

**Data Validation Standard Operating Procedures for  
Organic Analysis**

**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**

**REGION 4**

**SCIENCE AND ECOSYSTEM SUPPORT DIVISION  
QUALITY ASSURANCE SECTION, MTSB**

**ATHENS, GEORGIA 30605-2720**

**REVISION 3.1**

**August 2008**

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I. INTRODUCTION

The U.S. EPA Contract Laboratory Program (CLP) supports a major portion of the sample analysis needs of the Superfund Program. It is the responsibility of the Quality Assurance Section of SESD, with the support of the Environmental Services Assistance Team (ESAT) contractor, to review these data and to document their quality in a thorough consistent manner.

## II. OBJECTIVE AND SCOPE

The objective 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) SOM01.2, *Organic Analysis, Multi-Media, Multi-Concentration*, September, 2005, and revisions. This SOP follows the format and content of the *National Functional Guidelines for Organic Data Review Final, June 2007* (NFG), and revisions. Like the NFG, it provides guidance for areas of data review that require 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 contained in a separate 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.

## III. DATA PROCESSING STEPS

### 1. Summary

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 Science and Ecosystem Support Division (SESD), Athens, Georgia, and a copy, along with the electronic deliverable (EDD), are delivered to the Sample Management Office (SMO) / Data Assessment Support Services (DASS) contractor. 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 Webdat 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 re-calculated using the information submitted in the EDD. A report of this electronic 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 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 any Performance Evaluation Samples (PES) are scored and the data are appropriately qualified. In the event that 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 is prepared to complete the documentation of data quality, and the data are electronically entered into the Region IV laboratory information management system, *Element*. Review reports and project documents are maintained by the SESD Quality Assurance Section (QAS), and the data package is archived.

## 2. Data Review Documentation

### a. Computer Assisted Review

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

#### Document Contents:

1. Organic Data Review Summary Narrative - This narrative is in a letter format to summarize 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.
2. Time tracker - This document is for recording the time line and efforts at different stages of the data review process. When data entry into *Element* is required, this form must be executed and included in the data review documents. Any unusual circumstance 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.
3. PE Score (SPS-Web) - This form is generated by the SPS-Web program to report the evaluation of the results of the performance evaluation samples (PES) associated with the data package. Only the "lab" version of this form should be included.
4. The reviewed data with final qualifiers, (if any) as they appear in *Element*, are included in the data review report as a spreadsheet in Excel<sup>®</sup> format. They should also have evidence of peer review.

## IV. MULTI-MEDIA, MULTI-CONCENTRATION ANALYSES GC/MS DATA REVIEW

1. Applicability

This SOP is applicable to data collected using a gas chromatography/mass spectrometry (GC/MS) for volatile and semivolatile organic analyses, and gas chromatography-electron capture detection (GC-ECD) for pesticides/aroclor in water or soil media at low to medium concentrations. This SOP is based on the quality assurance and quality control (QA/QC) requirements specified in Exhibit D of SOW SOM01.2, and revisions, pertaining to trace and low/medium concentrations of volatile and semivolatile organic compounds, pesticides, and aroclors.

2. Holding Times / Preservation

Holding times are evaluated from the perspective of technical or actual holding times. These are determined as the age of the sample from date of sampling until preparation / extraction and analysis. The contractual holding times are determined from the Validated Time of Sample Receipt (VTSR) and are used for contract compliance and will not be the subject of this SOP.

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 US EPA guidance:

a. Pesticides and Semivolatiles:

Water (extraction)-

Day 1 thru 7	No flag
Day 8 thru 28	J all
Day 29 and greater	UR non-detect, J positive

Sediments (extraction)-

Day 1 thru 14	No flag
Day 15 thru 28	J all
Day 29 and greater	UR non-detect, J positive

Extracts (water and soil/sediments, analyses)-

Day 1 thru 40	No flag
Day 41 thru 60	J all
Day 61 and greater	UR non-detect, J positive

b. Volatiles - Water, Soil and Sediment (except Encore)

Day 1 thru 7	No flag
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Day 8 thru 14	No flag, except if pH>2, J all nonhalogenated aromatic results for water samples
Day 15 thru 28	J all
Day 29 and greater	UR non-detect, J positive

c. Volatiles - Soil analyzed using SW-846 Method 5030/5035

Encore Samples (preparation)	
Greater than 48 hours	J all
Greater than 96 hours	UR non-detect, J positive
Encore Samples (analyses from time of collection)	
Day 1 thru 14	No flag
Day 15 thru 30	J all
Day 31 and greater	UR non-detect, J positive

d. Data qualification is not automatically performed if temperature or other preservation requirements have not been 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.

3. System Performance

GC/MS instrument performance checks 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, and modern quadrupole instruments are designed to meet these criteria in full-scan mode. However, they are not applied to single ion monitoring (SIM) analyses. The instrument performance check solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are analyzed. However, in cases where a closing Continuing Calibration Verification (CCV) can be used as an opening CCV for the next 12-hour time period, then an additional tune verification is not required and the 12-hour time period begins with the injection of the CCV.

a. For VOA and TRACE 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 – Low/medium Volatiles, § 9.2). The following criteria must be met before analyses of blanks, standards and samples may proceed:

Table IV-1	
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

b. For SVOA 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). The following criteria must be met before analyses of blanks, standards and samples may proceed:

Table IV-2	
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.

c. For data obtained from the Contract Laboratory Program (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.

d. If samples are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check and are not preceded by an analysis of a closing CCV that meets the opening CCV criteria, qualify all data in those samples as unusable "R".



- e. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.
- f. 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. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).
- g. 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".
- h. 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.
- i. Any decision to use data associated with a BFB instrument performance check not meeting contract requirements should be noted, in the Data Review Narrative.
- j. 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 CLP PO 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 peak), note this for CLP PO action.

#### 4. Initial Calibration

- a. Volatiles: Initial calibration standard Relative Response Factors (RRFs) for all volatile target compounds, including 1,4-dioxane, must be greater than or equal to 0.050. 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 optional SIM technique. The reviewer should exercise

professional judgment regarding possible data qualification whenever similar ICAL problems affect DMCs.

b. Semivolatiles: Initial calibration standard RRFs for all semivolatile target compounds must be greater than or equal to 0.050. 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 optional SIM technique. The reviewer should exercise professional judgment regarding possible data qualification whenever similar ICAL problems affect DMCs.

Note: Any modified analysis accompanying a case may impact some of the preceding criteria. A copy of the flexibility clause should be present in the SDG. Refer to the Contract Laboratory Program (CLP) Web site at <http://www.epa.gov/oerrpage/superfund/programs/clp/modifiedanalyses.htm> for the specific method flexibility requirements.

c. 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.

If, for any reason, 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, the data report shall reflect the notation of the specific compound qualified as “R” with a custom qualifier explaining the unacceptable performance.

Table IV-3		
Initial Calibration		
QC Criterion	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
(GC/MS) RRF < 0.050	J	UR
(GC/MS) RRF ≥ 0.050	No qualification	
(All) %RSD > 20%	J	UJ
(All) %RSD ≤ 20%	No qualification	

## 5. Continuing Calibration

a. Volatiles: Continuing calibration standard RRFs for all volatile target compounds, including 1,4-dioxane, 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 should also be applied to Trace-VOA data, and to the optional SIM technique as well. The reviewer should exercise professional judgment regarding possible data qualification whenever similar ICAL problems affect DMCs.

b. 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 ICAL problems affect DMCs.

c. Pesticides / Aroclors: The %D of the Calibration Factors (CFs) for each of the target compounds must be less than or equal to 20.0%.

Note: Any modified analysis (MA) accompanying a case may impact some of the preceding criteria. A copy of the MA SOW should be present in the Sample Delivery Group (SDG). Refer to the Contract Laboratory Program (CLP) Web site at <http://www.epa.gov/oerrpage/superfund/programs/clp/modifiedanalyses.htm> for the specific modified analysis requirements.

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, the data report shall reflect the notation of the specific compound qualified as "R" with a custom qualifier explaining the unacceptable performance.

Table IV-4
Continuing Calibration Verification

QC Criterion	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
(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	

## 6. Blanks

The goal of the evaluation of blank results is 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 activity 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:

- a. 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.
- b. Target compounds below the 5X of the blank concentration shall be reported in samples as follows:
  - (1) If the sample result is less than the CRQL, report as non-detect at the sample CRQL: Example: blank = 12, sample = 6, CRQL = 10, report = 10U.
  - (2) If the sample result is greater than CRQL, add the U flag: Example: blank = 12, sample = 23, CRQL = 10, report = 23U
- c. 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.

d. No “B” flag is used for any organic data in Element. However, an 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 also been elevated above the CRQL.

e. Blank values are never subtracted from reportable values.

f. 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 on the basis of 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.

g. 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 are reported, as would 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.

h. Compounds formed when chlorinated water samples are extracted with methylene chloride should be carefully evaluated before they are reported. These include chlorinated cyclohexenes, cyclohexanes, and cyclohexanols. If these compounds are present in a sample, the reviewer must try to determine if chlorine was present from discussions with sampling personnel and/or other sources.

Table IV-5
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Blank Actions			
Blank Type	Blank Result	Sample Result	Action for Samples
Storage Blanks, Method Blanks, Clean-up Blanks, Instrument Blanks (Not Field QC) <sup>2</sup>	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
	Detects	$\geq$ CRQL and $< 5 \times$ blank <sup>1</sup>	Report result with a U
	Detects	$\geq$ CRQL and $> 5 \times$ blank <sup>1</sup>	No qualification

<sup>1</sup> 10x for common laboratory contaminants: (VOA) methylene chloride, acetone, 2-butanone; (SVOA) any of the six target phthalates, silicone compounds, octadecenamide, n-nitrosodiphenylamine and phthalic acid.

<sup>2</sup> If significant contamination of field, trip, and/or equipment rinsate blanks, the data user is informed via the data review narrative and by email.

## 7. Deuterated Monitoring Compounds

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

Since DMCs are associated with specific target analytes, if recovery of any one DMC 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 spiking error or final volume error (possible when all are recovered high), and whether apparent DMC 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 recovery is warranted.

In general, results are qualified if DMC recoveries are less than 10%. However, a few semivolatiles DMCs have lower recovery action limits that are less than 10%. For analytes associated with these DMCs, the qualification scheme differs. Please refer to the table below for details.

## 8. Surrogate Standards (Pesticides / Aroclors)

For the evaluation of surrogate recovery in pesticide / aroclor analyses, the factors discussed above should also be evaluated, in addition to the following: If one or both of the surrogates is 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. It must be remembered that, in the case of positive results for aroclors, the probability of positive interference for decachlorobiphenyl rises dramatically.

Table IV-6		
Deuterated Monitoring Compound / Surrogate Decision Matrix		
	Action	
	Detected Associated Compounds	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

#### 9. Matrix Spike / Matrix Spike Duplicates (MS/MSD)

As is 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 sample used as the MS/MSD are qualified as shown below using the limits given in Table 37 of the 2007 NFG.

Table IV-7
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Matrix Spike/Matrix Spike Duplicates (MS/MSD)		
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	

#### 10. Regional Quality Control / Performance Evaluation Samples

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. 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. The table below summarizes data qualification based on PES scoring results. 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. Additionally, 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 6 above. Sample TICs are not qualified based on TIC PES scoring. 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 IV-8
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	J
Action low or analyte missed	J	UR

11. Data qualification for internal standard performance is summarized below.

Table IV-9		
Internal Standard Decision Matrix for CLP GC/MS Analyses		
Criteria	Action	
	Detected Associated Compounds <sup>1</sup>	Non-detected Associated Compounds <sup>1</sup>
Area counts > 140% (for trace VOA) or > 200% (for low/med. GC/MS methods) of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	No qualification
10% ≤ Area counts < 60% (for trace VOA) or < 50% (for low/med. GC/MS methods) of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	J
Area counts < 10% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	UR
Area counts within inclusive ranges (60% - 140% for trace VOA, 50% - 200% for low/med.) of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	
RT difference > 20.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	R <sup>2</sup>	
RT difference ≤ 20.0 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, see Table 3 for Volatile and Table 2 for Semivolatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM01.1, Exhibit(s) D.
- 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.

12. Tentatively Identified Compounds (TIC)
- a. Examine the Form 1-TIC 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” 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 retained even when pesticide fraction also reported.
  - b. A formatted list of TICs reported by the laboratory should be provided by R4LIMS for each SDG. If these files are available, they are edited according to the paragraphs below. If they are not available, enter the name of each TIC, or copy from the NFG Report, into the spreadsheet template.
  - c. Eliminate any straight-chain, branched, or cyclic alkanes. Report these or whenever the laboratory reports total alkanes (any concentration) on the Form 1-TIC on one line as “Petroleum product” with no quantity, and qualify as “N,Z-01,CLP15”.
  - d. 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.
  - e. 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.
  - f. 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.
  - g. 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).
  - h. Qualify all identified TICs as “NJ,CLP15” and qualify the “Unidentified compound(s)” as “J,CLP15”.
  - i. Do not report an MRL for any identified TIC.

j. 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”.

k. 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.

### 13. Special Requirement - Pesticides/PCBs

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

a. 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 exceeds 25%, the laboratory assigns the P data qualifier flag. The reviewer should use professional judgment when evaluating pesticide analytes reported with a percent difference that exceeds 25%, and may use the table below as guidance. Multiple component analytes such as toxaphene and PCB arochlors should have no qualifiers assigned, since their qualitative identification is based on peak pattern matching. However, the reviewer should exercise professional judgment when evaluating positive hits for toxaphene and arochlors whenever large percent differences do exist. 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 IV-10			
Qualification of Pesticides/PCBs Based on % D Between Columns			
	%D > 25% ≤ 70%	%D > 70	
		< CRQL	> CRQL
Single Component	“N,CLP12”	“U” @ CRQL	“U,CLP13”
Multi-components (Toxaphene, PCBs)	No flag		

b. Each sample extract should have been diluted and re-analyzed if the initial results exceed X20 the CRQL for single component pesticides and X 16 the CRQL for multi-component toxaphene and PCBs.

c. Any analyte with a concentration > 10 ug/L for water and > 333 ug/kg for soil should be confirmed by GC/MS and flagged “C.” on the Form 1 by the laboratory, if confirmed. The reviewer should examine the procedure which was followed for at least one sample to verify that the requirements in Exhibit D-PEST, §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.

## V. DATA REVIEW PROCEDURES AND DOCUMENTATION

1. Use of Computer Aided Data Review and Evaluation (CADRE) and its successor, Electronic EXchange and Evaluation System (EXES)

As discussed in Section III, above, the results of electronic data review are utilized to assist the data review process. 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.

Each EXES NFG report is downloaded as a self expanding executable file and distributed to the data review team. The EXES NFG report is organized by SDG and includes the following elements:

Table V-1		
Elements of EXES Report		
Item	Extracted EXES Files	Contents
1	Final Flag 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).
2	Tentatively Identified Compounds (by samples and protocols)	A summary of the reported TICs for VOA and SV
3	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
4	Analytical Sequence (by protocols)	A summary of the standards and samples analyzed in an instrumental analytical sequence defined by the SOW
5	Pesticide Identification Summary for Single / Multiple Component Analytes	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
6	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.
7	Calibration Outliers (by protocols)	A summary of outliers identified in the calibrations and the list of impacted samples

Two (2) copies of CADRE/EXES reports should be printed for each SDG for the data reviewer. A copy should be included in the data validation documentation to

submit to OQADI to be maintained in the project file. The second copy should be archived with actual data package.

## 2. Data Qualifier Definitions

Region IV 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.

The following definitions 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. This qualifier set is provided here as Attachment 1 to this SOP.

### Qualifiers:

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 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.

## 3. Recording and Reporting of Data

Please refer to SOP "Data Processing and Final Production for Contract Laboratory Data in Element®.



4. Data Package Archives

The CLP data packages must be properly archived for future reference. For each data package, the form "Record Transfer Inventory" must be executed to record the proper information pertinent to the content. All the raw data, CADRE/EXES reports, and any communication records must be included. Multiple data packages from different projects could be stored in one single box if the space is available. Data packages for one Case that are stored in multiple boxes must be clearly identified on the Record Transfer Inventory forms. A proper numbering system must be maintained to have a unique number for each box for archive. A copy of the inventory form should be kept within the box and a copy to be filed in a centralized system. The data package boxes shall be maintained under the custody of SESD as described in the Data Package Audit and Data Entry/Validation SOP. For an example, see Attachment F.

VI. REFERENCES

Statement of Work (SOW) SOM01.2, Organic Analysis, Multi-Media, Multi-Concentration, September, 2005, and revisions.  
National Functional Guidelines for Organic Data Review Final, June 2007 (NFG).  
([www.epa.gov/superfund/programs/clp/analytic.htm](http://www.epa.gov/superfund/programs/clp/analytic.htm))

## Attachment 1

### Element® Qualifier Definitions as of Thursday, September 27, 2007 10:08:49AM

- 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.
- C-1** No sample container received
- C-2** Improper sample container used
- C-3** Sample container broken on receipt
- C-4** Sample container broken in the lab
- C-5** EnCore sampler received by the laboratory unlocked
- C-6** Sample aliquot taken from VOA vial with headspace (air bubble greater than 5-6 mm diameter).
- CL-1** BOD result estimated - Sample exhibited evidence of toxicity
- CL-2** DOC result higher than TOC result
- CL-3** Sample distillation not required for Ammonia
- 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
- CLP06** PE sample recovery less than control limits.
- CLP07** PE sample recovery outside warning limits.
- CLP08** PE sample recovery greater than control limits.
- 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 ICESA solution

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

**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 qualitative.

**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.

**F-1** No flash detected up to [Custom Value] °C

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

**F-3** Replicates not within method criteria

**H-1** Recommended holding time exceeded

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

**H-3** PT or QC Sample. Holding time met from beginning of prep.

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

**I-1** Ar1242 indistinguishable from 1248 - calculated as Ar1242

**I-2** Ar1248 indistinguishable from 1242 - calculated as Ar1248

**I-3** Ar1248 indistinguishable from 1254 - calculated as Ar1248

**I-4** Ar1254 indistinguishable from 1248 -calculated as Ar1254

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

**I-6** Constituents or metabolites of technical chlordane.

**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.

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

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

**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.
- NJ** Presumptive evidence that analyte is present; reported as a tentative identification with an estimated value.
- P-1** Sample improperly preserved
- P-2** Sample at improper pH
- P-3** Sample received unpreserved
- 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.
- Q-7** Results reported below routine MRL.
- QC-1** Analyte low in continuing calibration verification standard
- QC-2** Analyte 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%
- QL-5** Solid (matrix matched) LCS material
- 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%
- QM-7** The RPD and/or percent recovery for this QC spike analyte cannot be accurately calculated due to the high concentration of coeluting organic compounds in the sample matrix.
- QR-1** MRL verification recovery less than lower control limits.
- QR-2** MRL verification recovery greater than upper control limits.
- QS-1** Surrogate recovery not calculated due to sample dilution required by high analyte concentration.

**QS-2** Surrogate recovery can't be accurately calculated due to interference from coeluting organic compounds.

**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-1** The sample was filtered prior to analysis.

**SP-2** Elevated Reporting Limits due to limited sample volume.

**TC-2** Insufficient sample for TCLP extraction

**TC-3** Results represent analysis of filtrate only

**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.

**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.

**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

**XQ** Data is not being reported or may not have been fully reviewed and qualified.

**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

**Y-3** No compounds detected in the sample. Second column confirmation not required.

**Attachment 2 Data Review Summary Narrative Example**

April 10, 2008

Mr. Charlie Appleby  
Environmental Protection Agency, Region 4  
Science and Ecosystem Division  
980 College Station Road  
Athens, GA 30605-2720

**SUBJECT:**

Data Review and Validation

Project No. 08-0214

Case No. 37244

Work Order Nos.: C080808 and C081001

ESAT TDF No. 08-3938

EPA Sample Nos.: C080808-01 through 99; C081001-01 through 44

Sampling date(s): 02/11/08-02/13/08

Organic CLP Analyses:

Labname, City, State

Data for Site:

Sitename, City, State

Analyses Conducted:

Aroclors

Dear Mr. Appleby:

The ESAT Work Team reviewed data for one hundred forty-two soil samples analyzed for aroclors only, per CLP statement of work SOM01.1 in eight sample delivery groups (SDGs). The laboratory was submitted one performance evaluation sample (PES).

The samples were collected between 2/11/08 and 02/13/08, were received by the laboratory on 02/15/08, and the data package was received on 03/24/08 by the USEPA Quality Assurance Section, Region 4 SESD/MTSB. The final corrections for several sample results were received electronically on 04/08/08. These corrections were necessary because this review identified a handful of samples where the laboratory inadvertently reported nondetect results for either dilutions or reanalyses needed to quantitate aroclor 1268. The laboratory satisfied all technical and contractual analysis and extraction holding time limits except for samples C081001-13 (D4GC3), C081001-14 (D4GC4), C081001-15 (D4GC5), C081001-16 (D4GC6), C081001-17 (D4GC7), C081001-18 (D4GC8), C081001-19 (D4GC9), C081001-20 (D4GD0), C081001-21 (D4GD1), and C081001-22 (D4GD2) which were all extracted outside both technical and contractual holding times. The laboratory stated in the SDG narrative that "Due to an oversight by the analyst [the listed samples] were spiked with...EPA 8270 surrogates and not aroclor surrogates....These samples were re-extracted out of hold time...." All results for the above samples were "J" qualified.

Pertinent data quality factors are discussed below.

1. The laboratory scored within warning limits for the spiked aroclor in the soil PES.

2. Low surrogate recovery was observed for sample C080808-95 (D4GA6). All results for this sample were “J” qualified.
3. High recoveries for the surrogate DCB were observed for many samples in this case. Since this compound is a component of aroclor 1268, which was also reported for these samples, data qualification was based on the TCMX recoveries.
4. Toxaphene and/or chlordane patterns appeared to be present in a number of the samples but at levels which did not impact the aroclor identifications. Additionally, all aroclors identified by the laboratory exhibited significant weathering. Both the presence of toxaphene and the aroclor weathering would increase the quantitative uncertainties for aroclor results reported. The laboratory potentially would have reported somewhat different results if different aroclor peaks had been selected for quantitation. Data qualification was not performed on this basis.

Please refer to the attached PES scoring report, the EXES reports, and the attached marked result spreadsheets for further details. If you have any questions, please contact this office.

Very Truly Yours:

Approved:

Name  
Sr. Organic Data Reviewer  
ESAT Contract Organization

Name  
Region 4 ESAT Team Manager  
ESAT Contract Organization



**Attachment 3 Data Review Time Tracker Example**

**TIME TRACKER**

**VERSION 4.0**

CASE # :	37244	PROJECT #:	08-0214	TDF NO:	08-3938
LAB METHOD(S):	SOM01.1		LIMS METHOD CODE(S):	O101	
NUMBER OF SAMPLES:	143	VALIDATED TIME OF SAMPLE RECEIPT (VTSR):	02/15/08	DUE DATE:	04/08/08
SITE NAME:	Sitename, Cty, State			SITE ID:	A43T
Box Archival Inventory	08-55,56		PROGRAM:	SARA	
Work Order No.	C080808, C081001		TASK ORDER:	E123- 001 -42	
STAGE OR PERSON	INITIALS	DATE ACCEPTED	COMPLETION DATE	# Hours	
1. Received by EPA OQADI		03/24/08			
2. Evidentiary Audit	TM	03/12/08	03/25/08	20	
3. Data Reviewer	MEK/FRA/SS	03/24/08	04/09/08	47	
4. Spreadsheet Data Entry (Note precede qualifiers for selected SVs with a " " per marked up copy).	MEK/FRA/SS			Included as part of #3	
5. Spreadsheet Verification (return marked up copy of spreadsheet, edited hardcopy, and time tracker to reviewer)	SS	04/9/08	04/09/08	8	
6. Final Overview (memo, entry, content)	MEK/FRA	04/10/08	04/10/08	8	
7. Element Import	XXXXXXXX	XXXXXXXXXXXXXXXXXX	XXXXXXXXXXXXXXXXXX	XXXXXXXXXXXXXXXXXXXX	
8. Task Monitor (TOPO)					

**Sample and Method Information**

EPA Samples # (Separated by methods for cases with multiple lab methods applied)	V	SV	pest	pcb	PCDD/ PCDF	Metals		CN	OTHERS (specified)
						ICP/AES	ICP/MS		
C080808-01--99				X					
C0801001-01--43				X					
C0801001-44 (PES)				X					

**Notes/Comments:** Additional data (corrections) for requested samples received electronically on 04/08/08.

**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 : Labname, City, State

Site Name: Sitename, City, State

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-MW05)  
C081705-02 (ACW-C2-MW02) C081705-14 (ACW-C5-MW01) C081705-26 (ACW-C9-MW05) C081705-38 (ACW-C7-MW05)  
C081705-03 (ACW-C2-MW03) C081705-15 (ACW-C5-MW02) C081705-27 (FD-06) C081705-39 (ACW-C7-MW05)  
C081705-04 (ACW-C2-MW04) C081705-16 (ACW-C5-MW03) C081705-28 (ACW-C10-MW05) C081705-40 (ACW-C7-MW05)  
C081705-05 (ACW-C2-MW05) C081705-17 (ACW-C5-MW04) C081705-29 (ACW-C10-MW05) C081705-41 (ACW-C7-MW05)  
C081705-06 (ACW-C2-MW06) C081705-18 (ACW-C5-MW05) C081705-30 (ACW-C10-MW05) C081705-42 (FD-07)  
C081705-07 (ACW-C3-MW01) C081705-19 (ACW-C5-MW06) C081705-31 (ACW-C10-MW05) C081705-43 (FD-08)  
C081705-08 (ACW-C3-MW02) C081705-20 (ACW-C6-MW04) C081705-32 (ACW-C10-MW05) 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?	<u>  X  </u>	<u>      </u>	<u>      </u>
Were all required OC checks performed?	<u>  X  </u>	<u>      </u>	<u>      </u>
Were all required documents present?	<u>  X  </u>	<u>      </u>	<u>      </u>
Were requested detection limits met?	<u>  X  </u>	<u>      </u>	<u>      </u>

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)?	<u>  X  </u>	<u>      </u>	<u>      </u>
PCDD/PCDF extracted within 30 days of sampling?	<u>      </u>	<u>  X  </u>	<u>      </u>
Extracts analyzed within 40 days of extraction?	<u>  X  </u>	<u>      </u>	<u>      </u>
Were all samples/extracts properly preserved?	<u>  X  </u>	<u>      </u>	<u>      </u>
<b>For TCLP:</b> Were RCRA TCLP holding times met?	<u>      </u>	<u>  X  </u>	<u>      </u>

Remark:

### 3. GC/MS Tuning:

	Yes	N/A	No
Were PFK/DFTPP/BFB criteria met?	<u>      </u>	<u>  X  </u>	<u>      </u>
<b>Pesticides:</b> Were standards run in proper sequence?	<u>      </u>	<u>  X  </u>	<u>      </u>
Combined DDT/Endrin Breakdown acceptable?	<u>      </u>	<u>  X  </u>	<u>      </u>
Retention time windows defined?	<u>      </u>	<u>  X  </u>	<u>      </u>

**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:

Organic Data Review SOP  
Revision 3.1  
June 2008

<b>6. Blanks:</b>	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:

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

Remark:

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

Remark:

<b>9. Surrogates / PCDD/PCDF Internal Standards:</b>	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.

<b>10. Compound Identification / Quantification:</b>	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**

<b>Sample</b>	<b>Compound(s)</b>	<b>Laboratory Flag</b>	<b>ESAT Flag</b>	<b>Reason</b>
ACW-C5-MW06	FL-PRO	none	J	o-terpheyyl recovery exceeded QC limits

