log, if any, one each for appropriate Regional TPO, and the other one to EPA EMSL office in Las Vegas. The addresses of TPOs and office in Las Vegas are given in Appendix A-4.
- 2.1.8 **Filed Paperwork** - Upon completion of review, the following are to be filed within MMB files:
- a. Two copies of completed Data Assessment Narrative (Appendix A.2) each carry Appendix A.6.
 - b. Telephone Record Log (copy)
 - c. SMO Report (copy Appendix A-3)
 - d. CLP Reanalysis Request/Approval Record (copy)

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3.0 **Data Completeness**

Each data package is checked by a Regional Sample Control Coordinator (RSSC) completeness. A data package is assumed to be complete when all the deliverables required under the contract are present. If a data package is incomplete, the would call the laboratory for missing document(s). If the laboratory does not respond within a week, SMO and MMB coordinator of Region II will be notified

4.0 **Rejection of Data** - All values determined to be unacceptable on the Inorganic Analysis Data Sheet (Form I) must be lined over with a red pencil. As soon as review criteria causes data to be rejected, that data can be eliminated any further review or consideration.

5.0 Acceptance Criteria - In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.1 (pages 4-25) should be used. Additional guidance can be found in the National Inorganic Functional Guideline October 1, 1989.

6.0 SMO Contract Compliance Screening (CCS) - This is intended to aid reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from laboratory in response to CCS must be used by the reviewer.

7.0 Request for Reanalysis - Data reviewers must note all items of contract non-compliance within Data Assessment Narrative. If holding times and sample times have not been exceeded, TPO may request reanalysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record".

8.0 Record of Communication - Provided by the Regional Sample Control Center (RSCC) indicate which data packages have been received and are ready to be reviewed.

9.0 Rounding off numbers - The data reviewer will follow the standard practice.

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review)

Date: Jan. 1992
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	YES	NO
A.1.1 <u>Contract Compliance Screening Report (CCS)</u> - Present?	[]	
<u>ACTION:</u> If no, contact RSCC.		
A.1.2 <u>Record of Communication (from RSCC)</u> - Present?	[]	
<u>ACTION:</u> If no, request from RSCC.		

A.1.3 Trip Report - Present and complete? ☐ _____

ACTION: If no, contact RSCC for trip report.

A.1.4 Sample Traffic Report - Present? ☐ _____

Legible? ☐ _____

ACTION: If no, request from Regional Sample Control Center (RSCC).

A.1.5 Cover Page - Present? ☐ _____

Is cover page properly filled in and signed by the lab manager or the manager's designee? ☐ _____

ACTION: If no, prepare Telephone Record Log, and contact laboratory.

Do numbers of samples correspond to numbers on Record of Communication? ☐ _____

Do sample numbers on cover page agree with sample numbers on:

(a) Traffic Report Sheet? ☐ _____

(b) Form I's? ☐ _____

ACTION: If no for any of the above, contact RSCC for clarification.

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review)

Date: Jan. 1992
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A.1.6 Form I to IX Yes No

A.1.6.1 Are all the Form I through Form IX labeled with:

Laboratory name?	[]	
Case/SAS number?	[]	___
EPA sample No.?	[]	
SDG No.?	[]	___
Contract No.?	[]	
Correct units?	[]	___
Matrix?	[]	___

ACTION: If no for any of the above, note under Contract Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.6.2 Do any computation/transcription errors exceed 10% of reported values on Forms I-IX for:

(NOTE: Check all forms against raw data.)

(a) all analytes analyzed by ICP?	[]	___	___
(b) all analytes analyzed by GFAA?	[]	___	___
(c) all analytes analyzed by AA Flame?	[]	___	___
(d) Mercury?	[]	___	___
(e) Cyanide?	[]		

ACTION: If yes, prepare Telephone Log, contact laboratory for corrected data and correct errors with red pencil and initial.

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review)

Date: Jan. 1992
Number: HW-2
Revision: 11

		<u>YES</u>	<u>NO</u>
A.1.7	<u>Raw Data</u>		
A.1.7.1	Digestion Log* for flame AA/ICP (Form XIII) present?	[]	___
	Digestion Log for furnace AA Form XIII present?	[]	___
	Distillation Log for mercury Form XIII present?	[]	___
	Distillation Log for cyanides Form XIII present?	[]	___
	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	[]	___
	*Weights, dilutions and volumes used to obtain values.		
	Percent solids calculation present for soils/sediments?	[]	___
	Are preparation dates present on sample preparation logs/bench sheets?	[]	___
A.1.7.2	Measurement read out record present?		
	ICP	[]	___
	Flame AA	[]	___
	Furnace AA	[]	___
	Mercury	[]	___
	Cyanides	[]	___
A.1.7.3	Are all raw data to support all sample analyses and QC operations present?	[]	___
	Legible?	[]	___
	Properly Labeled?	[]	___

ACTION: If no for any of the above questions
in sections A.1.7.1 through A.1.7.3,
write Telephone Record Log and contact

laboratory for resubmittals.

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Title: Evaluation of Metals for the Contract
Laboratory Program
Appendix A.1: Data Assessment - Contract

Date: Jan. 1992
Number: HW-2
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Compliance (Total Review)

		YES	NO
A.1.8	<u>Holding Times</u> - (aqueous and soil samples) (Examine sample traffic reports and digestion/distillation logs.) Mercury analysis (28 days). exceeded? ☐ ☐ Cyanide distillation (14 days). exceeded? ☐ Other Metals analysis (6 months). exceeded? ☐ <u>NOTE:</u> Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of preparation (from raw data). Attach to checklist. <u>ACTION:</u> If yes, reject (red-line) values less than Instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.		
A.1.8.2	Is pH of aqueous samples for: Metals Analysis >2? ☐ ☐ Cyanides Analysis <12? ☐ ☐ <u>Action:</u> If yes, flag the associated metals and cyanides data as estimated.		
A.1.9	<u>Form I (Final Data)</u>		
A.1.9.1	Are all Form I's present and complete? ☐ ☐		

ACTION: If no, prepare telephone record log and contact laboratory for submittal.

- A.1.9.2 Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's? ☐ ☐
- Are soil sample results for each parameter corrected for percent solids? ☐ ☐
- Are all "less than IDL" values properly coded with "U"? ☐ ☐

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review)

Date: Jan. 1992
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Revision: 11

	<u>YES</u>	<u>NO</u>
Are the correct concentration qualifiers used with final data?	☐	☐

ACTION: If no for any of the above, prepare Telephone Record Log, and contact laboratory for corrected data.

- A.1.9.3 Are EPA sample # s and corresponding laboratory sample ID # s the same as on the Cover Page, Form I's and in the raw data? ☐ ☐
- Was a brief physical description of samples given on Form I's? ☐ ☐
- Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV? ☐ ☐

ACTION: If no for any of the above, note under Contract-Problem/Non-Compliance of the "Data Assessment Narrative".

A.1.10 **Calibration**

- A.1.10.1 Is record of at least 2 point calibration

present for ICP analysis? ☐ ☐

Is record of 5 point calibration present for
Hg analysis? ☐ ☐

Is record of 4 point calibration present for:

Flame AA? ☐ ☐

Furnace AA? ☐ ☐

Cyanides? ☐ ☐

Is one calibration standard at the CRDL level for
all AA (except Hg) and cyanides analyses? ☐ ☐

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

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review)

Date: Jan. 1992
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	<u>YES</u>	<u>NO</u>
A.1.10.2 Is correlation coefficient less than 0.995 for:		
Mercury Analysis?	☐	☐
Cyanide Analysis?		☐
Atomic Absorption Analysis?	☐	☐

ACTION: If yes, flag the associated data as estimated.

NOTE: The data validator shall calculate the correlation
coefficient using concentrations of the standards
and the corresponding instrument response
(e.g. absorbance, peak area, peak height, etc.).

A.1.10.3 In the instance where less than 4 standards are
measured in absorbance (or peak area, peak height, etc.)
mode, are the remaining standards analyzed in

concentration mode immediately after calibration
within $\pm 10\%$ of the true values? []

ACTION: If no, flag the associated data as estimated
if standards are not within $\pm 10\%$ of true values.
Do not flag the data as estimated in linear range
indicated by good recovery of standard(s).

A.1.11 **Form II A (Initial and Continuing Calibration Verification)-**

A.1.11.1 Present and complete for every metal and cyanide? [] _ _

Present and complete for AA and ICP when both are
used for the same analyte? [] _ _

ACTION: If no for any of the above, prepare Telephone
Record Log and contact laboratory.

A.1.11.2 Circle on each Form IIA all percent recoveries that
are outside the contract windows.
Are all calibration standards (initial and continuing)
within control limits:

Metals- 90-110%R? [] _

Hg - 80-120%R? [] _

Cyanides- 85-115%R? []

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ACTION: Flag as estimated (J) all positive data (not
flagged with a "U") analyzed between a
calibration standard with %R between 75-89%
(65-79% for Hg; 70-84% for CN) or 111-125%
(121-135% for Hg; 116-130% for CN) recovery and
nearest good calibration standard. Qualify results
<IDL as estimated (UJ) if the ICV or CCV %R is
75-89% (CN, 70-84% ; HG, 65-79%). Reject (red-line)
as unacceptable data if recovery of the ICV or
CCV is outside the range 75-125% (CN, 70-130%; Hg,
65-135%). Qualify five samples on either side of

YES NO

verification standard out of control limits.

A.1.11.3 Was continuing calibration performed every 10 samples
or every 2 hours? []

Was ICV for cyanides distilled? []

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

A.1.12 **Form II B (CRDL Standards for AA and ICP) -**

A.1.12.1 Was a CRDL standard (CRA) analyzed after initial
calibration for all AA metals (except Hg)? []

Was a mid-range calib. verification standard distilled
and analyzed for cyanide analysis? []

Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI)
for each ICP run? []
(Note: CRI for AL,Ba,Ca,Fe,Mg,Na,or K is not required.)

ACTION: If no for any of the above, flag as estimated
all data falling within the affected ranges.
The affected ranges are:
AA Analysis - **True Value $\pm$ CRDL
ICP Analysis - **True Value $\pm$ 2CRDL
CN Analysis - **True Value $\pm$ 0.5 x True Value.

**True value of CRA, CRI or mid-range standard. Substitute IDL for CRDL when IDL > C
Compute the concentration of the missing mid-range standard from the calibration rang

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A.1.12.2 Was CRI analyzed after ICV/ICB and before the final

YES NO

CCV/CCB, and twice every eight hours of ICP run? []

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

A.1.12.3 Circle on each Form IIB all the percent recoveries that are outside the acceptance windows.

Are CRA and CRI standards within control limits:

Metals 80 - 120%R? []

Is mid-range standard within control limits:

Cyanide 80 - 120%R? []

ACTION: Flag as estimated all sample results within the affected range if the recovery of the standard is between 50-79%; flag only positive data within the affected range if the recovery is between 121-150%; reject all data within the affected range if the recovery is less than 50%; reject only positive data within the affected range if the recovery is greater than 150%. Qualify 50% of the samples on either side of CRI standard outside the control limits.

Note: Flag or reject the final results only when sample raw data are within the affected ranges and the CRDL standards are outside the acceptance windows.

A.1.13 **Form III (Initial and Continuing Calibration Blanks)**

A.1.13.1 Present and complete? []

For both AA and ICP when both are used for the same analyte? []

Was an initial calibration blank analyzed? []

Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)? []

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	<u>YES</u>	<u>NO</u>
--	------------	-----------

ACTION: If no, prepare Telephone Record Log, contact laboratory and write in the Contract-Problems/Non-Compliance section of the "Data Assessment Narrative".

A.1.13.2 Circle on each Form III all calibration blank values that are above CRDL (or 2 x IDL when IDL > CRDL).

Are all calibration blanks (when IDL<CRDL) less than or equal to the Contract Required Detection Limits (CRDLs)? []

Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)? []

ACTION: If no for any of the above, flag as estimated (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value over CRDL (or 2xIDL) and nearest good calibration blank.
Flag five samples on either side of the calibration blank outside the control limits.

A.1.14 **FORM III (Preparation Blank) -**
(Note: The preparation blank for mercury is the same as the calibration blank.)

A.1.14.1 Was one prep. blank analyzed for:

each Sample Delivery Group (SDG)?	[<u> </u>]	<u> </u>
each batch of digested samples?	[<u> </u>]	<u> </u>
each matrix type?	[<u> </u>]	<u> </u>
both AA and ICP when both are used for the same analyte?	[<u> </u>]	<u> </u>

ACTION: If no for any of the above, flag as estimated (J) all the associated positive data <10 x IDLs for which prep. blank

was not analyzed.

NOTE: If only one blank was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

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		<u>YES</u>	<u>NO</u>
A.1.14.2	Is concentration of prep. blank value greater than the CRDL when IDL is less than or equal to CRDL?		[]
	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the prep.blank?		[]
	<u>ACTION:</u> If yes, reject (red-line) all associated data greater than CRDL concentration but less than ten times the prep. blank value.		
A.1.14.3	Is concentration of prep. blank value (Form III) less than two times IDL, when IDL is greater than CRDL?	[]	
	<u>ACTION:</u> If no, reject (red-line) all positive sample results when sample raw data are less than 10 times the prep. blank value.		
A.1.14.4	Is concentration of prep. blank below the negative CRDL?		[]
	<u>ACTION:</u> If yes, reject (red-line) all associated sample results less than 10xCRDL.		
A.1.15	<u>Form IV (ICP Interference Check Sample)</u>		
A.1.15.1	Present and complete?	[]	
	(NOTE: Not required for furnace AA, flame AA, mercury, cyanide and Ca, Mg, K and Na.)		
	Was ICS analyzed at beginning and end of run (or at least twice every 8 hours)?	[]	

A.1.15.2

[]

[]

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NO

[]

[]

[1]

[]

NOTE: If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

A.1.16.2 Was field blank used for spiked sample? _____ [] _____

ACTION: If yes, flag all positive data less than 4 x spike added as estimated (J) for which field blank was used as spiked sample.

A.1.16.3 Circle on each Form VA all spike recoveries that are outside control limits (75% to 125%).

Are all recoveries within control limits? [] _____

If no, is sample concentration greater than or equal to four times spike concentration? [] _____

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YES NO

ACTION: If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on Form V for which sample concentration is less than four times the spike concentration.

Are results outside the control limits (75-125%) flagged with "N" on Form I's and Form VA? [] _____

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

A.1.16.4 Aqueous

Are any spike recoveries:

(a) less than 30%? _____ [] _____

(b) between 30-74%? [] _____

(c) between 126-150%? _____ [] _____

(d) greater than 150%? _____ ☐

ACTION: If less than 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as estimated (J); if between 126-150%, flag as estimated (J) all associated aqueous data not flagged with a "U"; if greater than 150%, reject (red-line) all associated aqueous data not flagged with a "U".

A.1.16.5 **Soil/Sediment**

Are any spike recoveries:

(a) less than 10%? _____ ☐

(b) between 10-74%? _____ ☐

(c) between 126-200%? _____ ☐

(d) greater than 200%? _____ ☐

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	<u>YES</u>	<u>NO</u>
ACTION: If less than 10%, reject all associated data; if between 10-74%, flag all associated data as estimated; if between 126-200%, flag as estimated all associated data was not flagged with a "U"; if greater than 200%, reject all associated data not flagged with a "U".		

A.1.17 **Form VI (Lab Duplicates)**

A.1.17.1 Present and complete for: each SDG? ☐

each matrix type? ☐

each concentration range (i.e. low, med., high)? []

both AA and ICP when both are used for the same
analyte? []

ACTION: If no for any the above, flag as estimated
(J) all the data $\geq$ CRDL* for which duplicate
sample was not analyzed.

Note: 1. If one duplicate sample was analyzed for
more than 20 samples, then first 20 samples do not
have to be flagged as estimated.
2. If percent solids for soil sample and its duplicate
differ by more than 1%, prepare a Form VI for each
duplicate pair, report concentrations in ug/L
on wet weight basis and calculate RPD or Difference
for each analyte.

A.1.17.2 Was field blank used for duplicate analysis? []

ACTION: If yes, flag all data $\geq$ CRDL* as estimated
(J) for which field blank was used as duplicate.

A.1.17.3 Are all values within control limits (RPD 20% or
difference $\leq \pm$ CRDL)? []

If no, are all results outside the control limits
flagged with an * on Form I's and VI? []

ACTION: If no, write in the Contract - Problems/Non-
Compliance section of "Data Assessment Narrative".

* Substitute IDL for CRDL when IDL > CRDL.

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YES NO

NOTE: 1. RPD is not calculable for an analyte of the
sample - duplicate pair when both values are
less than IDL.
2. If the result of lab duplicate analyzed
by GFAA is rejectable due to coefficient of

correlation of MSA, analytical spike recovery,
or duplicate injections criteria, do not apply
precision criteria to metals analyzed by GFAA.

A.1.17.4 Aqueous

Circle on each Form VI all values that are:

RPD > 50%, or
Difference > CRDL*

Is any RPD greater than 50% where sample and duplicate
are both greater than or equal to 5 times *CRDL? _____ [____]

Is any difference** between sample and duplicate greater
than *CRDL where sample and/or duplicate is less than
5 times *CRDL? _____ [____]

ACTION: If yes, flag the associated data as estimated.

A.1.17.5 Soil/Sediment

Circle on each Form VI all values that are:

RPD > 100%, or
Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both
greater than or equal to 5 times *CRDL) :
_____ > 100%? _____ [____]

Is any **difference between sample and duplicate
(where sample and/or duplicate is less than 5x*CRDL) :
_____ > 2x*CRDL? _____ [____]

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate the difference.

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YES

NO

ACTION: If yes, flag the associated data as estimated.

A.1.18 **Field Duplicates**

A.1.18.1 Were field duplicates analyzed? []

ACTION: If yes, prepare a Form VI for each aqueous field duplicate pair. Prepare a Form VI for each soil duplicate pair, if percent solids for sample and its duplicate differ by more than 1%; report concentrations of soils in ug/l on wet weight basis and calculate RPDs or Difference for each analyte.

NOTE: 1. Do not calculate RPD when both values are less than IDL.
2. Flag all associated data only for field duplicate pair.

A.1.18.2 **Aqueous**

Circle all values on self prepared Form VI for field duplicates that are:

RPD > 50%, or
Difference > CRDL*

Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times *CRDL? []

Is any **difference between sample and duplicate greater than *CRDL where sample and/or duplicate is less than 5 times *CRDL? []

ACTION: If yes, flag the associated data as estimated.

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate the difference.

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YES NO

A.1.18.3 Soil/Sediment

Circle all values on self prepared Form VI for
field duplicates that are:

RPD >100%, or

Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both
greater than 5 times *CRDL) :

>100%? ☐ ☐

Is any **difference between sample and duplicate
(where sample and/or duplicate is less than 5x *CRDL) :

>2x *CRDL? ☐ ☐

ACTION: If yes, flag the associated data as estimated.

A.1.19 Form VII (Laboratory Control Sample) (Note: LCS - not
required for aqueous Hg and cyanide analyses.)

A.1.19.1 Was one LCS prepared and analyzed for:

each SDG? ☐ ☐

each batch samples digested/distilled? ☐ ☐

both AA and ICP when both are used for the same
analyte? ☐ ☐

ACTION: If no for any of the above, prepare Telephone
Record Log and contact laboratory for submittal

of results of LCS. Flag as estimated (J) all the data for which LCS was not analyzed.

NOTE: If only one LCS was analyzed for more than 20 samples, then first 20 samples close to LCS do not have to be flagged as estimated.

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate the difference.

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YES NO

A.1.19.2 Aqueous LCS

Circle on each Form VII the LCS percent recoveries outside control limits (80 - 120%) except for aqueous Ag and Sb.

Is any LCS recovery:	less than 50%?	<u> </u>	[<u> </u>]
	between 50% and 79%?		[<u> </u>]
	between 121% and 150%?	<u> </u>	[<u> </u>]
	greater than 150%?		[<u> </u>]

ACTION: Less than 50%, reject (red-line) all data; between 50% and 79%, flag all associated data as estimated (J); between 121% and 150%, flag all positive (not flagged with a "U") results as estimated; greater than 150%, reject all positive results.

A.1.19.3 Solid LCS

NOTE: 1. If "Found" value of LCS is rejectable due to duplicate injections or analytical spike recovery criteria, regardless of LCS recovery, flag the associated data

- as estimated (J).
2. If IDL of an analyte is equal to or greater than true value of LCS, disregard the "Action" below even though LCS is out of control limits.

Is LCS "Found" value higher than the control limits on Form VII? []

ACTION: If yes, qualify all associated positive data as estimated.

Is LCS "Found" value lower than the Control limits on Form VII? _____ []

ACTION: If yes, qualify all associated data as estimated.

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YES NO

A.1.20 **Form IX (ICP Serial Dilution) -**

NOTE: Serial dilution analysis is required only for initial concentrations equal to or greater than 10 x IDL.

A.1.20.1 Was Serial Dilution analysis performed for:

each SDG? [] _____

each matrix type? [] _____

each concentration range (i.e. low, med.)? []

ACTION: If no for any of the above, flag as estimated all the positive data $\geq 10 \times \text{IDLs}$ or $\geq \text{CRDL}$ when $10 \times \text{IDL} \leq \text{CRDL}$ for which Serial Dilution Analysis was not performed.

A.1.20.2 Was field blank(s) used for Serial Dilution Analysis? ☐ [] ☐

ACTION: If yes, flag all associated data $\geq 10 \times \text{IDL}$ as estimated (J). If $10 \times \text{IDL} \leq \text{CRDL}$, flag all data $\geq \text{CRDL}$.

A.1.20.3 Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater. ☐ [] ☐

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

A.1.20.4 Circle on each Form IX all percent difference that are outside the control limits for initial concentrations equal to or greater than $10 \times \text{IDLs}$ only.

Are any % difference values:

> 10%? ☐ [] ☐

$\geq 100\%$? ☐ [] ☐

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ACTION: Flag as estimated (J) all the associated sample data $\geq 10 \times \text{IDLs}$ (or $\geq \text{CRDL}$ when $10 \times \text{IDL} \leq \text{CRDL}$) for which percent difference is greater than 10% but less than 100%. Reject (red-line) all the associated sample results equal to or greater than $10 \times \text{IDLs}$ (or $\geq \text{CRDL}$ when $10 \times \text{IDL} \leq \text{CRDL}$) for which PD is greater than or equal to 100%.

YES

NO

Note: Flag or reject on Form I's only the sample results whose associated raw data are $\geq 10 \times \text{IDL}$ (or $\geq \text{CRDL}$ when $10 \times \text{IDL} \leq \text{CRDL}$)

A.1.21 Furnace Atomic Absorbtion (AA) OC Analysis

A.1.21.1 Are duplicate injections present in furnace raw data (except during full Method of Standard Addition) for each sample analyzed by GFAA? ☐ ☐

ACTION: If no, reject the data on Form I's for which duplicate injections were not performed.

A.1.21.2 Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL? ☐ ☐

Was a dilution analyzed for sample with analytical spike recovery less than 40%? ☐ ☐

ACTION: If no for any of the above, flag all the associated data as estimated.

A.1.21.3 Is *analytical spike recovery outside the control limits (85-115%) for any sample? ☐ ☐

ACTION: If yes, flag as estimated the affected sample results if the recovery is between 10-84%; if the recovery is between 115-200%, flag the associated positive sample results as estimated; reject the associated sample results if the recovery is less than 10%; reject positive sample results if the recovery is greater than 200%.

* Analytical spike is not required on the pre-digestion spiked sample.

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YES

NO

NOTE: Reject or flag the data only when the affected sample(s) was not subsequently analyzed by Method of Standard Addition.

A.1.22 Form VIII (Method of Standard Addition Results)

A.1.22.1 Present? []

If no, is any Form I result coded with "S" or a "+"? []

ACTION: If yes, write request on Telephone Record Log
and contact laboratory for submittal of Form VIII.

A.1.22.2 Is coefficient of correlation for MSA less than 0.990 for
any sample? []

ACTION: If yes, reject (red-line) the affected data.

A.1.22.3 Was *MSA required for any sample but not performed? []

Is coefficient of correlation for MSA less than 0.995? []

Are MSA calculations outside the linear range of the
calibration curve generated at the beginning of the
analytical run? []

ACTION: If yes for any of the above, flag all
the associated data as estimated (J).

A.1.22.4 Was proper quantitation procedure followed correctly
as outlined in the SOW on page E-23? []

ACTION: If no, note exception under Contract Problem/
Non-Compliance section of the "Data Assessment
Narrative", and prepare a separate list.

* MSA is not required on LCS and prep. blank.

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YES

NO

A.1.23 **Dissolved/Total or Inorganic/Total Analytes -**

A.1.23.1 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 (organic + inorganic) analytes on the same sample(s)? []

NOTE: 1. If yes, prepare a list comparing differences between all dissolved (or inorganic) and total analytes. Compute the differences as a percent of the total analyte only when dissolved concentration is greater than CRDL as well as total concentration.
2. Apply the following questions only if inorganic (or dissolved) results are (i) above CRDL, and (ii) greater than total constituents.
3. At least one preparation blank, ICS, and LCS should be analyzed in each analytical run.

A.1.23.2 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%? _____ [] _____

A.1.23.3 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%? _____ [] _____

ACTION: If more than 10%, flag both dissolved (or inorganic) and total values as estimated (J); if more than 50%, reject (red-line) the data for both values.

A.1.24 **Form I (Field Blank) -**

(Note: Designate "Field Blank" as such on Form I.)

A.1.24.1 Circle all field blank values on Form I that are greater than CRDL, (or 2 x IDL when IDL > CRDL).

Is field blank concentration less than CRDL (or 2 x IDL when IDL > CRDL) for all parameters

of associated aqueous and soil samples?
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If no, was field blank value already rejected
due to other QC criteria?

YES NO

[]

ACTION: If no, reject (except field blank results)
all associated positive sample data less
than or equal to five times the field blank
value. Reject on Form I's the soil sample
results that when converted to ug/L on wet
basis are less than or equal to five times
the field blank value in ug/L.

A.1.25 Form X, XI, XII (Verification of Instrumental Parameters).

A.1.25.1 Is verification report present for:

Instrument Detection Limits (quarterly)? [] ____

ICP Interelement Correction Factors (annually)? []

ICP Linear Ranges (quarterly)? [] ____

ACTION: If no, contact TPO of the lab.

A.1.25.2 Form X (Instrument Detection Limits) - (Note: IDL is not
required for Cyanide.)

A.1.25.2.1 Are IDLs present for: all the analytes? [] ____

all the instruments used? [] ____

For both AA and ICP when both are used for the same
analyte? []

ACTION: If no for any of the above, prepare
Telephone Record Log and contact

laboratory.

A.1.25.2.2 Is IDL greater than CRDL for any analyte? []

If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL.

[]

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YES NO

Action : If no, flag as estimated all values less than five times IDL of the instrument whose IDL exceeds CRDL.

A.1.25.3 Form XI (Linear Ranges)

A.1.25.3.1 Was any sample result higher than high linear range of ICP.

___ [] ___

Was any sample result higher than the highest calibration standard for non-ICP parameters?

___ [] ___

If yes for any of the above, was the sample diluted to obtain the result on Form I?

[]

ACTION: If no, flag the result reported on Form I as estimated(J).

A.1.26 Percent Solids of Sediments

A.1.26.1 Are percent solids in sediment(s):

< 50%

___ [] ___

< 10%

___ [] ___

ACTION: If yes, qualify as estimated all the results of a sample that has per cent solids between 10%-50% (i.e. moisture content between 50%-90%). Reject all the results of a sample that has per cent solids less than 10% (i.e. moisture content greater than 90%).

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

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Case#	_____	Site	_____	Matrix: Soil	_____
SDG#	_____	Lab	_____	Water	_____
Contractor	_____	Reviewer	_____	Other	_____

A.2.1 **Validation Flags-** The following flags 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**

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.

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

Contractual Qualifiers- The legend of contractual qualifiers applied by the 1a on Form I's is found on page B-20 of SOW ILM01.0.

A.2.2 The data assessment is given below and on the attached sheets.

STANDARD OPERATING PROCEDURE

Page 28 of 34

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.2: Data Assessment Narrative

Date: Jan. 1992
Number: HW-2
Revision: 11

A.2.2 (continuation)

STANDARD OPERATING PROCEDURE

Page 29 of

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.2: Data Assessment Narrative

Date: Jan. 1992
Number: HW-2
Revision: 11

A.2.2 (continuation)

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.2: Data Assessment Narrative

Date: Jan. 1992
Number: HW-2
Revision: 11

A.2.3 Contract-Problem/Non-Compliance

Signature _____

Signature _____

Verified by: _____ Date: _____

STANDARD OPERATING PROCEDURE

Page 31 of 3

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.3: Contract Non-Compliance
(SMO Report)

Date: Jan. 199
Number: HW-2
Revision: 11

CONTRACT NON-COMPLIANCE
(SMO REPORT)

Regional Review of Uncontrolled Hazardous Waste
Site Contract Laboratory Data Package

CASE NO. _____

The hardcopied (laboratory name)

Inorganic data package received at Region II has been reviewed and the quality assurance performance data summarized. The data reviewed included:

SMO Sample No.: _____

Conc. & Matrix: _____

Contract No. (_____) requires that specific analytical work be done and that associated reports be provided by the contractor to the Regions, EMSL-LV, and SM general criteria used to determine the performance were based on an examination of:

- | | |
|---------------------------------|------------------------------|
| - Data Completeness | - Duplicate Analysis Results |
| - Matrix Spike Results | - Blank Analysis Results |
| - Calibration Standards Results | - MSA Results |

Items of non-compliance with the above contract are described below.

Comments: _____

Reviewer's Initial

Date

STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.4: Mailing List for Data Reviewers

Date: Jan. 1992
Number: HW-2
Revision: 11

STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.5: CLP Data Assessment
Summary Form (Inorganics)

Date: Jan. 1992
Number: HW-2
Revision: 11

Page 34 of 34

Date: Jan. 1992
Number: HW-2
Revision: 11

Inorganic Analysis

Region

SITE

NO. OF SAMPLES/
MATRIX

REVIEWER (IF NOT ESD)

REVIEWER'S NAME

DPO: ACTION _____ FYI _____ COMPLETION DATE _____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES	_____	_____	_____	_____
2.	CALIBRATIONS	_____	_____	_____	_____
3.	BLANKS	_____	_____	_____	_____
4.	ICS	_____	_____	_____	_____
5.	LCS	_____	_____	_____	_____
6.	DUPLICATE ANALYSIS	_____	_____	_____	_____
7.	MATRIX SPIKE	_____	_____	_____	_____
8.	MSA	_____	_____	_____	_____
9.	SERIAL DILUTION	_____	_____	_____	_____
10.	SAMPLE VERIFICATION	_____	_____	_____	_____
11.	OTHER QC	_____	_____	_____	_____
12.	OVERALL ASSESSMENT	_____	_____	_____	_____

O = Data has no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTION ITEMS: _____

AREAS OF CONCERN: _____

NOTABLE PERFORMANCE: _____

DATA VALIDATION SOP

TEPH-01, REV. 2

TEPH

TEPH METHOD VALIDATION CHECKLIST

SITE:

DATE:

SDG:

Y N N/A

Data Completeness and Deliverables

Have any missing deliverables been received and added to the data package? _____

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Custody Documents and Narratives

Are chains of custody present and complete for all samples? _____

ACTION: Contact lab for replacement of missing documents.

Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? _____

ACTION: If any sample analyzed as a soil other than a TCLP, contains 50-90% water, flag all data as estimated. If the soil sample, other than TCLP, contains more than 90% water, all data would be flagged as unusable.

ACTION: If samples were not iced upon receipt, flag all positive results as estimated, and all non-detects "UJ".

Holding Times

Have any TEPH technical holding times, determined from date of collection to date of extraction been exceeded? _____

NOTE: Water and Soil sample must be extracted within 7 days of sample collection. Extracts must be analyzed within forty days of extraction.

Y N N/A

Table of Holding Time Violations

Sample	Sample Matrix	Date Sampled	Date Lab Received	Date Analyzed	Date Extracted
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated ("J" for detects and "UJ" for non-detects). If holding times were grossly exceeded (i.e., more than 2x the holding time), flag all positive data as estimated and reject all non-detects as unusable ("R").

Surrogate Recovery

Are surrogate recoveries summarized within the report?

ACTION: If no, contact the lab for explanation/re-submittals. If re-deliverables are not available, document in the narrative notes. (Surrogate % R limits 60-120%).

Were outliers marked as such?

ACTION: Circle outliers in red.

Were surrogate recoveries outside of specifications for any sample or method blank?

ACTION: If any surrogate recoveries are >10% but do not meet project requirements.

1. Flag all positive results as estimated.
2. Flag all non-detects as estimated detection limits when recoveries are less than the lower limit.
3. If recoveries are above the upper limit, do not flag non-detects.
4. If any surrogate recovery is less than 10%:
 - a. qualify positive results as estimated.
 - b. non-detects for that sample should be qualified as unusable.

Professional judgment is used to qualify data that have method blank surrogate recoveries outside acceptance limits in both the original and re-analysis.

Are there any transcription/calculation errors between the surrogate recoveries listed and raw data?

Y N N/A

Matrix Spike

Is a matrix spike/matrix spike duplicate summary present?

Were matrix spikes analyzed at the required frequency for each of the following matrixes:

a. low water?

b. low soil?

c. medium soil?

ACTION: If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

% Recovery = 70 – 130
RPD = 50%

How many matrix spike recoveries are outside QC limits?

water _____ out of _____

soil _____ out of _____

ACTION: Do not qualify associated sample results on the basis of the MS/MSD data alone. Use the MS/MSD results in conjunction with other QC criteria to determine the need for qualification of associated data. If the MS and MSD both have less than 10 percent recovery for an analyte, reject non-detect results for that analyte and qualify positive results for that analyte as estimated for the sample used for the MS/MSD analysis. If the MS and MSD both have greater than 200 percent recovery for an analyte, reject detected results for that analyte and qualify non-detect results for that analyte as estimated for the sample used for the MS/MSD analysis. Use professional judgment in applying this criterion to other samples.

Laboratory Control Sample

Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the laboratory defined limits of (70-120%)?

ACTION: Document in Data Assessment Narrative.

Y N N/A

Blanks

Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?

Upon examination of laboratory and field blank data, do any blanks contain positive results?

ACTION: If yes, qualify associated results as follows:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as non-detect ("U"). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as non-detectable at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

Prepare a list of sample effected.

Are there field/rinse/equipment blanks associated with every sample?

Calibration

Are raw data and summary sheets present for both initial and continuing calibrations?

Are the % RSD values for the initial calibration less than or equal to 20% or correlation coefficient greater than 0.995?

ACTION: Associated sample data for those analytes with % RSD > 20 will be qualified as estimated.

Y N N/A

Are the % D values between the true and measured concentration values for the continuing calibrations ± 20 ?

ACTION: If no, data following the last in-control standard to the next-in-control standard are potentially affected. Associated detected sample data will be qualified as estimated and associated non-detected sample data will be qualified as estimated if low bias is determined to be present.

Check calibration factors and % RSD values back to raw data for 10% of data received.

Are miss-calculations or transcription errors found?

NOTE: If yes, contact the laboratory.

Compound Quantitation and Reported Detection Limits

Check data for one or more detected target analytes per sample for ten percent of the data packages. Recalculate from the raw data to check for calculation and transcription errors.

Were miscalculation/transcription errors found?

ACTION: If errors are found 100% of the data will be evaluated. Contact the lab for the explanation/resubmittals.

Field Duplicates

Were field duplicates submitted for TEPH analysis?

ACTION: Where both the sample duplicate values are greater than 5 times the SQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 100 percent. Where one or both analytes of the field duplicate pair are less than 5 times the SQL, satisfactory precision is indicated if the field duplicate results agree within 2.5 times the SQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated ("J").

As part of the data validation process, the following validation qualifiers and their meanings will be used:

- "U" - Non-Detect – The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- "J" - Estimated Value – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
- "NJ" - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- "UJ" - Estimated Non-Detect – 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 significant QA/QC problems the analysis is invalid and provides no information as to whether the analyte is present or not. Once the data are flagged with "R", any further review or consideration is unnecessary.

If no determination of the overall bias of a result qualified as estimated can be made, the result will be flagged with "J". If the data reviewer can determine the overall bias for sample data qualified as estimated, the data reviewer will qualify the sample result as either an estimated minimum value (JL) or an estimated maximum value (JH).

DATA VALIDATION SOP
ORGANOTINS

STANDARD OPERATING PROCEDURE DATA VALIDATION OF ORGANOTIN ANALYSIS

A. INTRODUCTION

This Standard Operating Procedure (SOP) provides guidance for the validation of organotin analytical data generated from the analysis of water, soils, sediments, or tissue samples. The validation procedure was based upon the EPA Region II data validation SOP for Pesticides/PCBs (EPA 1992)¹. A copy of the analytical protocol is provided as an appendix to the QAPP.

B. DATA VALIDATION PROCEDURES

1. Holding Times

Criteria: Holding times are established for both extraction and sample analysis. For aqueous, soil and tissue matrices, the extraction must begin within 7 days from sample collection, with analysis within 40 days of extraction.

Actions: The actions are summarized in Table 1. Actions may be taken for either time from sampling to extraction or time from extraction to analysis, but not both. If neither criteria are met, the exceedance which occurs first (i.e., time to extraction, or the criteria which results in additional qualifications or the "R" qualification takes precedence.

Table 1. Summary of Actions for Holding Times

Time from Sampling to Extraction			
Matrix	0-7 days	7-14 days	> 14 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Time from Extraction to Analysis			
Matrix	0-40 days	40-80 days	> 80 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

2. Initial Calibration

The gas chromatograph undergoes a 5 point initial calibration with all of the targeted analytes. The laboratory may use either a linear regression, or calculate response factors, to ensure proper linear response of the instrument.

Criteria: If linear regression is performed, the laboratory must demonstrate that the regression coefficient ("r") is greater than or equal to 0.995.

If calibration factors are used, the laboratory must demonstrate that the relative standard deviation of the calibration factors is less than 30%.

Actions: The actions are summarized in Table 2.

Table 2. Summary of Actions for Initial Calibration

Initial Calibration using Regression			
Matrix	$R \geq 0.995$	$0.95 < r < 0.995$	$R < 0.95$
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Initial Calibration using Calibration Factors			
Matrix	$RSD < 30\%$	$30\% < RSD < 80\%$	$RSD > 80\%$
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

3. Continuing Calibration

The gas chromatograph undergoes a 1 point daily calibration with all of the targeted analytes to ensure consistency with the initial calibration.

Criteria: If linear regression was used for the initial calibration, then the continuing calibration result calculated from the linear regression must agree within 25% of the nominal concentration.

If calibration factors were used for the initial calibration, the laboratory must demonstrate that the percent difference between the mean calibration factors from the continuing calibration and the average response factor from the initial calibration is less than 30%.

Actions: The actions are summarized in Table 3.

Table 3. Summary of Actions for Continuing Calibration

Calibration using Regression			
Matrix	75 – 125% of nominal value	20 – 74% or 126 – 180% of nominal value	< 20% or > 180% of nominal value
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Calibration using Calibration Factors			
Matrix	$\%D < 30\%$	$30\% < \%D < 80\%$	$\%D > 80\%$
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

4. Surrogate Recoveries

For soil, water and tissue samples, surrogate is added to the sample extract prior to derivitization.

Criteria: The laboratory must demonstrate that the surrogate recovery falls between 30 and 160%. If the initial analysis fails this criterion, then the samples must be reprocessed once.

Actions: The actions are summarized in Table 4. Chromatograms should be reviewed prior to taking any action to ensure that there were not interferences or coeluting peaks which resulted in the surrogate recoveries falling outside of the control limit. If it is the Reviewer's professional opinion that the non-compliant surrogate recoveries were due to interferences or co-eluting peaks, no action may be required.

No action is required if the surrogates were diluted out.

Table 4. Summary of Actions for Surrogate Recoveries

	%R < 10%	10 < %R < 30%	30 < %R < 160%	%R > 160%
Positives	"R"	"J"	Accept	"J"
Non-Detects	"R"	"UJ"	Accept	Accept
Note: No action is taken if the surrogates are diluted out.				

5. Method and Field QC Blanks

Method blanks are prepared by the laboratories to identify the potential for laboratory contamination. Field QC blanks are provided by the field team to identify potential contamination by the targeted analytes which may be introduced during field activities.

Criteria: There should be no detected targeted analytes in the method or field QC blanks.

Actions: If the targeted analytes are reported in the method or field QC blanks, and are present at a concentration less than 5 times the blank concentrations in the samples (adjusting for matrix variation and sample size), the reported sample results are potential false-positives and should be qualified with a "U". No action is taken if the sample concentration is greater than 5 times the method or field QC blank results. Likewise, when associated samples are "not detected" for the analyte, no qualification of data is necessary.

6. Data Calculations and Reported Concentrations

The reported concentrations for the targeted analytes in at least one sample in the Sample Delivery Group should be recalculated and verified. If a discrepancy is noted, the laboratory should be contacted to provide a sample calculation. Corrective actions may be required, as appropriate.

If the laboratory employs a confirmation column to verify sample identifications, it is recommended that the Reviewer verify that the concentrations on both columns are comparable. If significant differences are noted which are attributable to interferences or coeluting peaks, the Reviewer should consider reporting the results from the confirmation column.

7. Sample Moisture Contents

If the sediment samples exhibit moisture contents in excess of 50%, the actions in Table 6 should be taken.

Table 6. Actions for Excessive Moisture Contents in Sediments

	%Moist < 50%	50 < %Moist < 90%	%Moist > 90%
Positives	Accept	"J"	"M"
Non-Detects	Accept	"UJ"	"M"

8. Project Specific Requirements

If the direction of the bias can be determined from the analytical results, then the data reviewer can apply the following data qualifiers:

- Potential high bias: JH or UJ
- Potential low bias: JL or UJL

If the direction of the bias can not be determined, or the assessment of bias is contradicted by the quality control results (e.g., holding time exceedance with high surrogate recoveries), then the overall bias is undetermined, and the "J" or "UJ" qualifiers are appropriate.

Data Validation SOP

HW-23B, Rev. 1.0

Pesticides/PCBs

Note: The most current SOP for validating PCB data is HW-23B. However, until a Regional Data Validation SOP can be prepared for Pesticides (i.e., utilizing analytical method SW-846 8081a), Data Validation SOP HW-23 should be used in conjunction with the QA/QC criteria detailed in SW-846 method 8081a.

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ SDG# _____

LAB: _____ SITE: _____

1.0 Data Completeness and Deliverables

1.1 Has all the data been submitted in CLP
deliverable format? 1 1

1.2 Have any missing deliverables been received
and added to the data package? 1 1

ACTION: Call lab for explanation/resubmittal of any
missing deliverables. If lab cannot provide
them, note the effect on review of the data
in the reviewer narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter
present? 1 1

2.2 Are the case number and/or SDG number contained
in the narrative or cover letter? 1 1

3.0 Data Validation Checklist

3.1 Does this data package contain:

Water data?

Waste data?

Soil/solid data?

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

POLYCHLORINATED BIPHENYLS1.0 Traffic Reports and Laboratory Narrative

- 1.1 Are traffic report and chain-of-custody forms present for all samples?

11

ACTION: If no, contact lab for replacement of missing or illegible copies.

- 1.2 Do the traffic reports, chain-of-custody forms or SDG narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?

 11

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, non detects shall be qualified as unusable, "R."

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

2.0 Holding Times

- 2.1 Have any PCB technical holding times, determined from date of collection to date of extraction, been exceeded?

 11

Water and waste samples for PCB analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction. Soils and solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

ACTION: If technical holding times are exceeded, flag all positive results as estimated, "J," and sample quantitation limits "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 re-analysis, the reviewer must use

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

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."

3.0 Surrogate Recovery (Form II)

- 3.1 Were the recoveries of tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) presented on CLP Surrogate Recovery Summary forms (Form II), or equivalent, for each of the following matrices?

a. Water/Waste	<u>11</u>	___	___
b. Soil/Solid	<u>11</u>	___	___

- 3.2 Are all the PCB samples listed on the appropriate surrogate recovery form for each of the following matrices?

a. Water	<u>11</u>	___	___
b. Waste	<u>11</u>	___	___
c. Soil/Solid	<u>11</u>	___	___

ACTION: Call lab for explanation/resubmittals.
If missing deliverables are unavailable,
document the effect in the data assessment.

- 3.3 Did the laboratory provide their developed in-house Surrogate recoveries?

<u>11</u>	___	___
-----------	-----	-----

ACTION: If no, use 70 -130% recovery to qualify in section 3.4 below.

- 3.4 Were surrogate recoveries of TCMX or DCB outside of the laboratory-established upper (UCL) or lower (LCL) control limits for any sample or blank?

___	<u>11</u>	___
-----	-----------	-----

ACTION: Circle all outliers in red.

ACTION: No qualification is done if surrogates are diluted out. If recovery for both surrogates is

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

below the LCL, but above 10%, flag all results for that sample "J". If recovery is < 10% for either surrogate, qualify positive results "J" and flag non-detects "R". If recovery is above the UCL for both surrogates qualify positive values "J".

Note: DCB is used when PCBs are determined as Aroclors. DCB is the internal standard when determining PCB congeners and TCMX the surrogate.

- 3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis?

11

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgement. However, flag positive hits as estimate (J) if confirmed by GC/MS analysis.

- 3.6 Are there any transcription/calculation errors between raw data and Form II?

 11

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document the effect in data assessments.

4.0 Laboratory Control Sample

- 4.1 Are raw data and percent recoveries present for all Laboratory Control samples as required by Method 8000B (section 8.5) and Method 8082 (section 8.4.2)?

11

Verify that QC check samples were extracted and analyzed by the same procedures used for the actual samples.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation /resubmittals. Make note in the data assessment.

NOTE: For aqueous samples, an additional QC check sample must be prepared and analyzed when any analyte in a matrix spike fails the required acceptance criteria (see section 5.3 below). The additional QC check sample must contain each analyte that failed in the MS analysis.

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

Note: When the results for matrix spike analysis indicates a problem due to sample matrix effects, the LCS results are used to verify the laboratory can perform the analysis in a clean sample.

4.2 Were Laboratory Control Samples analyzed at the required concentration for all analytes of interest as specified in Method 8000B (sec.8.5)?

11 — —

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgement to determine the effect on the data.

4.3 Were the LCS recoveries within the laboratory's in-house per cent recoveries (if not available, use 70 - 130%)

11 — —

4.4 If no, were Laboratory Control Samples re-analyzed?

11 — —

Note: Corrective action must be taken when one or more of the analytes of interest fail the QC acceptance criteria (Method 8000B, section 8.7.4)

ACTION: If QC check samples were not re-analyzed, or a general system problem is indicated by repeated failure to meet the QC acceptance criteria specified in the method, make note in the data assessment and use professional judgement to determine the effect on the data.

5.0 Matrix Spikes (Form III)

5.1 Are all data for one matrix spike and matrix duplicate (unspiked) pair (MS/Dup) or matrix spike/matrix duplicate (MS/MSD) present and complete for each matrix Method 8082 (section 8.4.1)?

11 — —

NOTE: For soil and waste samples showing detectable amounts of organics, the lab may substitute replicate samples in place of the matrix spike (see Method 8000B-40, section 8.5.3).

5.2 Have MS/Dup or MS/MSD results been summarized on modified CLP Form III?

11 — —

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

for each of the following matrices? (One MS/Dup, MS/MSD must be performed for every 20 samples of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month (Method 8000B-39 (section 8.5)).

a. Water	<u>11</u>	___	___
b. Waste	<u>11</u>	___	___
c. Soil/Solid	<u>11</u>	___	___

ACTION: If any MS/Dup or MS/MSD data are missing, take the action specified in 3.2 above.

- 5.4 Were the 70 - 130% recoveries used to compare the matrix spike recoveries, or did the lab use the optional QC acceptance criteria discussed in Method 8000B-40 (section 8.5.3.1)?

List the criteria used and make note in data assessment.

Criteria used _____

- 5.5 Was the matrix spike prepared at the proper spike concentration? (Method 8000B, section 8.5.1-8.5.2) 11 ___ ___

For aqueous organic extractable, the spike concentration should be prepared according options in: Method 8000B-40, (section 8.5.1 and 8.5.2).

ACTION: No action is taken based on MS or replicate data alone. However, using informed professional judgement, the data reviewer may use the matrix spike or laboratory replicate results in conjunction with other QC criteria and determine the need for some qualification of the data. In some instances it may be determined that only the replicate or spiked samples are affected. Alternatively, the data may suggest that the laboratory is having a systematic problem with one or more analytes, thereby affecting all associated samples.

6.0 Blanks (Form IV)

STANDARD OPERATING PROCEDURE

USEPA Region II
SW846 Method 8082Date: May, 2002
SOP HW-23B, Rev.1.0

YES NO N/A

6.1 Was reagent blank data reported on CLP equivalent
Method Blank Summary form(s) (Form IV)?☒ ☐ ☐6.2 Frequency of Analysis: Has a reagent blank been
analyzed for every 20 (or less) samples
of similar matrix or concentration or each
extraction batch?☒ ☐ ☐ACTION: If any blank data are missing, take action as
specified above (section 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.6.3 Chromatography: review the blank raw data -
chromatograms, quant reports or data system
printouts.Is the chromatographic performance (baseline
stability) for each instrument acceptable for
PCBs?☒ ☐ ☐ACTION: Use professional judgement to determine the
effect on the data.7.0 ContaminationNOTE: "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/reagent/cleanup blanks
have positive results for PCBs? When applied as
described below, the contaminant concentration
in these blanks are multiplied by the sample
Dilution Factor and corrected for % moisture
when necessary.☐ ☒ ☐7.2 Do any field/rinse blanks have positive
PCB 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.

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YES NO N/A

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 sample results due to contamination. Use the largest value from all the associated blanks.

Sample conc > EDL but < 5 x blank	Sample conc < EDL & is < 5 x blank value	Sample conc > EDL & > 5 x blank value
Flag sample result with a "U"	Report EDL & qualify "U"	No qualification is needed

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

7.3 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.

8.0 GC Apparatus and Materials

8.1 Was the proper gas chromatographic capillary column used for the analysis of PCBs?

Action: Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (Method 8082, section 4.2) ☐ ☐ ☐

8.2 Indicate the specific type of narrow bore or wide bore (.53 mm ID, fused silica GC columns, such as DB-608 and DB-1701 or equivalent).

column 1: _____

column 2: _____

ACTION: Note any changes to the suggested materials in section 8.1 above in the data assessment. Also note the impact (positive or negative) such changes have on the analytical results.

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YES NO N/A

9.0 Calibration and GC Performance

9.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS, replicates?

a. Samples	<u>11</u>	___	___
b. All blanks	<u>11</u>	___	___
c. Matrix spike samples	<u>11</u>	___	___
d. 5 pt. initial calibration standards	<u>11</u>	___	___
e. calibration verification standards	<u>11</u>	___	___
f. Laboratory Control samples (LCS)	<u>11</u>	___	___

ACTION: If no, take action specified in 3.2 above.

9.2 Are data summary forms (containing calibration factors or response factors) for the initial 5 pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence?

11 ___ ___

Note: Calibration Aroclor mixtures other than 1016/1260 may be used (as per approved project QA plan)

NOTE: If internal standard calibration procedure is used (Method 8000B-15(section 7.4.2.2)), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (Method 8000B-16 (section 7.4.2.1)), then calibration factors must be used. The internal standard approach is highly recommended for PCB congener analysis.

ACTION: If any data are missing or it cannot be determined how the laboratory calculated calibration factors or response factors, contact the lab for explanation/resubmittals. Make necessary corrections and note any problems in the data assessment.

9.3 Are there any transcription/calculation errors between raw data and data summary forms?

___ 11 ___

ACTION: If large errors exist, call lab for

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YES NO N/A

explanation/resubmittal, make necessary corrections and document the effect in data assessments.

- 9.4 Are standard retention time (RT) windows for each PCB peak of interest presented on modified CLP summary forms?

☐ ☐ ☐

ACTION: If any data are missing, or it cannot be determined how RT windows were calculated, call the lab for explanation/resubmittals. Note any problems in the data assessment.

NOTE: Retention time windows for all PCBs are established using retention times from three calibration standards analyzed during the entire analytical sequence (Method 8000B, section 7.6).

Best results are obtained using retention times which span the entire sequence; i.e., using the calibration verification/continuing calibration standards analyzed every 12 hours.

- 9.5 Were RT windows on the confirmation column established using three standards as described above?

☐ ☐ ☐

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.4 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

ACTION: Note potential problems, if any, in the data assessment.

- 9.6 Do all standard retention times in each level of the initial 5 pt. calibrations for PCBs fall within the windows established during the initial calibration sequence?

☐ ☐ ☐

ACTION i: If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standard spanning the entire sequence were used to obtained RT windows. If the lab used three standards from the 5 pt., RT windows may be too tight. If so, RT windows should be recalculated as per Method 8081B-15 (section 7.4.6).

ii. Alternatively, check to see if the chromatograms contain peaks

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YES NO N/A

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 but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

- 9.7 Has the linearity criteria for the initial calibration standards been satisfied for both columns? (% RSD must be < 20.0% for all analytes).

11 — —

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).

- 9.8 Does the calibration verification/continuing Calibration standard contain the PCB peaks of interest, analyzed on each working day, prior to sample analyses (Method 8082, sections 7.6.2)?

11 — —

- 9.9 Has a calibration verification/continuing calibration standard been analyzed after every 10 samples and at the end of each analytical sequence (Method 8082, section 7.6.2)

11 — —

ACTION: If no, take action as specified in section 3.2 above.

- 9.10 Has the percent difference (%D) exceeded $\pm 15\%$ for any PCB analyte in any calibration verification/Continuing calibration standard?

11 — —

- 9.11 Has a new 5 pt. initial calibration curve been generated for those PCB analytes which failed in the calibration verification/continuing calibration standard (8000B, section 7.7.3), and all samples which followed the out-of-control calibration verification/standard continuing calibration Standard?

11 — —

ACTION: If the %D for any analyte exceeded the $\pm 15\%$ criterion and the instrument was not recalibrated for those analytes, qualify positive results for all associated samples (those which followed the out-of-control standard) "J" and sample quantitation limits "UJ". If the %D was > 90%

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YES NO N/A

for any analyte, qualify non-detects "R", unusable.

- 9.12 Have retention time (RT) windows been properly calculated for each analyte of interest (Method 8000B, section 7.6), using RTs from the associated calibration verification/continuing standard?

☒ ☐ ☐

ACTION: If no, take action specified in section 3.2 above

- 9.13 Do all standard retention times for each calibration verification/continuing calibration standard fall within the windows established during the initial calibration sequence?

☒ ☐ ☐

- 9.14 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows

☒ ☐ ☐

ACTION: If the answer to either 9.13 or 9.14 above is no, check the chromatograms of all samples which followed the last in-control standard. All samples analyzed after the last in-control standard must be re-injected, if initial analysis indicated the presence of the specific analyte that exceeded the retention time criteria. If samples were not re-analyzed, document under Contract Non-compliance in the Data Assessment.

Reviewer has two options to determine how to qualify questionable sample data. First option is to determine if possible peaks are present within daily retention time window. If no possible peaks are found, non-detects are valid. If possible peaks are found (or interference), qualify positive hits as presumptively present "NJ" and non-detects are rejected "R". Second option is to use the ratio of the retention time of the analyte over the retention time of either surrogate. The passing criteria is ± 0.06 RRT units of the RRT of the standard component. Reject "R" all questionable analytes exceeding criteria, and "NJ" all other positive hits.

For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use option 2 specified in paragraph above.

- 9.15 Are there any transcription/calculation errors

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YES NO N/A

between raw data and data summary forms?

☐ ☒ ☐

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document the effect in data assessments under "Conclusions".

10.0 Analytical Sequence Check (Form VIII-PEST)

10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column?

☒ ☐ ☐

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses?

☒ ☐ ☐

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.

10.3 Were the TCMX/DCB surrogate RTs for the samples within the mean surrogate RT from the initial calibration?

☒ ☐ ☐

Action: If no, see "Action" in section 9.14 above

11.0 Extraction Techniques for Sample Preparation

Method 8081B permits a variety of extraction techniques to be used for sample preparation. Which extraction procedure was used?

1. Aqueous samples:

1. Separatory funnel (Method 3510)

☒ ☐ ☐

2. Continuous liquid-liquid extraction (Method 3520)

☒ ☐ ☐

3. Solid phase extraction (Method 3535)

☒ ☐ ☐

4. Other

☒ ☐ ☐

2. Solid samples:

1. Soxhlet (Method 3540)

☒ ☐ ☐

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YES NO N/A

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| 2. Automated Soxhlet (Method 3541) | ☐ | ☐ | ☐ |
| 3. Pressurized fluid (Method 3545) | ☐ | ☐ | ☐ |
| 4. Microwave extraction (Method 3546) | ☐ | ☐ | ☐ |
| 5. Ultrasonic extraction (Method 3550) | ☐ | ☐ | ☐ |
| 6. Supercritical fluid (Method 3562) | ☐ | ☐ | ☐ |
| 7. Other | ☐ | ☐ | ☐ |

11.1 Extract Cleanup - Efficiency Verification (Form IX)

- 11.1.1 Method 8082 (section 7.2) references method 3660 (sulfur) and 3665A (sulfuric acid) to use for Cleaning extracts. Were one or both method used? ☐ ☐ ☐

ACTION: If no, take action specified in 3.2 above. If data suggests cleanup was not performed, make note in the data assessment.

NOTE: Method 3620A, Florisil, may be used per approved project QA plan. The method does not list which analytes and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct PCB list. If not stated or available, use the CLP listing or accept what the laboratory used.

- 11.2 Are all samples listed on modified CLP PCBs Florisil/Cartridge Check Form? ☐ ☐ ☐

ACTION: If no, take action specified in 3.2 above.

- 11.3 Was GPC Cleanup (method 3640A) performed? ☐ ☐ ☐

NOTE: GPC cleanup is not required and is optional. The reviewer should check Project Plan to verify requirement.

- 11.4 Were the same PCB analytes used in calibration used to check the efficiency of the cleanup procedures? ☐ ☐ ☐

- 11.5 Are percent recoveries (% R) of the PCBs and surrogate compounds used to check the efficiency of the cleanup procedures within lab's in-house QC limits (use 70-130% if not available) ☐ ☐ ☐

70-130% for GPC calibration? ☐ ☐ ☐

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YES NO N/A

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 80%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for PCBs. Use professional judgement to qualify positive results if recoveries are greater than the upper limit.

12.0 PCB Identification

12.1 Has CLP Form X or equivalent, showing **retention time** data for positive results on the two GC columns, been completed for every sample in which a PCB was detected?

11 — —

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and cleanup verification forms)?

— 11 —

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error in the data assessment.

12.3 Are retention times (RT) of sample compounds within the established RT windows for both columns/analyses?

11 — —

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently.

Were there any false negatives?

— 11 —

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YES NO N/A

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation provided when sample concentration was sufficient (> 10 ug/ml) in the final extract?

11 — —

ACTION: Indicate with red pencil which Form I results were confirmed by GC/MS and also note in data assessment.

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%?

11 — —

NOTE: The method requires quantitation from one column. The second column is to confirm the presence of an analyte. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

<u>% Difference</u>	<u>Qualifier</u>
0-25%	none
26-70%	"J"
71-100%	"NJ"
>100% *	"R"
100-200% (Interference detected)**	"NJ"
>50% (PCBs value is <CRQL)	"U"

When the reported PCBs value is <CRQL and the %D is >50%, raise the value to the CRQL and qualify with "U" (non-detect).

* Check the chromatogram. If pattern is confirmed qualify "J". If pattern is mixed, has interference, or the PCB cannot be positively determined due to weathering, qualify "JN".

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YES NO N/A

If PCB can not be confirmed, qualify the PCB as "R".

- ** When the reported %D is 100-200% but interference is detected in either column, qualify the data with "NJ".

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 values. Were any errors found? 11

NOTE: Single-peak PCBs results can be checked for rough agreement between quantitative results obtained on the two GC columns. 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 interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

- 13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture? 11

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 EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the 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.

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YES NO N/A

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable? ☒ ☐ ☐

14.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ☐ ☒ ☐

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for PCB 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. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made to determine the proper identification of field duplicates.

DATA VALIDATION SOP
RADIOCHEMICAL ANALYSES

ENVIRONMENTAL DATA SERVICES

RADIOCHEMICAL ANALYSES VALIDATION CHECKLIST (SOP)

SITE:

DATE:

SDG:

Y N N/A

Data Completeness and Deliverables

Have any missing deliverables been received and added to the data package?

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Validation Radiochemical Data

The data validation criteria outlined in this section applies to radiochemical data obtained via beta and gamma spectrometry.

RADIOCHEMICAL ANALYSES

Sample Receipt/Lab Case Narrative

Are all chains-of-custody present for all samples?

ACTION: If no, prepare Telephone Record Log and contact lab for submission of missing or illegible copies.

Do chain of custody records or lab case narrative indicate any problems with sample receipt , condition of samples, analytical problems or special circumstances affecting the quality of the data?

Y N N/A

Holding Times

Have any radiological technical holding times, determined from date of collection to date of analysis, been exceeded?

The holding time for soil is 6 months from date samples were collected, with the exception of Be-7 (3 months).

Table of Holding Time Violations

Sample ID	Sample Matrix	Preserved?	Date Sampled	Date Lab Received	Date Analyzed
-----------	---------------	------------	--------------	-------------------	---------------

ACTION: If technical holding times are exceeded document in the Data Assessment Narrative 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 time on the data.

Sample Replicate Analysis

Is a duplicate summary form present?

Were replicates analyzed at the required frequency?

One per twenty field samples analyzed by each system?

ACTION: If no, prepare Telephone Record Log and contact laboratory for explanation/resubmittals. Note any impact on data quality in the Data Assessment Narrative.

Raw data present should include the following:

- A. Detector used
- B. Analyst initials
- C. Date analyzed
- D. Sample I.D.
- E. Value obtained for initial analyses
- F. Value obtained for replicate analyses
- G. Mean value
- H. Duration of counts in minutes
 - samples
 - background
- I. CPM's for samples and background

Y N N/A

Are all associated replicate values within allowable limits (overlap of the 2 sigma bands)?

ACTION: Qualify associated data as estimated if the above criterion is not met.

Blanks

Is a method blank summary present?

Frequency of Analysis:

Has a reagent/method blank been analyzed for Pb-210 with each analytical batch?

Has a blank container been counted by gamma detector each week associated samples are counted?

ACTION: If any method blank data are missing, prepare a Telephone Record Log and contact lab for an explanation/ resubmittal. If method blank data are not available, reject all associated positive data. However using professional judgement, the data reviewer may substitute field blank or trip blank data for missing method blank.

Contamination

Do any of the method/instrument/reagent blanks have positive results for radiological isotopes of concern?

When applied as described below, the contaminant concentration in these blanks are multiplied by any dilution factor and corrected for % moisture.

ACTION: Prepare a list of samples associated with contaminated blanks.

ACTION: If a compound is found in a blank, but not found in the sample, no action is taken.

If a blank has a positive result for an analyte, qualify associated sample data as follows:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag the sample result as a non-detect ('U'). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detected at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5x criteria, such that a comparison of the total contamination is actually made.

Target Analytes

Are the radiochemical analysis data summaries present (example: Form I RAD) with required header information, for each of the following:

- | | | | | |
|----|--|-------|-------|-------|
| a. | Samples and or fractions as appropriate? | _____ | _____ | _____ |
| b. | Duplicates? | _____ | _____ | _____ |
| c. | Blanks? | _____ | _____ | _____ |

Are the radiological raw data including beta and gamma instrument printouts and calculations in the sample package for each of the following:

- | | | | | |
|----|--|-------|-------|-------|
| a. | Samples and/or fractions as appropriate? | _____ | _____ | _____ |
| b. | Duplicates? | _____ | _____ | _____ |
| c. | Blanks? | _____ | _____ | _____ |

ACTION: If any data are missing prepare Telephone Record and contact lab to obtain explanation/resubmittals. Note any impact on the data quality in the Data Assessment Narrative.

Y N N/A

Analyte Quantitation and Reported Detection Limits

Are there any transcription/calculation errors in data summary results?

Check at least two positive values. Were any errors found?

Are the LLD adjusted to reflect sample dilutions?

ACTION: If errors are large, contact lab for explanation/ resubmittal, make any necessary corrections and note errors in the Data Assessment Narrative.

BETA Spectrometry Initial Calibration

Are the initial calibration summary forms present and complete for each detector used to quantitate samples?

Are the following records present for each detector used:

Identification of Detection?

Analysts initials?

Date and time of calibration?

Name/Activity/Date of certified NBS material?

Counts per minute?

Duration of counting?

Calculated Efficiencies?

ACTION: Contact the laboratory for submission of any missing data .

Are efficiencies of each detector used greater than 15%?

ACTION: Qualify as estimated results associated with a non-compliant calibration.

Y N N/A

BETA Spectrometry Continuing Calibration

Are the continuing calibration summaries present and complete?

Has a continuing calibration standard been analyzed twice every week of sample analysis per instrument?

ACTION: List below all sample analysis that were not within one week of the previous continuing calibration set.

ACTION: If any forms are missing or if a continuing calibration standard was not analyzed twice during each week of every sample analysis, prepare Telephone Log and contact lab for explanation/resubmittal.

Are the following continuing calibration records present for each detector used:

Control charts and logs defining check source results.

ACTION: Contact laboratory for submission of any missing data.

Are the check source count rates within three standard deviations of the mean?

ACTION: Qualify as estimated results associated with a non-compliant calibration.

Recovery Factors

For the Lead-210 analyses is the chemical recovery or yield greater than 20%?

ACTION: Reject all associated data if recovery is less than 20%.

Y N N/A

GAMMA Spectrometry Continuing Calibration

Are the continuing calibration summaries present and complete?

Has a continuing calibration standard been analyzed twice during each week of sample analysis per instrument?

ACTION: List below all sample analysis that were not within one week of the previous continuing calibration set.

ACTION: If any forms are missing or if a continuing calibration standard was not analyzed twice during each week of every sample analysis, prepare Telephone Log and contact lab for explanation/resubmittal.

Are the following continuing calibration records present for each detector used:

Control charts and logs defining check source results and efficiency limits?

Blank results for background control (gamma only)?

ACTION: Contact laboratory for submission of any missing data.

Are efficiencies of selected energies within three standard deviations of the mean?

Are the resolutions of each peak less than 5 KEV?

ACTION: Qualify as estimated sample results associated with non-compliant calibrations.

Y N N/A

Field Duplicates

Were any field duplicates submitted for radio chemical analysis?

ACTION: Where both the sample and duplicate values are greater than 5 times the SQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 100 percent. Where one or both analytes of the field duplicate pair are less than 5 times the SQL, satisfactory precision is indicated if the field duplicate results agree within 2.5 times the SQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated ('J').

SOP RAD-1
Prepared 8/05
REV: 3

ITEMS MARKED THROUGHOUT THE DOCUMENT WITH AN *

Data Validation SOP

HW-22, Rev. 2

Semivolatile Organics

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8270C" December, 1996. Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. 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," October 1999. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

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 defined on page 4.

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 SW846 Analytical Method 8270C.

YES NO N/A

DEFINITIONS

Acronyms

BNA - base neutral acid (another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB - decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
l - liter
ml - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
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

YES NO N/A

TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

YES NO N/A

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."
- NJ - 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.

YES NO N/A

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.
- A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.
- X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ LAB: _____

SITE NAME: _____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable
format? [] _____

ACTION: If not, note the effect on review of the data
in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter
present? [] _____

2.2 Are case number and SDG number(s) contained
in the narrative or cover letter? [] _____

YES NO N/A

II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the 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 Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations 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 flagged 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, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semi volatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semi volatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within 40 days of the date of extraction.

YES NO N/A

Table of Holding Time Violations

Sample ID	Sample Matrix	Date Sampled	(See Traffic Report)		
			Date Lab Received	Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If technical holding times are 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 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 results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

YES NO N/A

3.0 Surrogate Recovery (Form II)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water []

b. Low/Med Soil []

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water []

b. Low/Med Soil []

ACTION: If CLP deliverables or equivalent are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk? [] ___
ACTION: Circle all outliers in red.

YES NO N/A

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank ___ []

If yes, were samples re-analyzed? []

Were method blanks re-analyzed? []

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

- 1. Flag all positive results as estimated ("J").
- 2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
- 3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

- 1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".
- 2. Non-detects for that fraction should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses.

YES NO N/A

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

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)

4.1 Have the semivolatile Matrix Spike and
Matrix Spike Duplicate or duplicate unspiked
Sample recoveries been listed on the CLP
Recovery Form (Form III)?

NOTE: This method may not require a Matrix Spike
Duplicate. Lab should submit MS/MSD or MS and
Duplicate unspiked sample. (see section 8.4.2,
page 8270C-22)

4.2 Were matrix spikes analyzed at the required
frequency for each of the following matrices:

a. Low Water	<u> </u>	<u> </u>
b. Low Solid	<u> </u>	<u> </u>
c. Med Solid	<u> </u>	<u> </u>

ACTION: If any matrix spike data are missing, take
the action specified in 3.2 above. It may be
necessary to contact the lab to obtain the
required data.

YES NO N/A

NOTE: If the data has not been reported on CLP forms, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

- 4.3 Were matrix spikes performed at concentration > 100ug/L ? ☐ ☐ ☐
- 4.4 Were any semivolatile spike recoveries outside QC limits (compare to the values in Table 6, page 8270C-39 and 40) or Lab's in-house generated criteria? ☐ ☐ ☐
- 4.5 Were any RPD's for matrix spike and matrix spike duplicate recoveries outside QC limits? ☐ ☐ ☐

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the data reviewer may use the matrix spike / matrix spike duplicate and duplicate unspiked results in conjunction with other QC criteria to determine the need for some qualification of the data.

YES NO N/A

4.6 Was a LCS analyzed with each analytical batch? (See section 8.4.3, page 8270C-22)

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

4.7 Were any LCS recovery outside the interim acceptance criteria of 70 - 130% or outside lab's in-house generated limits?

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present?

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch?

5.3 Has a method blank been analyzed for each GC/MS system used ?

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

YES NO N/A

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

Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?

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

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/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

YES NO N/A

6.2 Do any field/rinse/ blanks have positive results
for target analytes and/or TICs (if required,
see paragraph 10 below)? []

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) must be used to qualify data.
Blanks may not be qualified because of
contamination in another blank. Field blanks
must be qualified for outlying surrogates,
poor spectra, instrument performance or
calibration QC problems.

ACTION: Follow the directions in the table below to
qualify sample 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).

**For Common Phthalate
Esters:**

Sample conc. > CRDL,
but < 10x blank
result

Sample conc. is
< CRDL & < 10x blank
result

Sample conc. > CRDL &
> 10x blank result

**For Other
Contaminants:**

Sample conc. > CRDL,
but < 5x blank result

Sample conc. < CRDL &
< 5x blank result

Sample conc. > CRDL &
> 5x blank result

Action:

Flag sample result
with a "U"

Report CRDL and
qualify with a "U"

No qualification is
necessary

YES NO N/A

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

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required. (see section 7.6.2, page 8270C-19)

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 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270C? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column. ☐

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

YES NO N/A

8.0 GC/MS Instrument Performance Check

8.1 Are the GC/MS Instrument Performance Check Forms
(Form V) present for decafluorotriphenylphosphine
(DFTPP)? [] ____

NOTE: The performance solution should also contain 4,4-DDT,
pentachlorophenol, and benzidine to verify
injection port inertness and column performance.
The degradation of DDT to DDE and DDD must be
less than 20% total and the response of
pentachlorophenol and benzidine should be
within normal ranges for these compounds (based
upon lab experience) and show no peak degradation
or tailing before samples are analyzed. (see section 5.5
page 8270C-11).

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

8.3 Has an instrument performance check solution
been analyzed for every twelve hours of sample
analysis per instrument? [] ____

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

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

YES NO N/A

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

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

8.4 Have the ion abundances been normalized to m/z 198? [] ____

8.5 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 TOPO must be notified.

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

8.7 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.

8.8 Are the spectra of the mass calibration compound acceptable? [] ____

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

YES NO N/A

9.0 Target Analytes

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

- a. Samples and/or fractions as appropriate ☐ ☐
- b. Matrix spikes and matrix spike duplicates ☐ ☐
- c. Blanks ☐ ☐
- D. Lab control samples ☐ ☐

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270C-13)? ☐ ☐

ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, 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. Matrix spikes and matrix spike duplicates (Mass spectra not required) ☐ ☐
- c. Blanks ☐ ☐
- D Lab control samples ☐ ☐

ACTION: If any data are missing, take action specified in 3.2 above.

YES NO N/A

9.4 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.

9.5 Are the lab-generated standard mass spectra of identified semivolatile compounds present for each sample? ☐ ___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.6 Is the RRT of each reported compound within ± 0.06 RRT units of the standard RRT in the continuing calibration? ☐ ___

9.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? ☐ ___

YES NO N/A

9.8 Do the relative intensities of the characteristic ions in the sample agree within $\pm 30\%$ of the corresponding relative intensities in the reference spectrum? ☐ ☐

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 9.6, 9.7, and 9.8.

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.

10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form I's, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? ☐ ☐

10.2 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 ☐ ☐

ACTION: If any TIC data are missing, take action specified in 3.2 above.

YES NO N/A

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)? []

- ACTION: i. Flag with "R" any target compound listed as a TIC.
- ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? []

10.5 Do TIC and "best match" standard relative ion intensities agree within $\pm 20\%$? []

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". 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".

YES NO N/A

11.0 Compound Quantitation and Reported Detection Limits

NOTE: Average Response Factor from the initial calibration is used for quantitation.

11.1 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? []

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two co-eluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and % moisture in case of soil samples? []

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

YES NO N/A

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). 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 (if present) 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.

12.0 Standards Data (GC/MS)

12.1 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 3.2 above.

13.0 GC/MS Initial Calibration (Form VI)

13.1 Are the Initial Calibration Forms (Form VI) present and complete for the semivolatile fraction?

ACTION: If any calibration forms or standard raw data are missing, take action specified in 3.2 above.

YES NO N/A

13.2 Are all **average RRF's** of the four System Performance Check Compounds (**SPCCs**): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol and 4-nitrophenol > 0.050?

ACTION: If no:
CONTRACTUAL - Circle all outliers in red.
Document in the Data Assessment under contract non compliance.

ACTION: TECHNICAL - For any target analyte, CCC or SPCC with average **RRF <0.05**

1. "R" all non-detects;
2. "J" all positive results.

13.3 Is the % RSD for each individual Calibration Check Compound (CCC) - Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, Diphenylamine, Di-n-octyl phthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol and 2,4,6-Trichlorophenol less than 30%?

ACTION: If no:
CONTRACTUAL - Circle all outliers in red.
Document in the Data Assessment under contract non compliance.

TECHNICAL - All positive hits for that particular CCC must be qualified "J". If % RSD > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

YES NO N/A

13.4 If % RSD for one or more target analytes exceeds 15%,
is the MEAN of the % RSD values for ALL analytes in
the calibration less than or equal to 15%?

ACTION: If yes:
CONTRACTUAL - The initial calibration is
valid and the average RF from the initial
calibration is used to quantitate sample results.

TECHNICAL - If the % RSD is $\geq$ 15.0% for any
Individual target analyte, qualify positive
results for that analyte "J". If % RSD > 90%,
flag all positive results for that analyte "J" and
non-detect results for that analyte "R" unusable.

13.5 If the MEAN % RSD is greater than 15%, Did the
laboratory calculate first or second order
regression fit of the calibration curve ?

ACTION: If no:
CONTRACTUAL - The initial calibration is
not valid, Document in the Data Assessment
under contract non compliance.

TECHNICAL - If % RSD is > 15.0% for any
individual target analyte, qualify positive
results for that analyte "J". When % RSD > 90%,
flag all positive results for that analyte "J"
and non-detect results for that analyte "R"
unusable.

NOTE: Analytes previously qualified "U" due to
blank contamination are still considered
as "hits" when qualifying for calibration
criteria.

YES NO N/A

13.6 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (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 in data assessments.

13.7 Do the target compounds for this SDG include Pesticides?

13.8 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?

ACTION: If DDT percent breakdown exceeds 20%:

- i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".
- ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

YES NO N/A

14.0 GC/MS Calibration Verification (Form VII)

14.1 Are the Calibration verification Forms (Form VII)
present and complete for all compounds of
interest? ☐ _____

14.2 Has a calibration verification 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 calibration
verification analysis for each instrument
used.

ACTION: If any forms are missing or no calibration
verification standard has been analyzed
within twelve hours of every sample analysis,
call lab for explanation/resubmittal. If
calibration verification data is not
available, flag all associated sample data as
unusable ("R").

	YES	NO	N/A
14.3 Do any of the SPCCs have an RRF <0.05 ?	___	<u>[]</u>	___
If YES, did the lab take corrective action as in section 7.4.4.2, page 8270C-17.	<u>[]</u>	___	
ACTION: If no: CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.			
ACTION: TECHNICAL - For any target analyte, SPCC or CCC with RRF <0.05			
1. "R" all non-detects;			
2. "J" all positive results.			
14.4 Do any of the CCCs have a %D between the initial and verification RRF which exceeds 20.0%?	___	<u>[]</u>	___
ACTION: If Yes: CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.			
TECHNICAL - All positive hits for that particular CCC must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.			
14.5 Do any target compounds have a % D between the initial and verification RRF which exceeds 20.0%?	___	<u>[]</u>	
ACTION: If yes: CONTRACTUAL - Circle all outliers in red.			
TECHNICAL - All positive hits for that particular target compound must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.			

YES NO N/A

14.6 If %D for one or more target analytes exceeds 20%,
Is the MEAN of the %D values for all analytes in
The calibration less than or equal to 20%?

ACTION: If yes:
CONTRACTUAL - The initial calibration is
valid and the average RF from the initial
calibration is used to quantitate sample results.

If no:
The initial calibration is invalid. Document
in the Data Assessment under contract non compliance.

14.7 Are there any transcription/calculation errors in
the reporting of average response factors (RRF) or
percent difference (%D) between initial and
continuing 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 document effect(s) in the
data assessments.

YES NO N/A

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration?

[] [] []

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	LowerLimit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

- ACTION:
- i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.
 - ii. Non-detects associated with IS > 100% should not be qualified.
 - iii. If the IS area is below the lower limit (<50%), 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).

YES NO N/A

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard? ☐ ☐ ☐

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Field Duplicates

16.1 Were any field duplicates submitted for 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. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

Data Validation SOP

TOC-01, Rev. 1

TOC

ENVIRONMENTAL DATA SERVICES

TOC ANALYSES VALIDATION CHECKLIST (SOP)

SITE:

DATE:

SDG:

Y N N/A

Data Completeness and Deliverables

Have any missing deliverables been received and added to the data package?

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.

Validation TOC Data

The data validation criteria outlined in this section applies to Total Organic Carbon data obtained via method Lloyd Kahn.

TOC ANALYSES

Sample Receipt/Lab Case Narrative

Are all chains-of-custody present for all samples?

ACTION: If no, prepare Telephone Record Log and contact lab for submission of missing or illegible copies.

Do chain of custody records or lab case narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?

ACTION: If any sample analyzed as a soil contains 50-90% water, all data should be flagged as estimated. If a soil sample other than a TCLP contains greater than 90% water, all data should be rejected.

ACTION: If samples were not iced when received at the lab, flag all positive results as estimated and all non-detects as "UJ".

Holding Times

Have any TOC technical holding times, determined from date of collection to date of analysis, been exceeded?

The holding time for soil is 14 days from date samples were collected.

Table of Holding Time Violations

Sample ID	Sample Matrix	Preserved?	Date Sampled	Date Lab Received	Date Analyzed
-----------	---------------	------------	--------------	-------------------	---------------

ACTION: If technical holding times are exceeded flag all positive results as estimated "J" and all quantitation limits as estimated "UJ", and document in the Data Assessment Narrative 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 time on the data. At a minimum, all results must be qualified "J", but the reviewer may determine that non-detect data are unusable (R). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

Calibration

Was correlation coefficient as calculated for the five level initial calibration ≥ 0.995 ?

Were initial and continuing calibration verifications evaluated (one for every ten samples) and found to be within the acceptance limits (80 - 120%).

ACTION: Document in the Data Assessment Narrative.

Sample Replicate Analysis

Is a duplicate summary form present?

Were replicates analyzed at the required frequency?

One per twenty field samples analyzed by each system.

ACTION: If no, prepare Telephone Record Log and contact laboratory for explanation/resubmittals. Note any impact on data quality in the Data Assessment Narrative.

Are all associated replicate values within allowable limits ($\pm 35\%$ RPD)?

ACTION: Qualify associated data as estimated if the above criterion is not met or R if in the judgement of the validator the data are unusable.

Sample Matrix Spike

Were matrix spikes analyzed at the required frequency?

One per twenty field samples analyzed per system.

ACTION: If no, prepare telephone record and contact lab for explanation/re-submittal. Note any impact on data quality in the data assessment narrative.

Are all associated matrix spike recoveries within the recovery limits (50-150%)?

ACTION: Values outside limits are qualified as estimated "J" or "R" if in the judgement of the validator the data are unusable.

Laboratory Control Sample

Was LCS analyzed at the required frequency?

One per twenty field samples analyzed per system.

ACTION: If no, prepare telephone record and contact lab for explanation/re-submittal. Note any impact on data quality in the data assessment narrative.

Are all associated LCS recoveries within the recovery limits (50-150%)?

ACTION: Values outside limits are qualified as estimated "J" or "R" if in the judgement of the validator the data are unusable.

Blanks

Is a method blank summary present?

Frequency of Analysis:

Has a reagent/method blank been analyzed for each analytical batch?

ACTION: If any method blank data are missing, prepare a Telephone Record Log and contact lab for an explanation/ resubmittal. If method blank data are not available, reject all associated positive data. However using professional judgement, the data reviewer may substitute field blank or trip blank data for missing method blank.

Contamination

Do any of the method/instrument/reagent blanks have positive results for TOC?

When applied as described below, the contaminant concentration in these blanks are multiplied by any dilution factor and corrected for % moisture.

ACTION: Prepare a list of samples associated with contaminated blanks.

ACTION: If a compound is found in a blank, but not found in the sample, no action is taken.

If a blank has a positive result for an analyte, qualify associated sample data as follows:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag the sample result as a non-detect ('U'). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detected at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5x criteria, such that a comparison of the total contamination is actually

made.

Target Analytes

Are the TOC analysis data summaries present with required header information, for each of the following:

- a. Samples?
- b. Duplicates?
- c. Blanks?

_____	_____	_____
_____	_____	_____
_____	_____	_____

Are the TOC raw data including instrument printouts and calculations in the sample package for each of the following:

- a. Samples?
- b. Duplicates?
- c. Blanks?

_____	_____	_____
_____	_____	_____
_____	_____	_____

ACTION: If any data are missing prepare Telephone Record and contact lab to obtain explanation/resubmittals. Note any impact on the data quality in the Data Assessment Narrative.

Analyte Quantitation and Reported Detection Limits

Are there any transcription/ calculation errors in data summary results?

Check at least two positive values per SDG. Were any errors found?

Are the SQL's adjusted to reflect sample dilutions and percent moisture?

_____	_____	_____
_____	_____	_____
_____	_____	_____

ACTION: If errors are large, contact lab for explanation/ resubmittal, make any necessary corrections and note errors in the Data Assessment Narrative.

Y N N/A

Field Duplicates

Were any field duplicates submitted for TOC analysis? _____

ACTION: Where both the sample and duplicate values are greater than 5 times the SQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 70 percent. Where one or both analytes of the field duplicate pair are less than 5 times the SQL, satisfactory precision is indicated if the field duplicate results agree within 2.5 times the SQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated (J).

ITEMS MARKED THROUGHOUT THE DOCUMENT WITH AN *

Data Validation SOP

HW-24, Rev. 1

Volatile Organics

SOP NO. HW-24

Revision 1

June 1999

STANDARD OPERATING PROCEDURE FOR THE VALIDATION OF ORGANIC DATA
ACQUIRED USING SW-846 METHOD 8260B (Rev 2, Dec 1996)

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) :
CAPILLARY COLUMN TECHNIQUE

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA SW-846, Method 8260B. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Chapter Two, Rev 3, December 1996; Method 8000B, Rev 2, December 1996; Method 8260B, Rev 2, December 1996; and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," February, 1994. This document covers technical as well as method specific problems; however situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

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 defined on page 25.

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

YES NO N/A

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YES NO N/A

ACTION: If all the 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".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated ($>10^{\circ}\text{C}$), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The holding time for aqueous samples is 14 days.

The holding time for soils is 10 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. If uncertain about preservation, contact the laboratory/sampling team to determine whether or not samples were preserved.

ACTION: If holding times are 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

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YES NO N/A

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any surrogate has a recovery of $< 10\%$:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and reported data?

_____ [] _____

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Samples/Matrix Spikes (CLP Form III Equivalent)

4.1 Have the volatile Laboratory Control Samples (LCS) recoveries been listed on the laboratory reporting form?

11 _____

NOTE: If the data has not been reported, then contact the laboratory/project officer to obtain the information necessary to evaluate the spike recoveries in the MS, MSD, and LCS. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

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S))))))))))Q
YES NO N/A

- | | | | | |
|-----|---|--------------|-------------|-------------|
| 4.5 | Have the volatile Matrix Spike (MS) / Matrix Spike Duplicate (MSD) recoveries been listed on the laboratory reporting form? | <u> 1 </u> | <u> </u> | <u> </u> |
| 4.6 | Were matrix spikes analyzed at the required frequency for each of the following matrices: | | | |
| a. | Water | <u> 1 </u> | <u> </u> | <u> </u> |
| b. | Soil | <u> 1 </u> | <u> </u> | <u> </u> |
| c. | Med Soil | <u> 1 </u> | | |

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, 8260B-25, Sect. 8.4.2)

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

- 4.7 How many MS/MSD volatile spike recoveries are outside QC limits?

Water

Soil

_____ out of _____

out of

- 4.8 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside OC limits?

Water

Soil

_____ out of _____

out of

ACTION: Circle all outliers with a red pencil.

- 4.9 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000B-41, Sect. 8.5.4.

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YES NO N/A

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.

NOTE: MS/MSD criteria apply only to the original sample, its dilutions, and the associated MS/MSD samples.

1. If the recovery is $>$ upper in-house limit/130%, only positive values for the affected compound(s) are flagged "J".
2. If the recovery is $<$ lower in-house limit/70%, flag positive values for the affected compound(s) "J" and non-detects "UJ".
3. If two or more MS/MSD recoveries were $<$ 10% qualify all positive results "J" and all non-detects "R".

5.0 Blank (CLP Form IV Equivalent)

5.1 Is the Method Blank Summary form present? 11

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported for samples of similar matrix, or concentration level, and for each extraction batch? []

5.3 Has a method blank been analyzed for each GC/MS system used? []

ACTION: If any method blank data are missing, take action as specified in section 3.2. If not available, use professional judgement to determine if the associated sample data should be qualified.

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

Is the chromatographic performance (baseline stability) for each instrument acceptable for the volatiles? ☐

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

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YES NO N/A

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/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

11

- 6.2 Do any trip/field/rinse/ blanks have positive results for target analytes and/or TICs?

_____ 11 _____

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (May 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 outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

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YES NO N/A

	Sample conc > CRQL but < 10x blank value	Sample conc < CRQL & <10x blank value	Sample conc > CRQL & >10x blank
Methylene Chloride Acetone Toluene 2-Butanone	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank
Other contam- inants	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

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

NOTE: The reporting of TIC compounds may or may not be required.

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 trip/field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in Data Assessment that there is no associated trip/field/rinse/equipment blank. For analytes with high concentrations, use professional judgement on qualification of these values and make note in Data Assessment. Exception: samples taken from a drinking water tap do not have associated field blanks.

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S))))))))))Q
YES NO N/A

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used. For the analysis of volatiles, the method requires requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

11 _____

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

- 8.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?
- 8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?
- 8.3 Has an instrument performance check solution (BFB) been analyzed for every twelve hours of sample analysis per instrument?(see Table 4, SW-846, page 8260B-36)

11 _____

11 _____

[1] _____

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

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If the laboratory/project officer/appropriate official cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

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S)))))))))))))q
YES NO N/A

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

8.4 Have the ion abundances been normalized to m/z 95? []

8.5 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, take action as specified in section 3.2.

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

8.7 Have the appropriate number of significant figures (two) been reported? []

ACTION: If large errors exist, take action as specified in section 3.2.

8.8 Are the spectra of the mass calibration compound acceptable? []

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

9.0 Target Analytes (CLP Form I Equivalent)

9.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate 11 _____

b. Matrix spikes and matrix spike duplicates []

c. Blanks []

d. Laboratory Control Samples 1

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S)))))))))))))q
YES NO N/A

- 9.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?
- 9.8 Do the relative intensities of the characteristic ions in the sample agree within $\pm 30\%$ of the corresponding relative intensities in the reference spectrum?

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 non detected ("U") at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

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.

10.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

- 10.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier?

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

- 10.2 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

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S)))))))))))))q
YES NO N/A

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method CROL's adjusted to reflect sample dilutions and, for soils, sample moisture?

[1]

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 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 section 3.2 above.

13.0 GC/MS Initial Calibration (CLP Form VI Equivalent)

13.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?

11 _____

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

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S))Q
YES NO N/A

13.2 Are all average RRFs > 0.050?

NOTE: (Method Requirement) For SPOC compounds, the individual RRF values must be the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

11 _ _

13.3 Are response factors stable over the concentration range of the calibration. The % relative standard deviation (%RSD) 15.0% as per SW-846, 8260B-17 Sect. 7.3.6.2.

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 15.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE:

Analytes previously qualified "U" due to blank

11 _ _

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S)))))))))))))Q
YES NO N/A

contamination are still considered as "hits" when qualifying for calibration criteria. 111 — —

13.4 Was the % RSD determined using RRF or CF?

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

13.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

14.0 GC/MS Calibration Verification (CLP Form VII Equivalent) []

14.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest? []

14.2 Has a calibration verification 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 calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

14.3 Was the % D determined from the calibration verification determined using RRF or CF?

If no, what method was used

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S)))))))))))))Q
YES NO N/A

to determine the calibration verification?
Document any effects to the case in the
Data Assessment.

14.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

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

NOTE: The above data qualification action applies regardless of method requirements.

14.5 Do any volatile compounds have a $RRF < 0.05$?

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with a red pencil.

ACTION:
If $RRF < 0.05$,
qualify all
positive

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S)))))))))))))Q
YES NO N/A

> + 100%.

3. If the IS area is below the lower limit (< - 50%), qualify all associated non-detects (U-values) "J".
4. 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" and positive results as estimated "J".

15.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

[] _____

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Field Duplicates

16.1 Were any field duplicates submitted for volatile 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 Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

DEFINITIONS

Acronyms:

BFB	-	bromofluorobenzene
BNA	-	base neutral acid
CCC	-	calibration check compound
CF	-	calibration factor
CLP	-	contract laboratory program
CRQL	-	contract required quantitation limit
% D	-	percent difference or percent drift
GC/MS	-	gas chromatography/mass spectroscopy
IS	-	internal standard
l	-	liter
LCS	-	laboratory control sample
Kg	-	kilograms
m	-	meter
mm	-	millimeter
MS	-	matrix spike
MSD	-	matrix spike duplicate
m/z	-	mass to charge ratio
QC	-	quality control
RIC	-	reconstructed ion chromatogram
RPD	-	relative percent difference
RRF	-	relative response factor
RRT	-	relative retention time
RSD	-	relative standard deviation
RT	-	retention time
SDG	-	sample delivery group
SOP	-	standard operating procedure
SPCC	-	system performance check compound
TIC	-	tentatively identified compound
TCLP	-	toxicity characteristic leach procedure
ug	-	micrograms
VOA	-	volatile organic acid

DEFINITIONS

Data Qualified Definitions:

- 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".
- NJ - 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.