USEPA REGION 4

Science and Ecosystem Support Division
Management & Technical Support Branch
Quality Assurance Section
980 College Station Road
Athens, Georgia 30605-2720

Data Validation Standard Operating Procedures

For

CONTRACT LABORATORY PROGRAM ORGANIC DATA USING

GAS CHROMATOGRAPH/ MASS SPECTROMETER

AND

GAS CHROMATOGRAPH / ELECTRON CAPTURE DETECTOR

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Periodic Review

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HISTORY OF REVISIONS

Revision Number	Issue Date	Action	Description
0.0	02/16/2014	Original	Original SOP for the Quality Assurance Section applicable to Data Review / Validation for Contract Laboratory Data Generated Under SOM02.3 for Region 4

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A Data Review Summary Narrative
B Data Review-Time Tracker
C Data Review Summary Narrative (Manual Review)
D Data Review Assessment Report (Manual Review)
E Data Package/Archive Box Inventory Form

1.0 Purpose

The United States Environmental Protection Agency (USEPA), Contract Laboratory Program (CLP) is a key provider of analytical services to the Superfund Program. The Quality Assurance Section of the Science and Ecosystems Support Division (SESD), in conjunction with the Environmental Services Assistance Team (ESAT) contractor, is responsible for providing data review and validation services in support of Superfund data collection activities performed within Region 4. The purpose of this Standard Operating Procedure (SOP) is to assist in the technical review of data generated by the contract laboratories using Statement of Work (SOW) SOM02.3, *Organic Superfund Methods, Multi-Media, Multi-Concentration*, September 2015, and revisions. This SOP follows the format and content of the *National Functional Guidelines for Superfund Organic Methods Data Review,* August 2014 (NFG), and revisions. Like the NFG, it provides guidance for areas of data review requiring considerable professional judgment. In addition, it specifies data quality requirements and procedures that are unique to the needs of Region 4, including the formats of data review reports. Procedures for entering qualified data into the Region 4 LIMS system are not included in this SOP. This document does not discuss risk assessment and the user must seek other assistance in this area. In addition, determining contract compliance is not the intended objective of these guidelines.

2.0 Applicability

This Standard Operating Procedure is applicable to the review of water, soil and sediment organic data collected using gas chromatography/mass spectrometry (GC/MS) for volatile and semivolatile organic analyses, and gas chromatography-electron capture detection (GC-ECD) for pesticides and Aroclors. Sample analyses include trace and low/medium concentrations of volatile and semivolatile organic compounds, pesticides and Aroclors.

This document provides the criteria for performing technical and quality assurance reviews of data generated by contract laboratories under the CLP SOW – SOM02.3. Criteria are based on the quality assurance/quality control (QA/QC) and technical requirements specified in Exhibit D of SOW SOM02.3. This SOP incorporates the content of the NFG and provides additional guidance to limit the use of professional judgment by taking into account region-specific data review and validation requirements and reporting formats, etc. Contract compliance or data usability issues pertinent to risk assessment activities, are not addressed in this document.

This SOP shall be followed without deviation to ensure that a consistent data review product is provided to the Region 4 - CLP Organic Task Order/Contract Officer Representative (TO/COR). If the data reviewer(s), using professional judgment, decide to take exception to any of the criteria or actions specified in this SOP, he/she must consult the TO/COR prior to making any changes. No deviations from the specified criteria or actions stipulated in this SOP will be undertaken by the data reviewer(s) unless those changes are authorized, in writing, by the TO/COR.

Authorized deviations will be documented in the data review memorandum.

3.0 Personnel Qualifications

For EPA personnel a minimum of a four-year degree from an accredited college or university in a scientific field is required. Experience in analyzing environmental samples, and in performing data review / validation is recommended.

4.0 Procedural Steps: Data Processing

Samples are collected by EPA, contractor, or state personnel and then are submitted to an assigned contract laboratory for analysis. The laboratory analyzes the samples according to specified analytical protocols, assembles a data package and an electronic data file in accordance with specifications in the contract. The original data package is submitted to the SESD, Athens, Georgia, and a copy, along with the electronic data deliverable (EDD), is delivered to the Sample Management Office (SMO) / Data Assessment Support Services (DASS) contractor.

4.1. Contract Compliance Screening

At SMO/DASS, the data package and the EDD are checked for compliance with the contract. A Contract Compliance Screening (CCS) report is issued to the region and is posted on the SMO Portal web site. The EDD is then processed electronically to evaluate QC performance against the NFG and Region 4 data quality guidelines by the Electronic data eXchange and Evaluation System (EXES). Currently, for the routine organic contracts, a SEDD Stage 3 EDD is submitted by the laboratories. Under the SEDD Stage 3 protocol, all results are recalculated using the information submitted in the EDD.

4.2. SESD Level 4 Review

4.2.1. Electronic Data Review - National Functional Guideline Report

An electronic report of the EXES review (the NFG report) is submitted to the region, along with a text file containing the results, qualified in accordance with the Region 4 data qualifier hierarchy. The data package delivered to SESD is audited for evidentiary completeness. The EXES report(s) of the electronic review (if available for all samples in the case) is examined to identify any issues that warrant further investigation The results of Performance Evaluation Samples (PES) are scored and the data are appropriately qualified. If examination of the electronic review results and/or PES scoring results reveals discrepancies and/or serious data quality issues, the reviewer may investigate by going back to the hard copy data package.

4.2.2. Selected Manual Review

Selected sample results for each case are manually evaluated for correct analyte identification and proper quantitation by checking hardcopy chromatograms, mass spectra, and/or quantitation reports. Samples selected for these additional checks are

selected using the reviewer's professional judgment in conjunction with EXES findings and the PES scoring performance. Selected manual integrations, if performed by the laboratory, are also spot-checked.

4.2.3. Addition of Qualifiers

Region 4 data qualifiers, intended to provide the customer with a more complete understanding of the factors affecting data quality, are added to the results. See Attachment A for a list of qualifiers. The up-to-date qualifier list is maintained in Element®. A report of this review completes the documentation of data quality. Finally, the results are electronically entered into the Region 4 laboratory information management system Element®.

4.2.4. Full Manual Data Review

If no electronic review was performed or the report(s) is not available, the data are manually reviewed for technical quality and for compliance with Region 4 data quality requirements, beginning with the case or SDG (Sample Delivery Group) narrative, the original unprocessed or raw data, the QC summary forms, and the sample tracking and processing information included in the package. Region 4 data qualifiers, intended to provide the customer with a more complete understanding of the factors affecting data quality, are added to the results. A report of this review completes the documentation of data quality. Finally, the results are electronically entered into the Region 4 laboratory information management system, Element®

4.2.5. Review reports and project documents are maintained by the SESD Quality Assurance Section (QAS), and the data packages are submitted to the SESD Records Room. Completed data validation reports should contain the following statement: A Stage 4 validation consisting of electronic and manual review was performed on the organic samples submitted for this case.

4.3. Review / Validation of Data

4.3.1. Holding Times/Preservation

4.3.1.1. Data qualification is not automatically performed if temperature or other preservation requirements were not met. The impact on data quality of deviations in temperature and/or other sample preservation will be evaluated after consultation between QAS and the project leader. Any appropriate qualifiers will be added after this consultation.

4.3.1.2. Holding times are evaluated based on technical holding times. (See Section 4.3.1.3.) These are determined as the age of the sample from date and time of sample collection to the data and time of sample preparation/digestion, and analysis. The contractual holding times are determined from the Validated Time of Sample Receipt (VTSR) and are used for contract compliance but will not be addressed in this SOP.

4.3.1.3. The following guidance is based on past practice in Region 4 and on the best available information on matrix holding times from 40CFR Part 136 requirements, as well as other USEPA guidance: The technical holding time is calculated from the time and date of sample collection to the date of analysis. The time and date of collection is located on the Traffic Report/Chain-of-Custody (TR/COC) form included in the analytical data package. The dates of sample preparation and analysis are located on Form 1A-OR and the raw data. If holding times are exceeded or proper preservation has not occurred, describe this in the data review summary case narrative and take the appropriate actions.

Table 4.1			
Holding Times for Semivolatiles, Pesticides and Aroclors			
Matrix	Holding Time	Action	
		Detect	Non-detect
Aqueous/water samples, TCLP/SPLP aqueous and TCLP/SPLP leachate (extraction)	Samples extracted within7 days of collection.	No action required.	No action required.
	Samples extracted at >7 days and ≤28 days of collection.	J	UJ
	Samples extracted at ≥29 days of collection.	J	UR
Sediments and TCLP/SPLP non-aqueous (extraction)	Samples extracted within 14 days of collection.	No action required.	No action required.
	Samples extracted at >14 days and ≤28 days of collection.	J	UJ
	Samples extracted at ≥29 days of collection.	J	UR
TCLP/SPLP leachates	Leachate extracted within 7 days of TCLP/SPLP procedure completion.	No action required.	No action required.
	Leachate extracted at >7 days and ≤28 days of TCLP/SPLP procedure completion.	J	UJ
	Leachate extracted at ≥29 days of TCLP/SPLP procedure completion.	J	UR
Extracts (Water Soil/Sediments) and TCLP/SPLP leachates	Analyzed within 40 days of extraction.	No action required.	No action required.
	Analyzed > 40 days and ≤60 days of extraction.	J	UJ
	Analyzed ≥61 days of extraction	J	UR

Table 4.2			
Holding Times for Volatiles			
Matrix	Holding Time	Action	
		Detect	Non-detect
Water, TCLP/SPLP aqueous filtrate, Soil, Sediment (Except ENCORE)	Non-preserved Samples analyzed within7 days of collection.	No action required.	No action required.
	Preserved Samples analyzed within 14 days of collection.	No action required	No action required
	Non-preserved Samples analyzed at >7 days and ≤14 days of collection.	J all non-halogenated aromatic results in water samples	UJ all non-halogenated aromatic results in water samples
	Samples analyzed at >14 days and ≤28 days of collection.	J	UJ
	Samples analyzed at ≥29 days of collection.	J	UR
Soil—ENCORE Preparation	Prepared > 48 hours from collection.	J	UJ
	Prepared > 96 hours from collection	J	UR
Soil—ENCORE Analysis	Samples analyzed ≤14 days from collection.	No action required.	No action required.
	Samples analyzed >14 days and ≤30 days from collection.	J	UJ
	Samples analyzed >31 days from collection.	J	UR
TCLP/SPLP-Soil	Sample Extracted ≤14 days from collection.	No action required	No action required
	Samples extracted at >14 days and ≤28 days of collection.	J	UJ
	Samples extracted at ≥29 days of collection.	J	UR
TCLP/SPLP-Leachate	Non-preserved Samples analyzed within7 days of leaching procedure completion.	No action required	No action required
	Preserved Samples analyzed within 14 days of leaching procedure completion.	No action required	No action required
	Non-preserved analyzed at >7 days and ≤14 days of leaching procedure completion.	J all non-halogenated aromatic results in water samples	UJ all non-halogenated aromatic results in water samples
	Analyzed at >14 days and ≤28 days of leaching procedure completion.	J	UJ
	Analyzed at ≥29 days of leaching procedure completion.	J	UR

4.3.2. System Performance

4.3.2.1. GC-MS Instrument Performance Check (Form 5-OR)

GC/MS instrument performance checks (IPC) are performed to ensure adequate mass resolution, identification, and to some degree, sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials. Modern quadrupole instruments are designed to meet these criteria in full-scan mode. However, they are not applied to selected ion monitoring (SIM) analyses. The IPC solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are analyzed. However, when a closing Continuing Calibration Verification (CCV) is used as the opening CCV for the next 12-hour time period, an additional ICP is not required, and the 12-hour time period begins with the injection of the CCV.

4.3.2.2. For Trace Volatile and Low/Medium Volatile analyses, after the instrument has been set to the manufacturer's recommended criteria, a 50 ng aliquot of 4-bromofluorobenzene (BFB) is introduced into the mass spectrometer (see SOW Exhibit D – Trace Volatiles § 9.2 and Low/medium Volatiles, § 9.2). The following criteria must be met before analyses of blanks, standards and samples may proceed.

TABLE 4.3

Key Ions and Ion Abundance Criteria for BFB

Mass	Ion Abundance Criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 80.0% of mass 95
95	base peak, 100% Relative Abundance
96	5.0 - 9.0% of mass 95 (see NOTE)
173	less than 2.0% of mass 174
174	50.0 - 120% of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101% of mass 174
177	5.0 - 9.0% of mass 176
NOTE	All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z

4.3.2.3. For Semivolatile analyses, after the instrument has been set to the manufacturer's recommended criteria, a 50 ng aliquot of decafluorotriphenylphosphene (DFTPP) is introduced into the mass spectrometer (see SOW Exhibit D – Semivolatiles, § 9.2).

4.3.2.4. The following criteria must be met before analyses of blanks, standards and samples may proceed:

Table 4.4

Key Ions and Ion Abundance Criteria for DFTPP

Mass	Ion Abundance Criteria
51	10.0 - 80.0% of mass 198
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	10.0 - 80.0% of mass 198
197	Less than 2.0% of mass 198
199	5.0 - 9.0% of mass 198
275	10.0 - 60.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present but less than mass 443
442	Greater than 50.0% but less than or equal to 100% of mass 198
443	15.0 - 24.0% of mass 442
NOTE	All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 maybe up to 100% that of m/z 198.

4.3.2.5. For data obtained from the CLP, the preceding criteria are evaluated as part of the CCS process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports.

4.3.2.6. If samples are analyzed without a preceding compliant instrument performance check (IPC) or are analyzed more than 12 hours after the IPC and are not analyzed within 12 hours of a preceding closing CCV that meets the opening CCV criteria, qualify all data in those samples as unusable "R".

4.3.2.7. If the laboratory has made minor transcription errors not significantly affecting the data, the data reviewer should make the necessary corrections on a copy of the form. The corrected copy should be filed in the data validation case file.

4.3.2.8. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Note this for Regional Laboratory Contracting Representative (COR) action.

4.3.2.9. For BFB: If mass assignment is in error (e.g., m/z 96 is indicated as the base peak rather than m/z 95), classify all associated data as unusable "R".

4.3.2.10. For DFTPP: If mass assignment is in error (e.g., m/z 197 is indicated as the base peak rather than m/z 198), classify all associated data as unusable "R".

4.3.2.11. If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. When applying professional judgment to this topic, the most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance. This issue is more critical for Tentatively Identified Compounds (TICs) than for target analytes

4.3.2.12. Any decision to use data associated with a BFB/DFTPP instrument performance check not meeting contract requirements should be noted in the Data Review Narrative.

4.3.2.13. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in the applicable SOW section, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note any concerns or questions regarding laboratory performance in the data review narrative for COR action. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the BFB or DFTPP peak), note this for COR action.

4.3.3. Initial Calibration (Forms 6A-OR, 6B-OR, 6C-OR, 6D-OR, 6E-OR, 6F-OR)

4.3.3.1. Volatiles (Trace and Low/Medium): Relative Response Factors (RRFs) for all volatile target compounds must be greater than or equal to 0.050 in all initial calibration levels. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 20.0% for the volatile target compounds. These criteria also apply to the SIM technique. The

reviewer should exercise professional judgment regarding possible data qualification whenever similar ICAL problems affect deuterated monitoring compounds (DMCs).

4.3.3.2. Semivolatiles: RRFs for all semivolatile target compounds must be greater than or equal to 0.050 in all initial calibration levels. The %RSD of the initial calibration RRFs must be less than or equal to 20.0% for the semivolatile target compounds. These criteria also apply to the SIM technique. The reviewer should exercise professional judgment regarding possible data qualification whenever similar ICAL problems affect DMCs.

4.3.3.3. Pesticides / Aroclors: The Percent Relative Standard Deviation (%RSD) of the Calibration Factors (CFs) for each of the target compounds must be less than or equal to 20.0%. The reviewer should exercise professional judgment regarding possible data qualification whenever similar ICAL problems affect surrogates

4.3.3.4. If the ICAL indicates that any specific compound has performed so poorly (a very high %RSD or very low response factors for the points on the ICAL) that the qualitative analysis for that individual compound is in question, qualify the results for that compound as "R" with a custom qualifier explaining the unacceptable performance.

Note: Any modified analysis (MA) accompanying a case may modify some of the preceding criteria. A copy of the MA SOW should be present in the Sample Delivery Group (SDG) data package. Refer to the MA for the specific modified analysis requirements.

Table 4.5

Initial Calibration

QC Criterion	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
Initial calibration not performed at specified frequency and sequence	Use professional judgment	Use professional judgment
Initial calibration not performed at the specified concentrations	J	UJ
(GC/MS) RRF < 0.050	J	UR
(GC/MS) RRF ≥ 0.050	No qualification	
(All) %RSD > 20%	J	UJ
(All) %RSD ≤ 20%	No qualification	

4.3.4. Continuing Calibration Verification (CCAL) (Form 7A-OR, 7C-OR, 7D-OR)

4.3.4.1. Volatiles (Trace and Low/Medium): Continuing calibration standard RRFs for both opening and closing checks for all volatile target compounds must be greater than or equal to 0.050. The Percent Difference (%D) of the sequence-beginning continuing calibration RRFs must be less than or equal to 20.0% for the volatile target compounds. For sequence-ending calibration verifications, the %D must be less than or equal to 35%. These criteria are also applied to the optional SIM technique. The reviewer should exercise professional judgment regarding possible data qualification whenever similar CCAL problems affect DMCs.

4.3.4.2. Semivolatiles: Continuing calibration standard RRFs for all semivolatile target compounds must be greater than or equal to 0.050. The %D of the sequence- beginning continuing calibration RRFs must be less than or equal to 20.0% for the semivolatile target compounds. For sequence-ending calibration verifications, the %D must be less than or equal to 35%. These criteria should be applied to SIM data as well. The reviewer should exercise professional judgment regarding possible data qualification whenever similar CCAL problems affect DMCs.

4.3.4.3. Pesticides / Aroclors: The %D of the Calibration Factors (CFs) for each of the target compounds must be less than or equal to 20.0% in both opening and closing checks. The reviewer should exercise professional judgment regarding data qualification whenever similar CCAL problems affect surrogates.

Note: Any modified analysis (MA) accompanying a case may modify some of the preceding criteria. A copy of the MA SOW should be present in the Sample Delivery Group (SDG) data package. Refer to the MA for the specific modified analysis requirements.

4.3.4.4. If, for any reason, the CCAL indicates that any specific compound has performed so poorly (a very high %D or very low response factors) that the qualitative analysis for that individual compound is in question, qualify the results for that compound as "R" with a custom qualifier explaining the unacceptable performance

Table 4.6		
Continuing Calibration Verification		
QC Criterion	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
CCV not performed at required frequency	Use professional judgment	Use professional judgment
CCV not performed at specified concentration	Use professional judgment	Use professional judgment
(GC/MS) RRF < 0.050	J	UR
(GC/MS) RRF ≥ 0.050	No qualification	
(GC/MS – Sequence Beginning) %D > + 20%	J	No qualification
(GC/MS – Sequence Beginning) %D > - 20%	J	UJ
(GC/MS – Sequence Beginning) %D ≤ 20%	No qualification	
(GC/MS – Sequence Ending) %D > + 35%	J	No qualification
(GC/MS – Sequence Ending) %D > - 35%	J	UJ
(GC/MS – Sequence Ending) %D ≤ 35%	No qualification	
(Pesticide / Aroclor) %RSD > + 20%	J	No qualification
(Pesticide / Aroclor) %RSD > - 20%	J	UJ
(Pesticide / Aroclor) %RSD ≤ 20%	No qualification	

4.3.5. Blanks (Form 1A-OR, Form 1B-OR, Form 4-OR)

Blank results are evaluated to determine the existence and magnitude of contamination resulting from laboratory activities. Only blanks associated with laboratory activities, i.e. method blanks, instrument blanks, storage blanks, etc., are evaluated during data validation. Blanks associated with field activities (i.e. trip blanks, equipment blanks, etc.) are not used to qualify sample data. However, gross contamination of field blanks should be discussed in the Data Review Narrative with regard to its impact on field sample data quality. If more than one blank is associated with a given sample, qualification shall be based upon a comparison with the associated blank having the highest concentration of a contaminant. The following are conventions that apply to evaluating blanks:

4.3.5.1. Except for common laboratory solvents and phthalates, an analyte found in a sample with a concentration five times (5X) or greater than the concentration in the blank should be considered for reporting without qualification. Any target compound reported at a concentration less than the CRQL in a sample and also present at any concentration in an associated method, instrument or storage blank will be reported at the CRQL and "U" qualified.

4.3.5.2. Target compounds detected at less than 5X the blank concentration shall be reported in samples as follows:

- If the sample result is < the CRQL, report as nondetects at the sample CRQL:
  Example: blank = 12, sample = 6, CRQL = 10, report = 10U

- If the sample result is > than CRQL, add the U flag:
  Example: blank = 12, sample = 23, CRQL = 10, report = 23U

4.3.5.3. Some analytes are more frequently found as contaminants and are considered to be common laboratory contaminants. A common laboratory contaminant found in a blank and also found in an associated sample shall be considered for reporting when present at a ratio of at least 10:1, sample to blank. The common laboratory contaminants are:

- VOA: Methylene chloride, acetone, 2-butanone
- SV: All target Phthalates
- PEST: There are no common pesticide contaminants.

4.3.5.4. The appropriate Element® qualifier (B-2, B-4, etc.) should be added whenever a positive result reported by the laboratory is "U" qualified in Element® because blank rules were not satisfied and the reporting limit has been elevated above the CRQL.

4.3.5.5. Blank values are never subtracted from reportable values.

4.3.5.6. If a sample contains an analyte that is also present in the associated storage blank, routine blank rules should be applied. Positive sample results associated with a positive storage blank result are not "J" qualified as estimated on this basis. However, the storage blank is treated analogously to the method blank and the Element® qualifier "CLP11" should be used whenever laboratory reported positive hits are "U" qualified based on storage blank contamination and the reporting limit has been elevated above the CRQL. The reviewer may qualify results as unusable (R) for gross instances of storage blank contamination.

4.3.5.7. Butoxyethoxyethanol and similar compounds are known to be common contaminants of tubing used in sampling equipment. It often occurs that the analytical method blanks do not contain the contaminant but samples and

field blanks/rinsate blanks do. It is important that the compounds be reported like any other "field contaminants", in order for the project leaders and sampling organizations to be made aware of this issue. In general, however, if a Tentatively Identified Compound (TIC) is identified in a sample and also in the associated blank, it is not reported.

4.3.5.8. The frequency and sequence of analysis for all of the required blanks should be consistent with requirements specified in SOM02.3. Exhibit D, Section 12.1.2.2 for each method.

Table 4.7

Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
Storage Blanks, Method Blanks, TCLP/SPLP LEB, Clean-up Blanks, Instrument Blanks (Not Field QC)[2]	Detected	Not detected	No qualification
	Detected	< CRQL	Report CRQL value with a U
	Detected	≥ CRQL and < 5 x blank[1]	Report result with a U
	Detected	≥ CRQL and ≥ 5 x blank[1]	No qualification

[1] 10x for common laboratory contaminants: (VOA) methylene chloride, acetone, 2-butanone; (SVOA) any of the six target phthalates.
[2] If significant contamination of field, trip, and/or equipment rinsate blanks, the data user is informed via the data review narrative and by email.

4.3.6. Deuterated Monitoring Compounds/Surrogates

4.3.6.1. Deuterated monitoring compounds (DMC) and/or Surrogates are reviewed to ensure that the results are within the acceptance criteria and, if not, that appropriate action is taken. DMC or Surrogate recovery outside the acceptance criteria must be evaluated for the effect produced on the sample results.

4.3.6.2. Since DMCs/Surrogates are associated with specific target analytes, if recovery of any DMC/Surrogate fails method criteria, results for the associated analytes are qualified as shown below. Prior to qualifying any data, the reviewer must evaluate the situation to determine whether a re-analysis of the sample exists in which better recovery was obtained, whether the analysis in question was the result of a dilution, whether the results indicate a DMC/Surrogate spiking error or final volume error (possible when all are recovered high), and whether apparent DMC/Surrogate

recovery problems are related to internal standard issues. If any of these situations occurs, the reviewer should exercise professional judgment, and may determine that no qualification for DMC/Surrogate recovery is warranted.

4.3.6.3. In general, results are qualified if DMC/Surrogate recoveries are less than 10%. However, a few semivolatile DMCs have lower recovery action limits that are less than 10%. For analytes associated with these DMCs, the qualification scheme differs. Refer Table 4.8 for details.

Table 4.8
Deuterated Monitoring Compound / Surrogate Decision Matrix

	Action	
	Detected Associated Compound	Non-detected Associated Compounds
% R > Upper acceptance limit	J	No qualification
10 % ≤ % R < lower acceptance limit	J	UJ
10 % ≥ % R > lower acceptance limit	J	UJ
10 % ≥ % R < lower acceptance limit	J	UR

4.3.7. Surrogate Standards (Pesticides / Aroclors)

For the evaluation of surrogate recovery in pesticide / Aroclor analyses, evaluate the factors discussed in Section 4.2.6, in addition to the following: If one or both of the surrogates are subject to interference, the reviewer must carefully evaluate whether it is valid to use the recovery information to qualify data. If only one surrogate appears to be free of interferences, data may be qualified based on that one surrogate alone. Note: Positive results for Aroclors increases the probability of positive interference for decachlorobiphenyl.

4.3.8. Internal Standards (Form 8A-OR)

Data qualification for internal standard performance is summarized below. If the internal standard performance criteria are grossly exceeded, note the potential effects on the data in the Data Review Narrative and for COR action.

Table 4.9		
Internal Standard Decision Matrix for CLP GC/MS Analyses		
Criteria	Action	
	Detected Associated Compounds[1]	Non-detected Associated Compounds[1]
Area counts > 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	No qualification
50% ≤ Area counts ≤ 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	No qualification
20% ≤ Area counts < 50% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	UJ
Area counts < 20% of 12-hour standard (opening CCV or mid- point standard from initial calibration)	J	UR
RT difference > 10.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	R[2]	R[2]
RT difference <10.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	

1. For compounds associated with each internal standard, for Volatile and Semivolatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation see SOM02.3, Exhibit(s) D, Section 17, Table 9.

2. Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable "R" if the mass spectral criteria are met.

4.3.9. Matrix Spike / Matrix Spike Duplicates (MS/MSD)

4.3.9.1. As advised in the NFG, data are normally not qualified based solely on the MS/MSD. However, in the absence of compelling information to the contrary, data only for the native SVOA sample used for the MS/MSD are qualified as shown below in Table 4.10.

4.3.9.2. As advised in the NFG, data are normally not qualified based solely on the MS/MSD. However, in the absence of compelling information to the contrary, qualify the native Trace VOA, Low/Medium VOA, Pesticide, and/or Aroclor sample used for the MS/MSD as shown below in Table 4.11.

Table 4.10		
Matrix Spike/Matrix Spike Duplicates (MS/MSD)--GC/MS Analyses		
Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit and >10%	J	UJ
%R < Lower Acceptance Limit and <10%	J	UR
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

Table 4.11		
Matrix Spike/Matrix Spike Duplicates (MS/MSD)—Volatiles, Pesticides, Aroclors		
Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit and >20%	J	UJ
%R < Lower Acceptance Limit and <20%	J	UR
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

4.3.10. Regional Quality Control / Performance Evaluation Samples

4.3.10.1. Performance Evaluation Samples (PESs) are incorporated into each project, for each set of analytes and each matrix, as needed. For larger projects, including sampling efforts extending for more than one week, multiple sets of PES may be used. The laboratories are required to prepare and analyze the PES with the field samples of the associated case and SDG.

4.3.10.2. If the PES is not prepared and/or analyzed concurrently with some or all samples of the case, the reviewer may decide that it is not appropriate to use

the PES for data qualification. Table 4.12 summarizes data qualification based on PES scoring results.

4.3.10.3. Sometimes spiked analytes are not evaluated by scoring software and data qualification is not made based on PES scoring when either lower limits do not exist or the analyte was not evaluated. The reviewer may describe instances in the narrative when the laboratory failed to identify a spiked compound for which lower limits did not exist but PES database statistics suggest that the analyte should still have been identified by the laboratory.

4.3.10.4. All analytes which are scored as PES contaminants, either less than or greater than the CRQL, are treated as method blank contaminants, applying standard blank rules described in section 4.2.5 of this SOP.

4.3.10.5. Sample TICs are not qualified based on TIC PES scoring.

4.3.10.6. If only one set of PES is included in a case, all samples will be qualified based on the PES scoring. If multiple sets of PES are included, all data for the associated sampling week will be qualified based on the PES scoring.

Table 4.12

PES Scoring Matrix for CLP Organic Analyses

PES scoring	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
Within warning limit	No qualification	No qualification
Action high or warning high	J	No qualification
Warning low	J	UJ
Action low or analyte missed	J	UR

4.3.11. Tentatively Identified Compounds (TIC)

4.3.11.1. Examine the Form 1B-OR for all identified TICs. Mass spectra of TICs are not routinely reviewed necessitating that all TIC results have the Element® qualifier "CLP15" or "TIC results Reported by Lab – IDs Not verified" attached. Eliminate all TICs reported by the laboratory with the "B" or "A" qualifier; or categorized by the laboratory as laboratory artifact, column bleed, etc. Eliminate all VOA or Semivolatile Extractable target analytes reported as TICs by the laboratory whenever results for that target analyte have also been reported. Target pesticides identified as TICs are not retained when pesticide fraction also reported; however, this information can be used as part of GC/MS confirmation.

4.3.11.2. A list of TICs reported by the laboratory is included in the Universal Deliverable spreadsheet posted on the SMO portal for each SDG. These spreadsheets are edited according to the paragraphs below.

4.3.11.3. If any straight-chain alkanes, branched alkanes, cyclic alkanes, or "total alkanes" are listed on Form 1- TIC, combine and report these on one line as "Petroleum product" with no quantity, and qualify as "N, Z-01, CLP15".

4.3.11.4. Eliminate any TIC that is less than the CRQL for the sample. Professional judgment may be applied if non-target analytes of known environmental concern or pesticide/Aroclor target analytes are identified at less than the CRQL.

4.3.11.5. For the VOA TICs, any TIC with more than 10 carbons is assumed to belong in the semi-volatile category, and is not reported. Similarly, for the semi-volatiles, any TIC with fewer than 10 carbons is assumed to belong in the volatile category and is not reported.

4.3.11.6. Change any unfamiliar TIC with a name that is incomplete (i.e., too long for the field and therefore not completely reported) or is missing a CAS number to "Unidentified compound(s)". Generally, all TICs reported as a generic class (i.e., unknown amide) are included as part of the unidentified compound total.

4.3.11.7. Combine any repeatedly named TICs onto one line, and add the quantities. This includes "Unidentified compound(s)". Do not add phrases like "3 isomers". Similarly named compounds with different structural formulae will not be combined (i.e., combine multiple entries of 1,2,4-trimethylnaphthalene, but report separately a single 2,4,6-trimethylnaphthalene).

4.3.11.8. Qualify all identified TICs as "NJ, CLP15" and qualify the "Unidentified compound(s)" as "J, CLP15".

4.3.11.9. Do not report an MRL for any identified TIC.

4.3.11.10. Each sample should have at least one TIC entry that reads, "Tentatively Identified Compounds" with a result that matches the sample CRQL. If no other TIC entries are to be reported for the sample, this entry is reported with a MRL that also matches the sample CRQL, qualified "U". If other TICs are reported, do not report the "Tentatively Identified Compounds" entry. The Element® system will accept a tilde, "~" (with no comma separator) in the qualifier cell as a switch to prevent reporting an analyte. In this case, the qualifier field will look like "~U" on the spreadsheet template.

4.3.11.11. As with the target analyte data import templates, re-save the TIC spreadsheet in the appropriate Excel 95 format to be compatible with other software systems.

4.3.12. Target Analyte Quantitation and Reported Contract Required Quantitation Limit

4.3.12.1. Any reported target analyte having a concentration in the injected extract dilution less than the lowest standard on the calibration curve is qualified "J" (CLP01).

4.3.12.2. Any reported target analyte having a concentration in the injected extract dilution greater than the highest standard on the calibration curve is qualified "J" (CLP02).

4.3.12.3. For soil/sediment samples that are high in moisture (i.e., < 30% solid), evaluation of the presence of each analyte depends on the anticipated interaction between the analyte and the total matrix as well as how the sample was processed.

4.3.12.4. If a soil/sediment was processed by eliminating most of the water, analytes that are highly water soluble under ambient conditions may be severely impacted such that their presence cannot be completely evaluated.

4.3.12.5. The reviewer may use professional judgment when evaluating results from samples with high moisture content, but typically uses Table 4.13.

Table 4.13		
Percent Solids Actions for Non-Aqueous Samples		
Criteria	Action	
	Detects	Non-Detects
%Solids ≥30%	No Action Required	No Action Required
%Solids < 30% but > 10%	J, CLP42	UJ, CLP42
%Solids ≤10%	J, CLP42	UR, CLP42

4.3.13. Pesticides / Aroclors – Additional Requirements

For the Region 4 QAS, the following special data qualification procedure for single component and multiple components pesticides/PCBs shall be followed.

4.3.13.1. Follow the National Functional Guidelines for Laboratory Control Sample (LSC) and Resolution check criteria and qualification

4.3.13.2. Single component pesticides are routinely analyzed on two dissimilar GC columns. Quantitation values are obtained from both GC columns and the percent difference (% D) calculated. The contract laboratory reports the lower of these two quantitation values. If the percent difference between the two columns exceeds 25%, the laboratory assigns the "P" data qualifier flag.

4.3.13.3. The reviewer may use professional judgment when evaluating pesticide analytes reported with a percent difference that exceeds 25%, but typically uses Table 4.14.

4.3.13.4. Multiple component analytes such as toxaphene and PCB Aroclors are not qualified for percent difference between columns, since their qualitative identification is based on peak pattern matching. However, the reviewer should exercise professional judgment when evaluating positive hits for Toxaphene and Aroclors whenever large percent differences exist.

4.3.13.5. It may be appropriate and necessary to manually compare sample and standard chromatograms for at least some of the samples in order to verify accuracy of laboratory's identification.

Table 4.14		
Qualification of Pesticides/PCBs Based on % D Between Columns		
Criteria	Single Component Pesticide	Multi-components (Toxaphene, PCBs)
%D > 25% ≤ 70%	N,CLP12	No Qualification
%D > 70 and < CRQL	"U" @ CRQL	No Qualification
%D > 70 and > CRQL	U,CLP13	No Qualification

4.3.13.6. Each sample extract should have been diluted and re-analyzed if the initial results exceed the established calibration range.

4.3.13.7. Any single component analyte with a concentration > 50 ug/L for water and > 1700 ug/kg for soil should be confirmed by GC/MS and flagged "C" on the Form 1A-OR by the laboratory, if confirmed. (The concentration triggers for GC/MS confirmation are 1250 ug/L and 42000 ug/kg for Toxaphene and 100 ug/L and 3300 ug/kg for Aroclors.) The reviewer should examine the procedure followed for at least one sample to verify that the requirements in Exhibit D- PEST, Section 11.1.2 have been met and the confirmed result should have the Element® qualifier "D-1" attached. Generally, pesticides identified by the laboratory as semivolatile extractable TICs only (i.e., no GC/MS pesticide standard injected to establish retention time) are not considered by the reviewer to be confirmed. If no confirmation was performed, note the fact in the Data Review Summary Narrative. If the laboratory performed a GC/MS confirmation, but could not confirm the presence of the suspected pesticide, then qualify the reported Element® result with "U, D-4" at the GC/MS reporting limit. Note and discuss this issue in the review narrative.

4.3.14. Data Qualifier Definitions

4.3.14.1. Region 4 applies qualifiers to the organic data as defined in the SOWs referenced above, and in the National Functional Guidelines with the exception of the qualifiers, B, E, and P, which are not used in Region 4 data reporting.

4.3.14.2. The definitions in Table 4.15 provide brief explanations of the qualifiers assigned to results during the electronic data validation process. An additional set of data qualifiers is applied as needed to provide further information to the data user about data quality. See Attachment A.

Table 4.15	
Qualifier Definitions	
C	The sample results are confirmed by other analytical techniques including analysis of a reference standard.
J	The analyte was positively identified, but the associated numerical value is an estimated concentration of the analyte in the sample based on its associated quality measures.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
R	The sample results are rejected due to serious deficiencies in the ability to analyze sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	The analyte was analyzed for, but was not detected above the method detection limit as defined in the SOWs.

5.0 DATA REVIEW DOCUMENTATION

A Data Review Document shall be prepared to document the organic data package validation. The document includes the Review Summary Narrative, the Time Tracker form, Performance Evaluation Sample (PES) Scores from the secure SPS-Web site, a copy of the spreadsheet used for data import into the Element® data system, and the EXES - NFG. These reporting elements are described below, and examples are included as attachments to this SOP.

5.1. Document Contents:

5.1.1. Organic Data Review Summary Narrative (Attachment B) - This narrative is in a letter format summarizing the information pertinent to the samples, analytical methods, highlights of findings, and a brief assessment of the overall data quality. Descriptions of major data quality issues and their impact on overall data quality should be presented. Attachment B is an example narrative for data review that was assisted by electronic data review. Attachment D is an example of a full manual review report.

5.1.2. Time tracker (Attachment C) - This form is for recording the time line and efforts at different stages of the data review process. This form must be utilized and included in the data review documents for CLP data. Any unusual circumstance encountered for the samples reviewed should also be documented here, including any factors affecting the level of effort required to complete the review or the timeliness of the product. The time tracker should include the peer review information as part of the validation package requirements. See Attachment C for an example.

5.1.3. PE Score (SPS-Web) - This form is generated by the SPS-Web program to report evaluation of the PES(s) results associated with the data package. Include the "EPA" and "laboratory" form versions as attachments to the data validation memorandum.

5.1.4. Excel® Spreadsheet - The reviewed data with final assigned qualifiers attached, (if any) as they appear in Element®, are included in the data review report as an Excel® spreadsheet. This should also have evidence of peer review

5.1.5. EXES Reports - Each EXES NFG report is downloaded as a self-expanding executable file and distributed to the data review team. Two (2) copies of CADRE/EXES reports should be printed for each SDG. One copy is placed in the project file and the other copy is used by the data reviewer for data review notations. The marked up copy is submitted to the Organic data validation TO/COR along with the Data Validation Report.

The EXES NFG report is organized by SDG and includes the following elements:

Extracted EXES Files	Contents
Final Qualifier Results	Tabulated sample results with DASS-assigned qualifiers by analytes per sample per protocol (method or fraction), such as Volatiles (VOA), semivolatiles (SV), pesticides (PEST), or aroclors (ARO).
Tentatively Identified Compounds (by samples and protocols)	A summary of the reported TICs for VOA and SV
Analytical Sample Listing (by protocols)	A summary of samples included in the SDG with dates and time of sample collection and analyses and analytical instruments used
Organic Analytical Sequence (by protocols)	A summary of the standards and samples analyzed in an instrumental analytical sequence defined by the SOW
Identification Summary	A summary per sample for the detected single component and multiple component pesticides with the percent difference (% D) of results between the analytical and confirmation columns
Data Review Results (by protocols)	Summary of evaluation/qualifications of each of the data quality control measures (calibrations, holding time, IPC/Tune, internal standards, laboratory blanks, matrix spikes, detection limits, SMC, surrogate, system performance, and data review criteria set options) and explanations of action taken to result in the sample data reported in the "Final Flag Results" section.
Initial Calibration Data Summary, RRF & CF Report	A summary of RRF & CF with %RSDs
Continuing Calibration Data Summary GC/MS	A summary of RRF with %Ds
Continuing Calibration Summary GC	A summary of CF with %Ds

5.2. Recording and Reporting of Data

Validated results along with data qualifiers are entered into the SESD laboratory information system Element®. Data results along with the Case narrative are reported from Element®.

5.3. Data Package Archives

5.3.1. The CLP data packages must be properly archived for future reference. For each data package, the form "Record Transfer Inventory" must be utilized to record the proper information pertinent to the content. All of the raw data, EXES reports, and any communication records must be included. Multiple data packages from different projects may be stored in one single box if sufficient space is available.

5.3.2. Data packages for one Case that are stored in multiple boxes must be clearly identified on the Record Transfer Inventory forms. An appropriate numbering system must be maintained to ensure that each box containing the data review supporting documentation, have a unique archive number.

5.3.3. A copy of the inventory form should be kept within the box and an additional copy filed in a centralized system. The data package boxes shall be maintained under the custody of SESD and archived per Region 4 guidelines. The Record Transfer Inventory Form is provided in Attachment E.

6.0 References

U.S. Environmental Protection Agency, Statement of Work for Organic Superfund Methods, Multi-Media, Multi-Concentration, SOM02.3, September 2015.

U.S. Environment Protection Agency, Contract Laboratory Program, National Functional Guidelines for Superfund Organic Methods Data Review, August 2014.

Attachment A

Region 4 LIMS
Element® Qualifier Definitions

as of Thursday, June 19, 2014 at 02:09:13 p.m.

A	The analyte was analyzed in replicate. Reported value is an average value of the replicates.
B-1	Analyte is found in the associated blank as well as in the sample (CLP B-flag).
B-2	Reporting level elevated due to trace amounts of analyte present in the method blank.
B-3	Level in blank does not impact data quality
B-4	Level in blank impacts MRLs.
B-5	Qualitative evidence of contamination in the blank at a concentration less than the MDL.
C-2	Improper sample container used
C-6	Sample aliquot taken from VOA vial with headspace (air bubble greater than 5-6 mm diameter).
C-7	Sample container leaked during transport.
C-8	Coring device sampler received by the laboratory unlocked
CL-1	BOD result estimated - Sample exhibited evidence of toxicity
CL-2	DOC result higher than TOC result
CLP01	Concentration reported is less than the lowest standard on calibration curve
CLP02	Concentration reported is greater than the highest standard on calibration curve
CLP03	Baseline instability in calibration or preparation blanks
CLP04	Analyte reported as potential false positive (% RSD > 20%, and result > MDL, but < CRQL)
CLP05	CLP ICP-MS method does not include: Al, Ca, Fe, Mg, K, & Na
CLP09	MRL elevated due to baseline instability.
CLP10	2,3,7,8-TCDF confirmed by second column.
CLP11	Storage blank contaminant
CLP12	Difference between GC columns above method warning limit
CLP13	Difference between GC columns above method action limit
CLP14	The analysis did not indicate the presence of the analyte. The data is rejected and the reported value is the Reporting Limit. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.
CLP15	TIC Results Reported as Identified by Lab - IDs Not Verified
CLP16	Initial Calibration Response Erratic

CLP17 Initial Calibration Relative Response Outside Method Control Limits

CLP18 Estimated Maximum Possible Concentration (EMPC) Reported

CLP20 Matrix Spike Recovery < 30%

CLP21 %RSD >20% for ICP Multiple Exposures

CLP22 Suspected interference from Al and/or Fe as noted in contractor ICSA solution

CLP23 Suspected over correction from Al and/or Fe as noted in contractor ICSA solution

CLP24 Result has not been confirmed by second column analysis.

CLP25 PE sample recovery scored as warning-low.

CLP26 PE sample recovery scored as warning-high.

CLP27 PE sample recovery scored as action low.

CLP28 PE sample recovery scored as action high.

CLP29 Matrix Spike recovery greater than 125%.

CLP30 Stage 4 validation consisting of electronic and manual review was performed for this data.

CLP31 Stage 4 validation consisting of full manual review was performed for this data.

CLP32 Continuing Calibration Relative Response Outside Method Control Limits

CLP33 Poor Chromatography - Split Peaks and/or Poor Peak Shape Present

CLP34 Percent recovery for the Post Digestion Spike was below the lower acceptance limit.

CLP35 Percent recovery for the Post Digestion Spike was above the upper acceptance limit.

CLP36 Identification/Concentration of analyte not confirmed by ICP-MS.

CLP37 ICP/MS tune not performed.

CLP38 ICP/MS tune not within required limits.

CLP39 Matrix Spike Recovery < 50%

CLP40 Samples received by laboratory above 6 C.

CLP41 Since 2,3,7,8-TCDD and 1,2,3,7,8-PeCDD both have Toxicity Equivalent Factors of 1.0 as assigned by the WHO, the R qualifier assigned to these two congeners following data validation were carried through to the TEQ calculated value at any concentration.

CLP42 Sample results are estimated "J" or "UJ" due to percent moisture content between 70%-89%, or sample results Rejected "R" due to moisture content greater than or equal to 90%.

CR [Custom Value]

D-1 The analyte is determined to be present. The presence of the analyte was confirmed by GC/MS.

D-2 Due to Matrix Interference, the sample cannot be accurately quantified. The reported result is estimated.

D-3 Sample diluted due to the presence of high levels of non-target analytes resulting

in elevated reporting limits.

D-4 MRL elevated due to interferences.

D-5 Estimated quantitation for one or more individual constituents comprising >10% of the total.

D-6 Presence of analyte confirmed by ICP-MS.

***EA-1** Skewness - [Custom Value] Right Skewed

***EA-2** Skewness - [Custom Value] Left Skewed

***EA-3** Kurtosis - [Custom Value] Mesokurtic

***EA-4** Kurtosis - [Custom Value] Leptokurtic

***EA-5** Kurtosis - [Custom Value] Platykurtic

***EA-6** Nitrogen

***EA-7** Phosphorus

***EA-8** Nitrogen + Phosphorus co-limited

***EA-9** Not Determined

***EA-A** Absent

***EA-P** Present

F-2 No flash detected up to 60° C (140° F).

H-1 Recommended holding time exceeded

H-2 PT or QC sample. Holding time met when calculated from preparation of whole volume.

H-4 Holding time expired prior to receipt by laboratory.

H-5 ASB-defined holding time exceeded.

H-6 Sample originally analyzed within holding time; some QC requirements not met. The reported result is from a second analysis performed for confirmation which occurred after the holding time expired.

H-7 Recommended preparation holding time exceeded

H-8 Recommended analytical holding time exceeded

I-5 Mixture of Aroclors in sample; predominant Aroclors reported

J The identification of the analyte is acceptable; the reported value is an estimate.

K The identification of the analyte is acceptable; the reported value may be biased high. The actual value is expected to be less than the reported value.

L The identification of the analyte is acceptable; the reported value may be biased low. The actual value is expected to be greater than the reported value.

MRL-1 MRL verification for Potable Water matrix (Drinking Water)

MRL-2 MRL verification for Non-Potable Water matrix

MRL-3 MRL verification for Soil matrix

MRL-4 MRL verification for Tissue matrix

MRL-5 MRL verification for Air matrix

MRL-6 MRL verification for Waste matrix

MRL-7 MRL Verification for other matrices (bottle blanks, etc)

MRL-8 MRL verification result less than the LOD.

MRL-9 MRL verification for TCLP matrix

N There is presumptive evidence that the analyte is present; the analyte is reported as a tentative identification.

NA Not Analyzed.

NA-1 Not Analyzed. Sample lost during preparation or analysis.

NA-10 Not Analyzed. Sample container broken when received.

NA-11 Not Analyzed. Sample container broken in laboratory.

NA-12 Sample has no measureable alkalinity. Original sample pH is less than 4.5.

NA-13 Not Analyzed. Screening indicates no possibility for a reportable acidity value.

NA-2 Not Analyzed. Canister received at 760mm pressure.

NA-3 Not Analyzed. Insufficient sample received for analysis.

NA-4 Not Analyzed or Reported due to Interferences.

NA-5 Not Analyzed. Cannot exceed TCLP regulatory levels based on Total Scan analyses.

NA-6 Not Analyzed. Sample did not flash. Percent Water and Percent Alcohol determinations not required.

NA-7 Not Analyzed. Sample is not aqueous. Percent Alcohol determination not required.

NA-8 Not Analyzed. Placeholder sample for DOC or other QC.

NA-9 Not Analyzed. No sample container received.

NJ Presumptive evidence that analyte is present; reported as a tentative identification with an estimated value.

P-2 Sample at improper pH

P-3 Sample received unpreserved

P-4 Sample received at pH > 2.

P-5 Sample received at pH < 12.

P-6 Incorrect reagent or technique used to preserve sample.

P-7 Sample received at pH above preservation requirements.

P-8 Sample received at pH below preservation requirements.

Q-1 The original extraction of this sample yielded QC recoveries outside control limits. It was re-extracted after the recommended maximum holding time.

Q-2 Result greater than MDL but less than MRL.

Q-3 Instrument not calibrated for all constituents of the total concentration result.

Q-4	Greater than 40 % difference between primary and confirmatory GC columns
Q-5	Serial dilution precision outside method control limits
Q-6	Appropriate QC not prepared and/or analyzed with this sample.
QC-1	Analyte concentration low in continuing calibration verification standard
QC-2	Analyte concentration high in continuing calibration verification standard
QC-3	Analyte calibration criteria not met
QC-4	Result greater than the highest point on the calibration curve
QC-5	Calibration check standard less than method control limits.
QC-6	Calibration check standard greater than method control limits.
QI-1	Internal standard was outside of method control limits.
QL-1	Laboratory Control Spike Recovery less than method control limits
QL-2	Laboratory Control Spike Recovery greater than method control limits
QL-3	Laboratory Control Spike Precision outside method control limits
QL-4	Laboratory Control Sample recovery less than 10%
QM-1	Matrix Spike Recovery less than method control limits
QM-2	Matrix Spike Recovery greater than method control limits
QM-3	Matrix Spike Precision outside method control limits
QM-4	Matrix Precision outside method control limits
QM-6	Matrix Spike Recovery less than 10%
QR-1	MRL verification recovery less than lower control limits.
QR-2	MRL verification recovery greater than upper control limits.
QS-3	Surrogate recovery is lower than established control limits.
QS-4	Surrogate recovery less than 10%
QS-5	Surrogate recovery is higher than established control limits
R	The presence or absence of the analyte can not be determined from the data due to severe quality control problems. The data are rejected and considered unusable.
SP-2	Elevated Reporting Limits due to limited sample volume.
T-0	No temperature blank present for cooler this sample was received in.
T-1	Sample received in cooler with temperature blank greater than 6 degrees C.
T-2	Sample received in cooler with temperature blank lower than recommended method limit.
T-3	Sample received unfrozen. Preservation requirement not met.
T-4	Samples received at ambient temperature.
TC-1	Cannot exceed TCLP regulatory levels based on Total Scan analyses
TC-6	Ambient lab temp. during TCLP dropped below method limits.

TC-7	Ambient lab temp. during TCLP exceeded method limits on the high side.
TC-8	Results for TCLP are greater than or equal to value reported. See Method 1311 Section 1.3.
U	The analyte was not detected at or above the reporting limit.
X-1	Non-target analyte
X-2	Matrix interference precludes recovery calculation
X-3	Co-eluting/interfering target analyte(s) preclude recovery calculation
X-4	Recovery not calculated due to CCV outside acceptance criteria
X-5	Spiked incorrectly.
X-6	Exclude value from QC data base. Refer to custom remark for details.
X-CH6	Sample is reducing in nature. Should not support hexavalent chromium
X-PDS	Post Digest Spike
XB-1	Carryover from high level sample
XD-1	Duplicate results less than MRL
XD-2	Duplicate results less than 5X MRL
XM-1	Sample background/spike ratio higher than method evaluation criteria
XS-1	Surrogate diluted out due to high analyte concentration
XS-2	Surrogate diluted out due to matrix interference
XS-3	Surrogate not reported due to matrix interference
Y-1	Data reported by memo
Y-2	Data should be limited to screening purposes only
***Z-01**	[Custom Value]
pH-1	pH is equal to or less than reported result.
pH-13	pH is equal to or greater than reported result.

** Qualifiers in **blue** and flagged with an asterik are Retained Qualifiers and will become your analytical result if used.*

Attachment B: Data Review Summary Narrative Example

May 14, 2014

Ms. Nancy Seabolt
Environmental Protection Agency, Region 4
Science and Ecosystem Support Division

980 College Station Road
Athens, GA 30605-2720

SUBJECT: **Organic Data Review and Validation**

Project No.:	14-0159
Case No.:	44267
Work Order No(s).:	C141901, C141902
ESAT TDF No.:	14T0532
EPA Sample Nos.:	C141901-01– 19, C141902-01
Sampling date(s):	04/12-13/14
Organic CLP Analyses:	XXXXXXXXXXXXXXXXXX
Data for Site:	XXXXXXXXXXXXXXXXXX
Analyses Conducted:	Low/Medium Volatiles

Dear Ms. Seabolt:

The ESAT Work Team reviewed data for one water trip blank and eighteen soil samples analyzed for Low/Medium Volatiles only, submitted in one sample delivery group (SDG). The laboratory was submitted one soil performance evaluation sample (PES) for this case.

The samples were collected between 04/12/14 and 04/13/14, were received by the laboratory on 04/14/14, and the data package was received on 05/09/14 by the USEPA Quality Assurance Section, Region 4 SESD/MTSB.

The laboratory analyzed all samples beyond both the contractual and technical holding time limits. The laboratory acknowledged in their narrative that contractual holding time limits were not satisfied, but did not provide any explanation. Accordingly, the results for all soil samples (C141901-02-19) were "J" qualified (H-8). Sample C141901-01 was a water trip blank and was not qualified on the basis of technical holding time.

All results associated with erratic initial and/or continuing calibration performance were "J" flagged with the appropriate Element® qualifier (CLP16 and/or QC-1/QC-2). Deuterated monitoring compounds (DMCs) were used as surrogates in each sample for GC/MS analysis to monitor extraction efficiency.

Pertinent data quality factors are discussed below.

1. The laboratory scored within warning limits for all spiked analytes in the soil PES except

for vinyl chloride, which was scored as action high, and dibromochloromethane, which was scored as warning low. Data qualification was not required for vinyl chloride since all sample results were nondetects. All dibromochloromethane results were "J" qualified (CLP25). Additionally, both chloromethane and methyl acetate were scored as PES contaminants at more than the Contract Required Quantitation Limit (CRQL). Both chloromethane and methyl acetate were treated as method blank contaminants during data qualification. The laboratory did not identify the spiked Tentatively Identified Compound (TIC) 2,2-dichloropropane. Data qualification was not made on the basis of TIC identifications.

2. Poor responses were obtained for 1,4-dioxane for all calibration levels analyzed. All results were nondetects for this compound and were "UR" qualified (CLP17, CLP32). Additionally, poor responses were obtained for acetone and/or bromomethane for some initial and continuing calibration standards analyzed. All associated results were nondetects for acetone and bromomethane and were "UR" qualified (CLP17 and/or CLP32).

3. High DMC recoveries were observed in samples C141901-03 and 15. All associated positive results were "J" qualified (QS-5).

4. Low DMC recoveries were observed in samples C141901-02, 05, 11, and 14. The associated vinyl chloride results were "J" qualified (QS-3).

5. Low area counts were observed for all internal standards in both the original and reanalysis of sample C141901-03. All results were "J" qualified for this sample (QI-1).

A Stage 4 validation consisting of both electronic and manual review was performed on the organic samples submitted for this case. Please refer to the attached EXES reports, the PES scoring report, and the attached final result spreadsheets for further details. If you have any questions, please contact this office.

Very Truly Yours:

Michael E. Keller

Chemist (Data Validation Team Lead)
Alion Science and Technology

Attachment C: Data Review Time Tracker Example

TIME TRACKER
VERSION 4.0

CASE # :	44267	PROJECT #:	14-0159	TDF NO:	14T0532
LAB METHOD(S):				LIMS METHOD CODE(S):	NA
NUMBER OF SAMPLES:	20	VALIDATED TIME OF SAMPLE RECEIPT (VTSR):	04/14/14	DUE DATE:	06/02/14
SITE NAME:				SITE ID:	
Box Archival Inventory	14-025				
Work Order No(s).	C141901, C141902	PROGRAM:	SARA	TASK ORDER:	4

	STAGE OR PERSON	INITIALS	DATE ACCEPTED	COMPLETION DATE	# Hours
1.	Received by EPA OQA		05/09/14		
2.	Evidentiary Audit	TM	05/12/14	05/13/14	3.5
3.	Data Review	MEK	05/14/14	05/15/14	10
4.	Secondary Review/Spreadsheet Verification (return marked up copy of spreadsheet, edited hardcopy, and time tracker to reviewer)				
5.	Final Overview (memo, entry, content)				
6.	Element Import				
7.	Task Monitor (TOPO)				

Sample and Method Information

EPA Samples # (Separated by methods for cases with multiple lab methods applied)	V	SV	Pest	PCBs	PCDD/ PCDF	Metals		CN	OTHERS (specified)
						ICP/AES	ICP/MS		
C141901-01-19	19								
C141902-01	1 PE								

Notes/Comments: Review took some additional time since the laboratory did not arrange calibration standards chronologically as required by the SOW. Please note that EXES did not qualify results based on missed technical holding times as it should. CCS reports did show missed contractual holding times as a defect.

Attachment D: Data Review Assessment Report (Manual Review) Example

Attachment 4 Data Review Assessment Report (Manual Review) Example

Data Quality Assessment Record (DQAR)

Review Date: 05/27/08 Analyses: FL-PRO Matrix: Water Project #: 08-0381

SDG /Lab File: ACW-C2-MW01, ACW-C6-MW05, ACW-C6-MW04

Laboratory : XXXXXXXXXXXXXXXXXXXXXXXX

Site Name: XXXXXXXXXXXXXXXXX

Check One: EPA ____ ESAT ____ CLP ____ Other (specify) **Non-CLP (RAS)**

Signature:

Reviewer

Sample Numbers:

Water:

C081705-01 (ACW-C2-MW01) C081705-13 (ACW-C4-MW06) C081705-25 (ACW-C9-MW04) C081705-37 (ACW-C6-MW03)
C081705-02 (ACW-C2-MW02) C081705-14 (ACW-C5-MW01) C081705-26 (ACW-C9-MW05) C081705-38 (ACW-C7-MW01)
C081705-03 (ACW-C2-MW03) C081705-15 (ACW-C5-MW02) C081705-27 (FD-06) C081705-39 (ACW-C7-MW02)
C081705-04 (ACW-C2-MW04) C081705-16 (ACW-C5-MW03) C081705-28 (ACW-C10- C081705-40 (ACW-C7-MW03)
C081705-05 (ACW-C2-MW05) C081705-17 (ACW-C5-MW04) C081705-29 (ACW-C10- C081705-41 (ACW-C7-MW04)
C081705-06 (ACW-C2-MW06) C081705-18 (ACW-C5-MW05) C081705-30 (ACW-C10- C081705-42 (FD-07)
C081705-07 (ACW-C3-MW01) C081705-19 (ACW-C5-MW06) C081705-31 (ACW-C10- C081705-43 (FD-08)
C081705-08 (ACW-C3-MW02) C081705-20 (ACW-C6-MW04) C081705-32 (ACW-C10- C081705-44 (FD-09)
C081705-09 (ACW-C3-MW03) C081705-21 (ACW-C6-MW05) C081705-33 (ACW-C4-MW01)
C081705-10 (ACW-C4-MW03) C081705-22 (ACW-C9-MW01) C081705-34 (ACW-C4-MW02)
C081705-11 (ACW-C4-MW04) C081705-23 (ACW-C9-MW02) C081705-35 (ACW-C6-MW01)
C081705-12 (ACW-C4-MW05) C081705-24 (ACW-C9-MW03) C081705-36 (ACW-C6-MW02)

I. SUMMARY OF PROBLEMS AND COMMENTS:

A summary of deficiencies noted for the method used to generate data for this project is presented below. For the purposes of this review, the QC limits specified in the analytical method have been applied to the data. Data qualifiers recommendations are made in accordance with the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic and Organic Data Review* (Functional Guidelines), and the Region 4 SOP, *Data Validation Standard Operating Procedures for Contract Laboratory Program Routine Analytical Services* (R4DVSOP), Rev. 2.1.

Surrogate o-terphenyl was above QC limits in sample C081705-19 (ACW-C5-MW06). The positive DRO result was "J" qualified in this sample.

II. Data Quality Assessment **(An explanation for any "no" answer must be provided)**
? = see remarks

1. Summary:	Yes	N/A	No
Were all requested analyses performed?	X		
Were all required QC checks performed?	X		
Were all required documents present?	X		
Were requested detection limits met?	X		

Remark:

2. Holding Times:	Yes	N/A	No
VOA/BNA/PEST prepared within 14 days of sampling (7 days for VOA aromatics in non-preserved samples)?	X		
PCDD/PCDF extracted within 30 days of sampling?		X	
Extracts analyzed within 40 days of extraction?	X		
Were all samples/extracts properly preserved?	X		
For TCLP: Were RCRA TCLP holding times met?		X	

Remark:

3. GC/MS Tuning:	Yes	N/A	No
Were PFK/DFTPP/BFB criteria met?		X	
Pesticides: Were standards run in proper sequence?		X	
Combined DDT/Endrin Breakdown acceptable?		X	
Retention time windows defined?		X	

Remark:

4.1 **Initial Calibration:**	Yes	N/A	No
Were %RSDs acceptable?	X		
Were RRFs acceptable?		X	
Was S/N acceptable?		X	
Were PCDD/PCDF ion ratios acceptable?		X	

Remark:

4.2 **Continuing Calibration:**	Yes	N/A	No
Were %RSDs acceptable?	X		
Were RRFs acceptable?		X	
Were PEST cont. calib. factors met?		X	
Was PCDD/PCDF S/N acceptable?		X	
Were PCDD/PCDF ion ratios acceptable?		X	

Remark:

5. **Spikes:**	Yes	N/A	No
Was a method spike analysis performed?	X		
Were matrix spike/m.s. duplicate analyses performed?	X		
Were acceptable recoveries obtained?	X		
Was acceptable precision obtained?	X		

Remark:

6. **Blanks:**	Yes	N/A	No
Were blank analyses performed?	X		
Were any contaminants noted?			X
If yes, were blank rules applied to the data?		X	

Remark:

7. **Performance Evaluation Sample:**	Yes	N/A	No
Was a P.E. Sample analyzed with the samples?			X
If yes, were acceptable results obtained?		X	

Remark:

8. **Internal Standard / PCDD/PCDF Recovery Standards:**	Yes	N/A	No
Were peak areas acceptable?		X	

Remark:

9. **Surrogates / PCDD/PCDF Internal Standards:**	Yes	N/A	No
Were peak areas acceptable?			X

Remark: Surrogate o-terphenyl was above QC limits in sample C081705-19 (ACW-C5-MW06). The positive DRO result was "J" qualified in this sample.

10. **Compound Identification / Quantification:**	Yes	N/A	No
Were all positive results confirmed?		X	
Was supporting documentation included?	X		
Was a check of the calculations performed?	X		
If yes, were results acceptable?	X		
PCDD/PCDF ion ratios acceptable?		X	

Remark:

	Yes	N/A	No
11. Tentatively Identified Compounds?			
Were TICs requested for these analyses?		X	
If yes, were results provided?		X	

Remark:

III. Data Summary

Acceptable except as noted.

DATA QUALIFIER EXPLANATIONS

Sample	Compound(s)	Laboratory Flag	ESAT Flag	Reason
ACW-C5-MW06	FL-PRO	none	J	o-terpheyl recovery exceeded QC limits

Attachment E

RECORD TRANSFER INVENTORY FORM EPA REGION IV							
Date:							
Division:	Science and Ecosystem Support				Section:		
Branch:	Office of Quality Assurance				Unit:		
Name of Contact Person:		Sandra Sims			Phone #:	706-355 - 8772	
					VMX:		
BOX		OF		EPA Series No.	018A	Year of Records:	20??
Series Titles:	Sampling and Analytical Data Files, Superfund Site-specific						
FOR RRP USE ONLY							
Disposition Schedule #:				Date Rec'd/Entered:			
Location:				Accession #:			

DESCRIPTION OF CONTENTS

Case No.	Project No.	Lab Name	Site	Type	Note