



U.S. Environmental Protection Agency, Region 2 Field Operations Quality Procedures

ADMINISTRATIVE STANDARD OPERATING PROCEDURE

Standard Operating Procedure for Validation of Pesticide Data

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4/1/2022	QA-HWSS-A-007
Author	
Name: Russell Arnone	
Title: Chemist	
Division/Branch/Section: LSASD/HWSB/HWSS	
Signature:	Date:
Review & Approvals	
Name: Narendra Kumar	
Title: Chemist, HWSS	
Signature:	Date:
Name: Donna Ringel	
Title: Chief, HWSS	
Signature:	Date:
Name: Jon Gabry	
Title: Chief, HWSB	
Signature:	Date:
Name:	
Title:	
Signature:	Date:

The table below identifies information about the reviews conducted of this Standard Operating Procedure (SOP).

REVIEW HISTORY		
Date	Reviewer Name	Changes Required (Y/N)

The table below identifies changes to this controlled document and the respective effective date(s) over time.

REVISION HISTORY		
Revision Number	Revision Description	Effective Date
0	Original Issue Note: Replaces SOP HW-36a, Rev.1 SOM02.2 (Pesticide Data Validation), October 2016	

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (USEPA) and other governmental employees. They do not constitute rule-making by the USEPA and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at a variance with the policies and procedures in this Standard Operating Procedure (SOP).

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1.0 PURPOSE AND APPLICABILITY

This document is designed to promote uniformity of data review of analytical data generated through the US EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Superfund Analytical Methods SFAM01.1 and any future editorial revisions of SFAM01.1. It is applicable to the review of Contract Laboratory Program (CLP) water, soil, sediment, waste, TCLP, SPLP and closely related matrices using Gas Chromatography/Electron Capture Detection (ECD) for Pesticide analyses.

The guidelines presented in this document will aid in establishing (a) if data meets the specific technical and quality control (QC) criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the user that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, the need to facilitate the progress of specific projects, and the availability for re-sampling. The user should note that while this document is to be used as an aid in the formal data review process, the site-specific quality assurance project plan, as well as professional judgement, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria. Professional judgment when used to qualify data including rejection of any data should be explained.

2.0 SUMMARY OF PROCESS OR METHODOLOGY

This document provides the criteria for performing technical quality assurance reviews of pesticide analytical data generated through the CLP program. Criteria are based on the quality assurance/quality control and technical requirements specified in Exhibit D of SOW SFAM01.1. This SOP incorporates much of the content of the National Functional Guidelines (NFG) and provides additional guidance specific to EPA Region 2.

Upon receipt by EPA Region 2, CLP data in the Sample Delivery Group (SDG) undergoes a technical quality assurance review based upon the criteria in this document. A report of this review is prepared by the data validator, reviewed by the EPA Task Order Contracting Officer Representative (TOCOR), and provided to the data user.

3.0 DEFINITIONS

3.1. See Appendix C – Definitions/Glossary of Terms

3.2. Acronyms and Abbreviations

The following acronyms and abbreviations may be found throughout this document.

%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%Resolution	Percent Resolution
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids, (also %S)
ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
\overline{CF}	Mean Calibration Factor
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
DAR	Data Assessment Report
DF	Dilution Factor
DL	Detection Limit
DQO	Data Quality Objectives
DV	Data Validation
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
EDS	Environmental Data Services
EICC	Electronic Internal Chain of Custody
EPA	Environmental Protection Agency (see also USEPA)
ESAT	Environmental Services Assistance Team
EXES	Electronic Data Exchange and Evaluation System
GC/ECD	Gas Chromatograph/Electron Capture Detector
HWSS	Hazardous Waste Support Section
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IUPAC	International Union of Pure and Applied Chemistry
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NFG	National Functional Guidelines
OSRTI	Office of Superfund Remediation and Technology Innovation
PDF	Portable Document Format

QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SP	SharePoint
SPLP	Synthetic Precipitation Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TSS	Total Suspended Solids
TOCOR	Task Order Contracting Officer Representative
TR/COC	Trip Report/Chain of Custody
TSS	Total Suspended Solids
USEPA	United States Environmental Protection Agency

* The above list may contain abbreviations not used in Pesticide analysis.

3.3. Data Qualifier Definitions

Data qualifier definitions are provided in the beginning of Appendix A.

4.0 RESPONSIBILITIES/QUALIFICATIONS

4.1. Qualifications

Data Validator must be familiar with the current CLP SOW and the documents referenced in Section 5.0 below.

4.2. Responsibilities

- 4.2.1. EPA TOCOR (when applicable) – will review data assessments reports and other deliverables prepared by contract data validators. They will update the MS Planner DV Flowboard indicating the progress of SDGs, post final deliverables to the EDS SharePoint site and send notification to clients via the established workflow.
- 4.2.2. Data Validator – will follow the criteria and actions provided in this document and prepare Data Assessment Reports (DAR) and Summary Reports, as necessary. If the

validator is an ESAT contractor employee, they will consult the EPA TOCOR when questions arise. They will update the DV Flowboard indicating progress of SDGs.

5.0 REFERENCES

National Functional Guidelines for Organic Superfund Methods Data Review, EPA 540-R-20-005, November 2020.

Contract Laboratory Program (CLP) Statement of Work (SOW) Superfund Analytical Method SFAM01.1.

FA-0010.1, Standard Operating Procedure for Development and Use of Field SOPs, December 2015.

U.S. EPA, 2007. Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents. EPA QA/G-6, EPA/600/B-07/001. April 2007.

QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations. Revision 0, January 2021.

6.0 PROCEDURAL STEPS

6.1. EXES Processing

At the Sample Management Office (SMO) the data package and electronic data deliverables (EDD) are checked for compliance with the contract. A Contract Compliance Screening Report (CCS) is issued and posted on the SMO portal website. The EDD is processed electronically to evaluate QC performance against the NFG and Region 2 criteria by EXES. An electronic report of the EXES review is also posted on the SMO portal website.

6.2. Initial Notification

The EICC SharePoint web application is setup to send an e-mail alert notification to EPA and ESAT data validators when a new data package is received and available for review and validation. Entry of data into the EICC SharePoint site will automatically trigger an e-proxy card to populate on the DV Flowboard in MS Planner.

Alternate electronic systems may be applied in the future.

6.3. DV Flowboard Updates

Update to DV Flowboard will be performed as per SOP QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations (or most current version).

6.4. Data Package Inspection

The EXES Data Manager (EDM) is a useful tool in the data review process. EDM will identify any missing and/or incorrect information in the data package. When available, the EDM should be reviewed as part of the initial data package inspection. The CLP laboratory may submit a reconciliation package for any missing items or to correct the data. If there are any concerns regarding the data package, contact the TOCOR.

An initial review of the data package is to be performed, taking into consideration all information specific to the sample data package, (e.g., modified analysis requests, trip report/chain-of-custody documentation, SDG narratives, etc.). The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP will supersede that in this SOP. The reviewer should access the HWSS SP Documents Dashboard to obtain a copy of the relevant documents.

The SDGs or cases routinely have unique samples that require special attention from the reviewer. These include field blanks, equipment blanks, trip blanks, and field duplicates which must be identified in the sample records. The sampling records (i.e., trip reports or COC records) should identify:

- 1) The Region where the samples were taken,
- 2) The case number,
- 3) The complete list of samples with the following information as applicable:
 - a. Sample matrix,
 - b. Field blanks (i.e., equipment, rinsate and trip),
 - c. Field duplicates,
 - d. Field spikes,
 - e. Shipping dates,
 - f. Preservatives, and
 - g. Laboratories involved

6.5. Data Review/Validation

The EXES electronic validation will apply most of the criteria and actions provided in Appendix A. The data validator will examine the EXES report to identify any issues that warrant further investigation. All EXES rejected data will be manually evaluated. The data validator will use the criteria and actions in Appendix A, as well as their own professional judgement to manually assess these data.

To use this SOP effectively, the reviewer should understand the analytical method. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information for the validator.

The Trip Report/Chain of Custody (TR/COC) documentation includes samples descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation and unusual events should be documented in the SDG narrative. The reviewer should also inspect any email, telephone or any communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office and USEPA Region 2.

All data are initially marked as "Reportable" (YES) in EDM before validation is begun. Sometimes, due to dilutions and/or re-analyses being performed, there will be multiple results for a single analyte from a sample. The following criteria and professional judgement are used to determine which result should be reported:

- 1) the analysis with the lower QL,
- 2) the analysis with the better QC results, and/or
- 3) the analysis with the higher result

Data validator will reconcile results from the multiple runs to provide results in one run and report. The analyte values and their respective QLs are then transferred into a single sample run. The runs and results that are not to be used are marked "not reportable" or entered "NO" in the "Reportable" fields of the EDM.

6.6. Data Assessment Report

The data validator will prepare a Data Assessment Report (DAR) documenting the results of their data review. This report will be formatted in accordance with the template provided in Appendix B. Modifications to the template are allowed at the discretion of the user.

6.7. Summary Report

If requested by the client on the Analytical Request Form (ARF), the data validator will prepare a Summary Report using the HWSS Summary Report application.

7.0 DATA AND RECORDS MANAGEMENT

7.1. DATA MANAGEMENT

Posting data to the SP EDS site is done in accordance with QA-HWSS-A-001, "Document Control Room, Data Dissemination and Archive Operations".

7.2. RECORDS MANAGEMENT

The data files uploaded to the EDS SharePoint site include:

- 1) Data Assessment Report (Adobe PDF),
- 2) Edited/Validated Sample Summary Report from SMO portal (Adobe PDF),
- 3) Edited/Validated EQUIS EDD report from SMO portal (MS Excel),
- 4) Generated Summary Report (MS Excel), if applicable, and
- 5) Generated Summary Report with Hits Only (MS Excel), if applicable.

In addition to the above stated documents, data validator also forwards the following files, which are not uploaded to EDS SharePoint:

- 6) The CCS Report from the SMO Portal (Adobe PDF),
- 7) Edit History Report from the SMO Portal (Adobe PDF)

All files stated above are saved to the Local Area Network (LAN) G: drive at DESADIV/HWSS/DATA VALIDATION/Site Name/Case #/SDG#. Files are renamed using the following naming convention, Case#_SDG#_Filetype.*, e.g., 12345_BAB12_S3VEM.xlsx.

Note: "M" in the file type signifies that the data has been manually validated by ESAT and/or EPA Staff.

Additional records management procedures are discussed in QA-HWSS-A-001, "Document Control Room, Data Dissemination and Archive Operations".

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

- 8.1. This SOP will be reviewed annually. Reviews will be documented on the Review History Table on page 2 of the SOP. The SOP shall be updated every 5 years, or more frequently, when necessary, due to significant changes.
- 8.2. The "Request for SOP Change Form", Appendix D is used to document changes and is appended to the final SOP until such time as the changes are incorporated into the body of the text of the SOP.

9.0 APPENDICES

Appendix A - Data Validation Criteria and Actions

Appendix B - Data Assessment Report Template

Appendix C - Definitions/Glossary of Terms

Appendix D - SOP Change Request Form (CRF)

Appendix A

Data Validation Criteria and Actions

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I. Data Validation Qualifier

The following are the definitions for the qualifiers assigned to results in the data review process. The reviewer should use these qualifiers as applicable.

Pesticide Table 1. Data Validation Qualifier Definitions

Data Qualifier	Definition
U	The analyte was analyzed for but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
UJ	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
C	The target analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS).
X	The target analyte identification was not confirmed when GC/MS analysis was performed.

NOTES:

1. Comments for sample results with data qualifiers other than “U” or no qualification based on professional judgement must be included in the DAR.
2. With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an “R” qualifier rather than a “UJ”.
3. Although a “J+” or a “J-” may appear as less ambiguous than a “J”, the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.
4. Criteria, evaluation, quantitation limits (QLs), calculations, acceptable ranges and related parameters and definitions are detailed in the applicable Statement of Work (SOW) and/or

National Functional Guidelines (NFG) documents referenced above and should be used as necessary for data validation. Such criteria when available in the project specific quality assurance plan (QAPP) document supersede SOW and/or NFG criteria. Such occurrences should be discussed with TOCORs.

II. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, analysis logs, raw data, and the data package narrative checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Action

Refer to Pesticide Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate (if requested) and field blanks or as specified in the project- specific data validation Standard Operation Procedures (SOPs).

1. If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.
2. If a discrepancy is noted between the sample analysis date on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Pesticide Table 2. Preservation and Holding Time Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/Non-aqueous	Samples received at temperature > 6°C	Outside maximum allowed temperature	J	UJ
Aqueous/Non-aqueous	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	No qualification	No qualification

	Not cooled at temperature $\leq 6^{\circ}\text{C}$	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	J	UJ
Aqueous/Non-aqueous	Cooled/Not cooled at temperature $\leq 6^{\circ}\text{C}$	TCLP/SPLP leachate samples prepared outside the 14-day technical holding time	J	R
Aqueous	Cooled at temperature $\leq 6^{\circ}\text{C}$	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	UJ*
		Samples and TCLP/SPLP leachates extracted within or outside the 7-day and analyzed outside the 40-day technical holding time	J	UJ*
Aqueous	Not cooled at temperature $\leq 6^{\circ}\text{C}$	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	J	UJ
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	UJ*

Aqueous	Not cooled at temperature $\leq 6^{\circ}\text{C}$	Samples and TCLP/SPLP leachates extracted within or outside the 7-day and analyzed outside the 40-day technical holding time	J	UJ*
Non-aqueous	Cooled at temperature $\leq 6^{\circ}\text{C}$	Samples extracted within the 14-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J-	UJ*
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J-	UJ*
Non-aqueous	Not cooled at temperature $\leq 6^{\circ}\text{C}$	Samples extracted within the 14-day and analyzed within the 40-day technical holding time	J	UJ
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J	UJ*
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J	UJ*

* If there is gross exceedance and considering all other QC factors, use professional judgment to qualify non-detects as unusable (R). If exceedance is minor, qualify non-detects as estimated (UJ).

III. Gas Chromatograph/Electron Capture Detector Instrument Performance Check

A. Review Items

Laboratory instrument performance check reports (if available), chromatograms, and data system printouts in the data package.

B. Objective

The objective of performing Gas Chromatograph/Electron Capture Detector (GC/ECD) instrument performance checks is to ensure adequate resolution and instrument sensitivity.

C. Action

Refer to Pesticide Table 3 for the evaluation criteria and corresponding actions for detected and non-detected analyte results. Apply the actions to the samples and blanks associated to the deficient RESC, ICAL PEM and/or mid-point ICAL standards in the same analytical sequence. Apply the actions to the samples and blanks associated to the deficient CCV PEM and mid-point pesticide calibration standards.

Pesticide Table 3. GC/ECD Instrument Performance Check Actions

Criteria		Action	
		Detects	Non-detects
RESC not performed at specified frequency and sequence		Use professional judgment	Use professional judgment
RESC % Resolution < 60% (INDA/INDB)	RESC %Resolution < 80.0% (INDC, primary column) %Resolution < 50.0% (INDC, secondary column)	NJ	R
PEM not performed at the specified frequency and sequence		R	R
PEM %Resolution < 90%		NJ	R
PEM: 4,4'-DDT %Breakdown > 20% and 4,4'-DDT is detected		J for 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE	No qualification
PEM: 4,4'-DDT %Breakdown > 20% and 4,4'-DDT is not detected		NJ for 4,4'-DDD and 4,4'-DDE	R for 4,4'-DDT

PEM: Endrin %Breakdown > 20% and Endrin is detected		J for Endrin, Endrin aldehyde, and Endrin ketone	No qualification
PEM: Endrin %Breakdown > 20% and Endrin is not detected		NJ for Endrin aldehyde and Endrin ketone	R for Endrin
PEM: Combined %Breakdown > 30%		Apply qualifiers as described above considering degree of individual breakdown	Apply qualifiers as described above considering degree of individual breakdown
Midpoint ICAL standard not performed at specified frequency		R	R
% Resolution < 90% (Midpoint INDA/INDB)	%Resolution < 80.0% (Midpoint INDC, primary column) %Resolution < 50.0% (Midpoint INDC, secondary column)	NJ	R

IV. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), initial calibration standard quantitation reports and chromatograms in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Action

Refer to Pesticide Table 4 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated to a deficient ICAL. Apply the actions to the samples and blanks in the same analytical sequence as the deficient ICAL.

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. If a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).
2. If the ICAL is not performed at the specified concentrations, qualify detects as estimated (J) and non-detects as estimated (UJ). This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of RT windows, CFs, mean CFs, or %RSDs, perform a more comprehensive recalculation. If the chromatogram display criteria are not met, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel to arrange for a revised report.
4. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated (J). No qualification for non-detects.
5. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when %RSD criteria are not met. The following guidelines are recommended:
 - a. If the %RSD criteria of any target analytes are not met, and the %RSD criteria are still not satisfied after eliminating either the high- or the low-point of the ICAL:
 - i. Qualify detects in the associated samples as estimated (J).
 - ii. Use professional judgment to qualify non-detects in the associated samples as estimated (UJ).
 - b. If the high-point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
 - i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
 - ii. Non-detects in the associated samples should not be qualified.
 - c. If the low-point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
 - i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
 - ii. For non-detects in the associated samples, use the lowest point of the linear portion of the ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).
6. Qualification of the target analyte data is not necessary based on the surrogate %RSD data alone. Use professional judgment to evaluate the surrogate %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Pesticide Table 4. Initial Calibration Actions

Criteria	Action	
	Detects	Non-detects
Initial Calibration not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specified concentrations	J	UJ
RT windows incorrect or Chromatogram criteria not met	J	R
%RSD for target analyte outside specified acceptance limits*	J	No qualification
%RSD for target analyte within specified acceptance limits*	No qualification	No qualification

- * %RSD ≤ 20.0% for single component target analytes except alpha-BHC and delta-BHC.
- % RSD ≤ 25.0% for alpha-BHC and delta-BHC.
- % RSD ≤ 30.0% for Toxaphene peaks.
- % RSD ≤ 20.0% for surrogates (TCX and DCB).

V. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Action

Refer to Pesticide Table 5 for the evaluation criteria and corresponding actions for detected and non- detected analyte results in samples associated with a deficient CCV. Apply the actions to the samples and blanks in the same analytical sequence as the deficient CCV PEMs or CCVs.

1. If the CCV PEM or CCV standard is not performed at the specified frequency and sequence, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified, if holding times have not expired and there is extract remaining. If a reanalysis cannot be performed, carefully evaluate all other available

information, including the quality of analyte peak shapes and RT match of surrogates on both columns, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results and qualification of all quantitative results as estimated (J). Otherwise, qualify all detects and non-detects as unusable (R).

2. If the CCV PEM is not performed at the specified concentration, qualify detects as estimated (J) and non-detects as estimated (UJ).
3. If the CCV standard is not performed at the specified concentration, qualify detects as estimated (J) and non-detects as estimated (UJ). Special consideration should be given to sample results at the opposite extreme of the calibration range if CCV concentration is not at the mid-point calibration range. Evaluate the ICAL performance in the concentration range of the detected analyte results.
4. If the RT of any target analyte in the CCV PEM and CCV standard is outside the RT window, carefully evaluate the associated sample results. All samples injected after the last in-control standard are potentially affected.
 - a. For detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analytes of interest. If the peaks are close to the expected RT window of the Pesticide of interest, it may require additional effort to determine if sample peaks represent the target analytes of interest. For example, the data reviewer may examine the presence of three or more standards containing the target analytes of interest that were analyzed within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT windows can be re-evaluated using the mean RTs of the standards.

If the peaks in the affected sample fall within the revised windows, qualify detects as estimated (J).
 - b. For non-detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analytes of interest.
 - i. If no peaks used for Pesticide analyte identification are present, non-detects should not be qualified.
 - ii. If any peaks present are close to the expected RT window of the analytes of interest, use professional judgement to qualify the non-detects as estimated (UJ).
5. If errors are detected in the calculations of either the %D or %Breakdown in the CCV PEM, %D in any CCV standard or %D for any Toxaphene peak in the applicable CCV standard, perform a more comprehensive recalculation.
6. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of either a PEM or a CCV standard as closing CCV exceeds 14 hours, carefully

evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).

7. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last sample or blank in the same analytical sequence exceeds 12 hours, carefully evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).
8. Qualification of the target analyte data is not necessary based on the surrogate %D in CCV PEM and CCV standards alone. Use professional judgment to evaluate the surrogate %D data in conjunction with the surrogate recoveries to determine the need for data qualification.
9. If more than 72 hours have elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene calibration verification standard, qualify detects and non-detects as unusable (R).

Pesticide Table 5. CCV Actions

Criteria	Action	
	Detects	Non-detects
CCV PEM or not performed at specified frequency and sequence	J	UJ
CCV PEM not performed at specified concentrations	J	UJ
CCV standard not performed at specified concentrations	J	UJ
RT outside specified RT window	J	UJ
CCV PEM %D for target analyte outside specified limit*	J	UJ
CCV standard %D for target analyte outside specified limit*	J	UJ
RT, CCV PEM %D, CCV %D, and time elapsed within specified limits	No qualification	No qualification
Time elapsed between opening CCV instrument blank and closing CCV exceeds 14 hours	J	R
Time elapsed between opening CCV instrument blank and last sample, or blank exceeds 12 hours	J	R

Time elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene calibration verification standard exceeds 72 hours	R	R
---	---	---

- * %RSD ≤ 25.0% for single component target analytes and surrogates (TCX and DCB).
- %RSD ≤ 25.0% for Toxaphene peaks.

VI. Blanks

A. Review Items

Laboratory Results Reports, chromatograms, and quantitation reports in the data package and sampling trip reports.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Action

Refer to Pesticide Table 6 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient blanks. Apply the actions to all samples associated with the method blank by the same preparation batch; all samples associated with the TCLP/SPLP LEB by the same leachate extraction batch; all samples associated with the ICAL instrument blank in the analytical sequence; all samples associated with the opening or closing CCV instrument blank in the same analytical sequence; and all samples associated with the sulfur blank by the same cleanup batch.

1. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Verify that data qualification decisions based on field quality control (QC) are supported by the project QAPP or the project-specific Standard Operating Procedures (SOPs). At a minimum, contamination found in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank that has the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.
2. For any method blank reported with results that are < QLs, no qualification is required for sample results that are ≥ QLs.
3. For any method blank reported with results ≥ QLs, report sample results that are ≥ QLs but < Blank Results at sample results and qualify as non-detect (U). No qualification is required for sample results that are ≥ QLs and ≥ Blank Results.

4. For TCLP/SPLP LEBs, sulfur cleanup blanks, instrument blanks, and field blanks (including equipment and rinse blanks), sample result qualifications listed in Pesticide Table 5 should apply if supported by the QAPP.
5. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified, or in the case of field QC, should at least be documented in the Data Review Narrative. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample.
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
7. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Pesticide Table 6. Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
	Not analyzed at the specified frequency	Non-detect	No qualification
		Detect	J
Method, TCLP/SPLP LEB, Sulfur cleanup, Instrument, Field (including Equipment and Rinse) Blank	Detect	Non-detect	No qualification
	Detect < QL	MDL ≤ Detect < QL	Report at QL and qualify U
		≥ QL	No qualification
	≥ QL	Detect < QL	Report at QL and qualify U
		≥ QL but < Blank Result	Report at sample result and qualify U
		≥ QL and ≥ Blank Result	No qualification
	Gross contamination	Detect	Report at sample result and qualify R

VII. Surrogate

A. Review Items

Laboratory surrogate reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate surrogate percent recovery (%R) to ensure that the analytical method is efficient

C. Action

Refer to Pesticide Table 7 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results associated with the deficient surrogates in samples. Qualification of the target analyte data is not necessary based on the surrogate %RSD data alone. Use professional judgment to evaluate the surrogate %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. If a reanalysis cannot be performed, qualify the data as unusable (R).
2. If any surrogate %R in a blank is outside the limits specified in the QAPP or in the SOW, special consideration should be taken to determine the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process.
3. If one or more samples in the same extraction batch have surrogate %R within the acceptance limits, use professional judgment to determine if the blank problem is an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for project management personnel action

Pesticide Table 7. Surrogate Actions

Criteria	Action*	
	Detects	Non-detects
Surrogate not present or not at specified concentration	J	UJ
RT out of specified RT window	Use professional judgment**	Use professional judgment**
RT within specified RT window	No qualification	No qualification
%R < 10% (sample dilution not a factor)	J-	R
%R < 10% (sample dilution is a factor)	No qualification	No qualification
10% < %R < 30%	J-	UJ
%R within specified Acceptance Limits (30% - 150%)	No qualification	No qualification
%R > 150%	J+	No qualification

* Diluted samples with dilution less than or equal to (\leq) 5 should be qualified for surrogates recovery outside criteria. Diluted samples with dilution factor greater than ($>$) 5, no qualification is applied.

** Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

VIII. Matrix Spike / Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, quantitation reports and chromatograms in the data package.

B. Objective

The objective of Matrix Spike (MS)/Matrix Spike Duplicate (MSD) analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Action

Refer to Pesticide Table 8 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the original samples associated with deficient MS/MSDs.

Apply the actions to the same analytes in the parent samples used for the MS/MSD analyses or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticide Table 8. MS/MSD Actions

Criteria	Action	
	Detects	Non-detects
MS/MSD not analyzed at specified frequency	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment*	Use professional judgment*
%R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (20%)	J	R
Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit	J	UJ
%R or RPD within specified Acceptance Limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

* Notify CLP PO if a **field blank** was used for the MS/MSD.

IX. Laboratory Control Standard

A. Review Items

Laboratory Results Reports, chromatograms, and data system printouts in the data package.

B. Objective

The objective is to evaluate accuracy of the analytical method and laboratory performance.

C. Action

Refer to Pesticide Table 9 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with the deficient LCSs. Apply the actions to all associated samples prepared together (in the same preparation batch) or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticide Table 9. LCS Actions

Criteria	Action	
	Detects	Non-detects
LCS not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
LCS %R limits not specified	Use professional judgment	Use professional judgment
%R < specified Lower Acceptance Limit	J-	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

X. Florisil Cartridge Performance Check

A. Review Items

Laboratory Results Reports, chromatograms, and data system printouts in the data package.

B. Objective

The objective is to evaluate the performance of the Florisil cartridge used for the Florisil cleanup procedure on sample extracts.

C. Action

Refer to Pesticide Table 10 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient Florisil Cartridge Performance Checks. Apply the actions to all associated samples, blanks and LCSs that have undergone Florisil cleanup (in the same cleanup batch) in the analytical sequence or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticide Table 10. Florisil Cartridge Performance Check Actions

Criteria	Action	
	Detects	Non-detects
Florisil Cartridge Performance Check not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit for target analytes (10%)	J	R
Expanded Lower Acceptance Limit for target analytes (10%) ≤ %R < Lower Acceptance Limit for target analytes (80%)	J	UJ
Lower Acceptance Limit for target analytes (80%) ≤ %R ≤ Upper Acceptance Limit for target analytes (120%)	No qualification	No qualification
%R > Upper Acceptance Limit for target analytes (120%)	J	No qualification
%R ≥ 5% (2,4,5-trichlorophenol)	Use professional judgment	Use professional judgment

XI. Gel Permeation Chromatography Performance Check

A. Review Items

Laboratory Gel Permeation Chromatography (GPC) calibration verification reports (if available), two ultraviolet (UV) traces, GPC cleanup blank quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate GPC cleanup efficiency.

C. Action

Refer to Pesticide Table 11 for the evaluation criteria and the corresponding actions for detected and non-detected analyte results in the samples associated with a deficient GPC Performance Checks. Apply the actions to all associated samples, blanks and LCSs that have undergone GPC cleanup (in the same cleanup batch) in the analytical sequence or as specified in the project-specific Standard Operating Procedures (SOPs).

1. If GPC calibration frequency, UV traces, and GPC blank criteria are not met, examine the raw data for the presence of high molecular weight contaminants, examine subsequent

sample data for unusual peaks, and use professional judgment to qualify the data. If the samples have been analyzed under unacceptable GPC criteria, notify the designated project management personnel.

If the RT shift of bis(2-ethylhexyl) phthalate and perylene is greater than (>) 5%, the GPC unit may be in an unstable temperature environment and subject to erratic performance. The expected result may be an unknown bias in the data. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified.

2. If GPC calibration verification is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects.
3. If errors are detected in the calculations of the %R in the GPC calibration verification, perform a more comprehensive recalculation.

Pesticide Table 11. Gel Permeation Chromatography Performance Check Actions

Criteria	Action	
	Detects	Non-detects
GPC calibration not analyzed at specified frequency or concentration	J	UJ
Analyte resolution in the most recent UV traces and/or RT shift that does not meet specified criteria	Use professional judgment	Use professional judgment
GPC blank not analyzed at the specified frequency and sequence	Use professional judgment	Use professional judgment
Analyte result in GPC blank \geq QL	Use professional judgment	Use professional judgment
GPC calibration verification not analyzed at specified frequency	J	UJ
%R < Expanded Lower Acceptance Limit for target analytes (10%)	J	R
Expanded Lower Acceptance Limit for target analytes (10%) \leq %R < Lower Acceptance Limit for target analytes (80%)	J	UJ
Lower Acceptance Limit for target analytes (80%) \leq %R \leq Upper Acceptance Limit for target analytes (120%)	No qualification	No qualification
%R > Upper Acceptance Limit for target analytes (120%)	J	No qualification

XII. Target Analyte Identification

A. Review Items

Laboratory Results Reports, quantitation reports, mass spectra, and chromatograms in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatography/Electron Capture Detector (GC/ECD) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Action

Refer to Pesticide Table 12 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the applicable samples, blanks and LCSs in the data package or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticide Table 12. Target Analyte Identification Actions

Criteria	Action	
	Detects	Non-detects
Detected target analyte RT outside specified RT window (false positive)	Report at QL and qualify U	Not applicable
Detected target analyte peak exhibits an interference with the potential detection of another target peak (false positive)	R	Not applicable
Reported non-detect target analyte RT within specified RT windows on both GC columns (false negative)	Use professional judgment to report results	Not applicable
Toxaphene peak RT windows overlap with single component target analytes or chromatographic interferences exist	Use professional judgment	Use professional judgment
Toxaphene peaks exhibit a marginal pattern-matching quality	Use professional judgment or Report results and qualify NJ	No qualification

Evident chromatographic interference or co-elution for the detected target analyte	Use professional judgment to Report results at lower value and qualify NJ or Report at QL and qualify U	Not applicable
%D for any target analyte 0% - 25%	No qualification	Not applicable
%D for any target analyte 26% - 200%	J	Not applicable
%D for any target analyte > 200% (Interference detected)	NJ	Not applicable
%D for any target analyte > 200% (Interference not detected*)	NJ	Not applicable

* Visual examination of the chromatograms should be performed to check for interference and compliance with SOW Technical Criteria for Identification. Note the finding in the report.

XIII. Gas Chromatograph/Mass Spectrometer Confirmation

A. Review Items

Laboratory Results Reports, sample preparation sheets, data package narrative, quantitation reports, and chromatograms in the data package.

B. Objective

The objective is to ensure the accuracy of the positive identification of a target analyte.

C. Action

Refer to Pesticide Table 13 below for the evaluation criteria and corresponding actions for detected analyte results in the samples.

Pesticide Table 13. GC/MS Confirmation Actions

Criteria	Action
	Detects
Analyte confirmed by GC/MS	C
Analyte indicated but not confirmed by GC/MS	X

XIV. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation sheets, data package narrative, quantitation reports, and chromatograms in the data package.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Action

Refer to Pesticide Table 14 below for the evaluation criteria and corresponding actions for the percent solids (% Solids) of the samples.

If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

Pesticide Table 14. Target Analyte Quantitation - Percent Solids of Sediment Actions

Criteria	Action	
	Detects	Non-detects
% Solids < 10.0%	J	R
10.0% ≤ % Solids < 30.0%	J	UJ
% Solids ≥ 30.0%	No qualification	No qualification

XV. Field Duplicates

A. Review Items

Review Chain of Custody and Trip Report (COC/TR) to identify which samples within the data package are field duplicates.

B. Objective

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision.

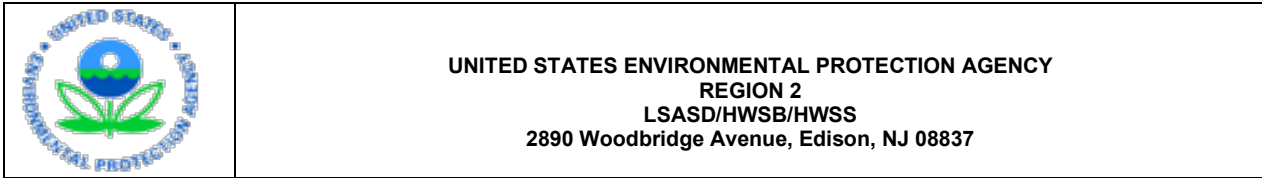
C. Action

In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

1. Identify which samples within the data package are field duplicates.
2. Estimate the relative percent difference (RPD) between the values for each compound.
3. If large RPDs (> 50%) is observed, confirm identification of samples, and note difference in the executive summary.

Appendix B

Data Assessment Report Template



EXECUTIVE NARRATIVE

Case No.: **SDG No.:**
Site: **Laboratory:**
Number of Samples: **Sampling dates:**
Analysis: **Validation SOP:**

QAPP:
Contractor:
Reference: DCN Number

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Major Findings:

Minor Findings:

COMMENTS:

Reviewer Name(s):

Approver's Signature:

Name:

Date:

Affiliation: USEPA/R2/LSASD/HWSB/HWSS

Appendix C

Definitions/Glossary of Terms

DEFINITIONS* & GLOSSARY OF TERMS

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.

Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICs); Laboratory Control Samples (LCSs); and Preparation Blanks.

Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.

Calibration Curve -- A plot of instrument response versus concentration of standards.

Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.

Holding Time -- The maximum amount of time samples may be held before they are processed.

- a. **Contractual** -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

- b. **Technical** -- The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.

Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).

Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be $3.14s$ above the blank, where "s" is the standard deviation of the 7 replicates.

Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Office of Solid Waste and Emergency Response (OSWER) -- The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
- b. Each 7-calendar day period (3-calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- c. Scheduled at the same level of deliverable.
- d. In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

** The above list is all inclusive and may contain terms not applicable to Pesticide Analysis.*

Appendix D

SOP Change Request Form (CRF)

REQUEST FOR SOP CHANGE					
Requestor Name:		Date of Initiation:			
Dept.:		SOP #:		Revision #:	Date:
SOP Title:					
Please Check One	MINOR REVISION		MAJOR REVISION		
CHANGE(S) (Use attachment if necessary):					
CHANGE FROM:					
CHANGE TO:					
REASON(S) FOR CHANGE(S):					
APPROVAL	NAME:	Signature/Date			
EPA Branch Chief / Section Chief/Team Leader					
EPA TOCOR					
REQUESTOR					
Effective Date					