Hazardous Waste Support Section
SOP No. HW-36A Revision 1
SOM02.2
Pesticide Data Validation

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NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by 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 variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment supersedes any guidance listed in this document.

Government contractors to the USEPA using this document to validate data should not hesitate to contact their Contracting Officer Representative with any questions regarding data validation or data package completeness.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

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ACRONYMS

%D	Percent Difference
%RSD	Percent Relative Standard Deviation
ARO	Aroclor
ASB	Analytical Services Branch
BFB	Bromofluorobenzene
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
HWSS	Hazardous Waste Support Section
INDA	Individual Standard Mixture A
INDB	Individual Standard Mixture B
INDC	Individual Standard Mixture C
LCS	Laboratory Control Sample
MS	Matrix Spike
MSD	Matrix Spike Duplicate
OSRTI	Office of Superfund Remediation and Technology Innovation
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAS	Routine Analytical Services

RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
$\overline{\textbf{RRF}}$	Mean Relative Response Factor
RRT	Relative Retention Time
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SCP	Single Component Pesticide
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCL	Target Compound List
TCLP	Toxicity Characteristics Leachate Procedure
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
TOPO	Task Order Project Officer
TR/COC	Traffic Report/Chain of Custody Record
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VTSR	Validated Time of Sample Receipt

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM02.2), and any future editorial revisions of SOM02.2, hereinafter referred to as the SOM02.2 SOW. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and 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 reviewer 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 availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
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 Quality Control (QC) criteria. The analyte may or may not be present in the sample.
C	This qualifier applies to results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS)
X	This qualifier applies to results when GC/MS analysis was attempted but unsuccessful

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.
If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/contacts.htm

HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

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

The analysis with the lower CRQL
The analysis with the better QC results
The analysis with the higher result

The analyte values and their respective CRQLs are then transferred into a single sample run. The runs that are not to be used are updated as "not reportable" or (N) in EDM.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A.	case number	i.e., 12345
B.	SDG name	i.e., BXY12
C.	level of validation performed	i.e., S3VE

Examples: **12345_BXY12_S3VE.xls**

12345_BXY12_S3VEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM02.2 SOW and any future editorial revisions of SOM02.2 for USEPA Region 2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) 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 supersede this Standard Operating Procedure. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
    c. Field duplicates;
    d. Field spikes;
    e. QC audit samples;
    f. Shipping dates;
    g. Preservatives; and
    h. Laboratories involved.

The TR/COC documentation includes sample 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 or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and USEPA Region 2.

Preservation

Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time [14 days from sample collection for

extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
4. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
5. If technical holding times are grossly exceeded, use professional judgment to qualify the data.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

Table 1. Holding Time Actions for Pesticide Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 7 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	Use professional judgment	
Non-Aqueous	No	≤ 14 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 14 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	Use professional judgment	

Gas Chromatograph with Electron Capture Detector (GC/ECD) Instrument Performance Check

Action:

1. Resolution Check Mixture
    a. The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column in order to use one Individual Standard Mixture (C). The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0% if two Individual Standard Mixtures (A and B) are to be used. If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.
        i. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ) (see Table 2).
        ii. Qualify non-detected compounds as unusable (R).
2. PEM
    a. If PEM analysis is not performed at the required frequency (see Pesticides Organic Analysis, of SOM02.2 NFG), qualify all associated sample and blank results as unusable (R).
    b. The resolution between any two adjacent peaks in the initial calibration and continuing calibration verification PEMs must be greater than or equal to 90% on each GC column. If PEM resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.
        i. Qualify detects as tentatively identified (NJ).
        ii. Qualify non-detects as unusable (R).
    c. If 4,4'-DDT breakdown is greater than 20.0%:
        i. Qualify detects for 4,4'-DDT as estimated (J).
        ii. Qualify detects for 4,4'-DDD and/or 4,4'-DDE as estimated (J).
        iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE were detected, qualify non-detects for 4,4'-DDT as unusable (R), and qualify detects for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity (NJ).
    d. If Endrin breakdown is greater than 20.0%:
        i. Qualify detects for Endrin as estimated (J).
        ii. Qualify detects for Endrin aldehyde and/or Endrin ketone as estimated (J).
        iii. If Endrin was not detected, but Endrin aldehyde and/or Endrin ketone were detected, qualify the non-detects for Endrin as unusable (R), and qualify detects for Endrin aldehyde and/or Endrin ketone as presumptively present at an approximated quantity (NJ).

e. If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0%, the reviewer should consider the degree of individual breakdown of 4,4'-DDT and Endrin and apply qualifiers as described in this section.

3. Mid-point Individual Standard Mixtures (A and B) or (C)
    a. If mid-point Individual Standard Mixture analysis is not performed at the required frequency (see Pesticides Organic Analysis, of SOM02.2 NFG), qualify all associated sample and blank results as unusable (R).
    b. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixtures (A and B) in the initial calibration and continuing calibration verification must be greater than or equal to 90.0% on each column. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixture (C) in the initial calibration and continuing calibration verification must be greater than or equal to 80.0% for the primary column and greater than or equal to 50.0% for the secondary column. If mid-point Individual Standard Mixtures (A and B) or (C) resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.
        i. Qualify detected target compounds that were not adequately resolved tentatively identified (NJ).
        ii. Qualify non-detects as unusable (R).
4. Note in the Data Review Narrative the potential effects on the sample data resulting from the instrument performance check criteria. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory has repeatedly failed to comply with the requirements for linearity, resolution, or 4,4'-DDT/Endrin breakdown.

Table 2. Gas Chromatograph with Electron Capture Detector (GC/ECD) Instrument Performance Check Actions

Criteria [Individual Standard Mixtures (A and B)]	Criteria (Individual Standard Mixture C)	Action
Resolution Check Mixture % Resolution <60.0%	Resolution Check Mixture % Resolution <80.0% (primary column) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R
PEM % Resolution <90.0%		Detects: NJ Non-detects: R
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected		Detects for 4,4'-DDT: J Detects for 4,4'-DDD: J Detects for 4,4'-DDE: J
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected		Non-detects for 4,4'- DDT: R Detects for 4,4'-DDD: NJ Detects for 4,4'-DDE: NJ
PEM: Endrin % Breakdown >20.0% and Endrin is detected		Detects for Endrin: J Detects for Endrin aldehyde: J Detects for Endrin ketone: J
PEM: Endrin % Breakdown >20.0% and Endrin is not detected		Non-detects for Endrin: R Detects for Endrin aldehyde: NJ Detects for Endrin ketone: NJ
PEM: Combined % Breakdown >30%		Apply qualifiers as described above considering degree of individual breakdown.
Mid-point Individual Standard Mixtures (A and B) % Resolution <90.0%	Mid-point Individual Standard Mixture (C) % Resolution <80.0% (primary column) Mid-point Individual Standard Mixture (C) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R
PEM analysis not performed at the required frequency (see Pesticides, of SOM02.2 NFG)		All results: R
Mid-point Individual Standard Mixtures analysis not performed at the required frequency (see Pesticides, of SOM02.2 NFG)		All results: R

Initial Calibration

Table 3. Concentration Levels of Calibration Standards

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3	CS4	CS5
alpha-BHC	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachlor-epoxide	5.0	10	20	40	80
4,4'-DDE	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
alpha-Chlordane	5.0	10	20	40	80
gamma-Chlordane	5.0	10	20	40	80
Tetrachloro-m-xylene	5.0	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160
Toxaphene	500	1000	2000	4000	8000

Action:

NOTES: At least one chromatogram from each of the Individual Standard Mixtures (A and B) or (C) must yield peaks that give recorder deflections between 50-100% of full scale.
Either peak area or peak height may be used to calculate the Calibration Factors (CFs) that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each CF for a given compound must be consistent. For example, if peak area is used to calculate the low-point CF for Endrin, the mid-point and high-point CFs for Endrin must also be calculated using peak area.

1. If the proper initial calibration sequence is not performed, or the steps of the initial calibration are not followed in the proper sequence, use professional judgment to evaluate

the effect on the data and notify the Contract Laboratory Program Project Officer (CLP PO) (see Table 4). This is especially critical for the low-level standards and non-detects.

2. If RT Windows are not calculated correctly, recalculate the windows and use the corrected values for all evaluations.
3. If the chromatogram display (recorder deflection) criteria are not met, use professional judgment to evaluate the effect on the data.
4. If the standard concentrations listed in Table 3 are not used, use professional judgment to evaluate the effect on the data and notify the CLP PO. This is especially critical for the low-level standards and non-detects.
5. The Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target compounds must be less than or equal to 20.0%, except for alpha-BHC and delta-BHC. The %RSD of the CFs for alpha-BHC and delta-BHC must be less than or equal to 25.0%. The %RSD of the CFs for each of the Toxaphene peaks must be $\leq$ 30% when 5-point ICAL is performed. The %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be less than or equal to 30.0%. If the %RSD criteria are not met, qualify detects as estimated (J) and use professional judgment to qualify non-detected target compounds.
6. If the %RSD criteria are within allowable limits, no qualification of the data is necessary.
7. At the reviewer's discretion, and based on the project-specific data quality objectives, consider a more in-depth review using the following guidelines:
    a. If any pesticide target compound has a %RSD greater than the maximum criterion, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
        i. Qualify detects for that compound(s) as estimated (J).
        ii. Qualify non-detected pesticide target compounds using professional judgment.
    b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
        i. No qualifiers are required for detects in the linear portion of the curve.
        ii. Qualify detects outside of the linear portion of the curve as estimated (J).
        iii. No qualifiers are required for pesticide target compounds that were not detected.
    c. If the low-point of the curve is outside of the linearity criteria:
        i. No qualifiers are required for detects in the linear portion of the curve.
        ii. Qualify low-level detects in the area of non-linearity as estimated (J).
        iii. For non-detected pesticide compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration. Notify the CLP PO if the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, RT, or resolution.
9. Qualify data for Toxaphene sharing the same RT Window with any Single Component Pesticide (SCP) in any Individual Standard Mixture using professional judgment.

Table 4. Initial Calibration Actions for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
Initial calibration is not performed or not performed in the proper sequence	Use professional judgment and notify CLP PO	
%RSD exceeds allowable limits*	J	Use professional judgment
%RSD within allowable limits*	No qualification	

* %RSD $\leq$ 20.0% for single component target compounds except alpha-BHC and delta-BHC.
%RSD $\leq$ 25.0% for alpha-BHC and delta-BHC.
%RSD $\leq$ 30.0% for Toxaphene peaks.
%RSD $\leq$ 30.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

Continuing Calibration Verification (CCV)

Action:

1. The RT Windows are used in qualitative identification. If the standards do not fall within the RT Windows, carefully evaluate the associated sample results (see Table 5). All samples injected after the last in-control standard are potentially affected.
    a. For non-detected target compounds in the affected samples, check to see if the sample chromatograms contain any peaks that are close to the expected RT Window of the pesticide of interest.
        i. If no peaks are present, consider non-detected values to be valid and no qualification of the data is necessary.
        ii. If any peaks are present close to the expected RT Window of the pesticide of interest, use professional judgment to qualify the non-detects as presumptively present (NJ).
    b. For detected compounds in the affected samples, if the peaks are within the RT Window, no qualification of the data is necessary. However, if the peaks are close to the expected RT Window of the pesticide of interest, the reviewer may take additional effort to determine if sample peaks represent the compounds of interest.
    For example, the reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT Window can be re-evaluated using the Mean Retention Times (RTs) of the standards.
        i. If the peaks in the affected sample fall within the revised window, qualify detects as tentatively identified (NJ).
        ii. If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable (R).
2. For the PEM, if the Percent Difference is not within ±25.0%, qualify associated detects as estimated (J) and non-detects as estimated (UJ).
3. For the Calibration Verification Standard (CS3), if the Percent Difference is not within ±25.0%, qualify associated detects as estimated (J) and non-detects as estimated (UJ).
4. If 4,4'-DDT %Breakdown is > 20.0%, qualify detected 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE as estimated (J). When 4,4'-DDT is not detected, but 4,4'-DDD and 4,4'-DDE are detected, qualify non-detected 4,4'-DDT as unusable (R) and detected 4,4'-DDD and 4,4'-DDE as presumptively present with estimated concentration (NJ).
5. If Endrin %Breakdown > 20.0%, qualify detected Endrin, Endrin aldehyde and Endrin ketone as estimated (J). When Endrin is not detected, but Endrin aldehyde and Endrin ketone are detected, qualify non-detected Endrin as unusable (R) and detected Endrin aldehyde and Endrin ketone as presumptively present with estimated concentration (NJ). If more than 14 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C), qualify all data as unusable (R). If the combined %

Breakdown for 4,4'-DDT and Endrin is > 30.0%, consider the degree of individual breakdown of 4,4'-DDT and Endrin and qualify as in Sections 4 and 5 accordingly.

6. If more than 12 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence, qualify all data as unusable (R).
7. If more than 72 hours has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3), qualify all data as unusable (R).
8. If the Percent Difference, time elapsed, and RTs are within acceptable limits, no qualification of the data is necessary.
9. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration.

Table 5. Continuing Calibration Verification (CCV) Actions for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT window	Use professional judgment	
%D not within limits*	J	UJ
Time elapsed is greater than acceptable limits**	R	
%D, time elapsed, and RT are within acceptable limits	No qualification	
PEM: 4,4'-DDT %Breakdown >20.0% 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 >20.0% and 4,4'-DDT is not detected	R for 4,4'-DDT	NJ for 4,4'-DDD and 4,4'-DDE
PEM: Endrin %Breakdown >20.0% and Endrin is detected	J for Endrin, Endrin aldehyde, Endrin ketone	No qualification
PEM: Endrin %Breakdown >20.0% and Endrin is not detected	R for Endrin	NJ for aldehyde and Endrin ketone
PEM: Combined % Breakdown > 30%	Apply qualifiers as described above considering degree of individual breakdown	Apply qualifiers as described above considering degree of individual breakdown

* See Actions 2 and 3

** See Actions 5, 6, and 7

Blanks

Action:

NOTES: The concentration of non-target compounds in all blanks must be less than or equal to 10 µg/L.
The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method.
Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

NOTES: "Water blanks, "drill blanks", and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.
All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compounds, instrument performance criteria, and spectral or calibration QC problems.
Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Samples taken from a drinking water tap do not have associated field blanks.
When applied as described in Table 6 below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a target SCP or Toxaphene is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 6).
2. If a target SCP or Toxaphene concentration in a method, field, or trip blank is less than the CRQL and:
    a. the sample concentration is less than the CRQL, report the CRQL value with a "U".
    b. the sample concentration is greater than or equal to the CRQL, no qualification is required.
3. If a target SCP or Toxaphene concentration in a method, field, or trip blank is greater than the CRQL and:
    a. the sample concentration is less than the CRQL, report the CRQL value with a "U".
    b. the sample concentration is greater than or equal to the CRQL, and less than or equal to the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a "U".

   c. the sample concentration is greater than or equal to the CRQL and greater than the blank concentration, no qualification is required.
4. If a target SCP or Toxaphene concentration in a method, field, or trip blank is equal to the CRQL and:
   a. the sample concentration is less than or equal to the CRQL, report the CRQL value with a "U".
   b. the sample concentration is greater than the CRQL, no qualification is required.
5. If gross contamination exists (i.e., saturated peaks, "hump-o-grams", "junk" peaks), raise the CRQL to the level of the blank contamination and report the associated sample data below this level as CRQL-U. Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
6. If contaminants are found in the method, field, or trip blanks, the following is recommended:
   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
      i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.
      ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.
7. If method blank data are unavailable, the reviewer may use professional judgment or substitute field blank data for missing method blank data.

NOTE: There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. 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 result.

Table 6. Blank Actions for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Sulfur Cleanup, Instrument, Field, Trip,TCLP/SPLP*	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL and > blank concentration	No qualification required
	= CRQL	≤ CRQL	Report CRQL value with a U
		> CRQL	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

*Note: For Trip blank qualification please contact TOCOR

Surrogate Spikes

Action:

If either surrogate spike recovery is outside the acceptance limits, the reviewer must consider the existence of coelution and interference in the raw data and use professional judgment to qualify data, as surrogate recovery problems may not directly apply to target analytes.

1. For any surrogate recovery greater than 150% (see Table 7):
    a. Qualify detected target compounds as biased high (J+).
    b. Do not qualify non-detected target compounds.
    c. Diluted samples with dilution factor less than or equal to 5 should be qualified for surrogate recovery outside criteria. Dilution factor greater than 5, no qualification applied.
2. If both surrogate recoveries are greater than or equal to 30%, and less than or equal to 150%, no qualification of the data is necessary. Diluted samples with dilution factor less than or equal to 5 should be qualified for surrogate recovery outside criteria. Dilution factor greater than 5, no qualification applied.
3. For any surrogate recovery greater than or equal to 10%, and less than 30%:
    a. Qualify detected target compounds as biased low (J-).
    b. Qualify non-detected target compounds as approximated (UJ).
    c. Diluted samples with dilution factor less than or equal to 5 should be qualified for surrogate recovery outside criteria. Dilution factor greater than 5, no qualification applied.
4. For any surrogate recovery less than 10%, the reviewer should examine the sample chromatogram to assess the qualitative validity of the analysis. If low surrogate recoveries are from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:
    a. Qualify detected target compounds as biased low (J-).
    b. Qualify non-detected target compounds as unusable (R).
    c. Diluted samples with dilution factor less than or equal to 5 should be qualified for surrogate recovery outside criteria. Dilution factor greater than 5, no qualification applied.
5. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of 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. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. Note, for Contract Laboratory Program Project Officer (CLP PO) action, analytical problems even if this judgment allows some use of the affected data.
6. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.
7. If surrogate RTs are within RT windows, no qualification of the data is necessary.

8. If the two surrogates were not added to all samples, MS/MSDs, standards, LCSs, and blanks, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

Table 7. Surrogate Actions for Pesticide Analyses

Criteria	Action*	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 150%	J+	No qualification
30% < %R < 150%	No qualification	
10% < %R < 30%	J-	UJ
%R < 10% (sample dilution not a factor)	J-	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

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

* Dilution samples with dilution factor less than or equal to 5 should be qualified for surrogate recovery outside criteria. Dilution factor greater than 5, no qualification applied.

Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

NOTES:

Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

NOTE: For a Matrix Spike that does not meet criteria, **apply the action to only the field sample used to prepare the Matrix Spike sample.** If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

1. 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.
2. MS/MSD %R and the Relative Percent Difference (RPD) between MS and MSD shall be calculated according the method, and shall be within the acceptance limits in Table 8.
3. MS/MSD actions are given in Table 9.

Table 8. MS/MSD %R and RPD Limits for Pesticide Analysis

Compound	%R for Water Samples	RPD for Water Samples	% R for Soil/Sediment Samples	RPD for Soil/Sediment Samples
Gamma-BHC (Lindane)	56-123	0 - 15	46 - 127	0 - 50
Heptachlor	40 - 131	0 - 20	35 - 130	0 - 31
Aldrin	40 - 120	0 - 22	34 - 132	0 - 43
Dieldrin	52 - 126	0 - 18	31 - 134	0 - 38
Endrin	56 - 121	0 - 21	42 - 139	0 - 45
4,4'-DDT	38 - 127	0 - 27	23 - 134	0 - 50

Table 9. MS/MSD Actions for Pesticide Analysis

Criteria	Action	
	Detect	Non-detect
%R < 20%	J	R
20% ≤ %R< Lower limit	J	UJ
Lower Acceptance ≤ %R; RPD ≤ Upper Acceptance Limit	No Qualification	No Qualification
%R or RPD > Upper Acceptance Limit	J	No Qualification

Laboratory Control Samples (LCSs)

Table 10. Pesticides Laboratory Control Sample (LCS) Spike Compounds and Recovery Limits

LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 – 120
Heptachlor epoxide	50 – 150
Dieldrin	30 – 130
4,4'-DDE	50 – 150
Endrin	50 – 120
Endosulfan sulfate	50 – 120
trans-Chlordane	30 – 130
Tetrachloro-m-xylene (surrogate)	30 – 150
Decachlorobiphenyl (surrogate)	30 – 150

Action:

NOTES: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.
All samples prepared and analyzed with an LCS that does not meet the technical acceptance criteria in the method will require re-extraction and re-analysis.

If the LCS criteria are not met, laboratory performance and method accuracy are in question. Use professional judgment to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

1. If the LCS recovery criteria are not met, use the LCS results to qualify sample data for the specific compounds that are included in the LCS solution (see Table 11).
    a. If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as estimated (J). Do not qualify non-detected target compounds.
    b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as estimated (J) and non-detects as unusable (R).
    c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.
    d. Use professional judgment to qualify non-LCS compounds. Take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.
2. If the LCS recovery is within allowable limits, no qualification of the data is necessary.
3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if a laboratory fails to analyze an LCS with each Sample Delivery Group (SDG), or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

Table 11. Laboratory Control Sample (LCS) Recovery Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

Florisil Cartridge Performance Check

Action:

NOTE: Florisil cartridge cleanup is mandatory for all extracts.

1. If the Florisil Cartridge Performance Check criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows (see Table 12):
   a. If the Percent Recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
   b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
   c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
   d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J) and qualify non-detected target compounds as unusable (R).
   e. If the Percent Recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.
2. Note in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Table 12. Florisil Cartridge Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > 120% (pesticide target compounds)	J	No qualification
80% ≤ %R ≤ 120%	No qualification	
10% ≤ %R <80% (pesticide target compounds)	J	UJ
%R < 10% (pesticide target compounds)	J	R
%R > 5% (2,4,5-trichlorophenol)	Use professional judgment*	

* Check sample chromatogram for interferences.

Gel Permeation Chromatography (GPC) Performance Check

Action:

NOTE: GPC cleanup is mandatory for all soil samples.

1. If GPC 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 in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.
2. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect. Qualify detected compounds as estimated (J) (see Table 13). Qualify all non-detected target compounds as unusable (R).
3. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
4. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
5. If high recoveries (i.e., greater than 120%) were obtained for the pesticides and surrogates during the GPC calibration check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
6. Note in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

Table 13. Gel Permeation Chromatography (GPC) Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R < 10% (pesticide target compounds)	J	R
10% ≤ %R < 80%	J	UJ
80% ≤ %R ≤ 120%	No qualification	
%R > 120% (pesticide target compounds)	J	No qualification

Target Compound Identification

Criteria:

1. The Retention Times (RTs) of both of the surrogates and reported target compounds in each sample must be within the calculated RT Windows on both columns. Tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the Mean RT (RT) determined from the initial calibration and Decachlorobiphenyl (DCB) must be within ±0.10 minutes of the RT determined from the initial calibration.
2. The Percent Difference (%D) for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns must be within the inclusive range of ±25.0.
3. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low-point standard of the initial calibration associated with those analyses.
4. Chromatograms must display Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.
5. If an extract must be diluted, chromatograms must display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale.
6. For any sample, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB.
7. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

Action:

1. If the qualitative criteria for both columns were <u>not</u> met, all target compounds that are reported as detected should be considered non-detected. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:
   a. If the detected target compound peak was sufficiently outside the pesticide RT Window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.
   b. If the detected target compound peak poses an interference with potential detection of another target peak, the reported value should be considered and qualified as unusable (R).
2. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound

should be included. Note in the Data Review Narrative all conclusions made regarding target compound identification.

3. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.
4. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, use professional judgment to determine how best to report, and if necessary, qualify the data according to the guidelines in Table 14 below.
5. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of Toxaphene is strongly suggested, report results as presumptively present (N).

Table 14. Action on Qualifying Positive Pesticide Result

Percent Differences	Qualifier
0% – 25%	No qualification
26% – 70%	J
71 – 200% (interferences detected)*	JN
> 50% (pesticide value < CRQL)**	U
> 200%	R

* When interferences are detected on either column, qualify the data as "JN".

** When the pesticide value is below CRQL and %D > 50%, raise the value to CRQL and qualify "U" undetected.

Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

Action:

NOTE: This confirmation is not usually provided by the laboratory. In cases where it is provided, use professional judgment to determine if data qualified with "C" can be salvaged if it was previously qualified as unusable (R).

1. If the quantitative criteria for both columns were met (≥ 5.0 ng/µL for SCPs and ≥ 125 ng/µL for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following guidance (see Table 15):
    a. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".
    b. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

Table 15. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation Actions

Criteria	Action
SCP/Toxaphene was confirmed by GC/MS	Detects C
SCP/Toxaphene was not confirmed by GC/MS	Detects X

Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Action:

1. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample.
2. Results between the MDL and CRQL should be qualified as estimated (J).
3. Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.
4. For non-aqueous samples, if the percent solids is greater than 30.0%, no qualification of the data is necessary. If the percent solids is less than or equal to 30.0% and greater than 10.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent solids is less than or equal to 10.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table 16).
5. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

Table 16. Percent Solids Actions for Pesticide Analysis For Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Solids > 30.0	No qualification	
10.0 < % Solids < 30.0	J	UJ
% Solids < 10.0	J	R

Field Duplicates

Action:

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

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

Overall Assessment of Data

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

APPENDIX A: GLOSSARY

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 (ICSs); 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.
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.
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.

Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

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.

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.

APPENDIX B: ORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DESA/HWSB/HWSS
2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No. :
Site:
Number of Samples:
Analysis:

SDG No.:
Laboratory:
Sampling dates:

QAPP
HWSS #:
Contractor Document #:

SUMMARY:

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

Critical Findings:

Major Findings:

Minor Findings:

COMMENT:

Reviewer Name(s):

Approver's Signature: **Date:**

Name:

Affiliation: **USEPA/R2/HWSB/HWSS**

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APPENDIX C: SAMPLE ORGANIC DATA SAMPLE SUMMARY

Case No:	00001	Contract:	XY1234	SDG No:	XY123	Lab Code:	00001
Sample Number:	BA1X5	Method:	Pest	Matrix:	Soil	MA Number:	
Sample Location:		pH:	8.2	Sample Date:	13322059	Sample Time:	13:08:00
% Moisture :	56.85			% Solids :			

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
2,4,5-T	3.9	ug/kg	1.0	U	U	Yes	S3VEM
2,4,5-TP (Silvex)	3.9	ug/kg	1.0	U	U	Yes	S3VEM
2,4-D	39	ug/kg	1.0	U	U	Yes	S3VEM
2,4-DB	39	ug/kg	1.0	U	U	Yes	S3VEM
Dalapon	97	ug/kg	1.0	U	U	Yes	S3VEM
Dicamba	3.9	ug/kg	1.0	U	U	Yes	S3VEM
Dichloroprop	39	ug/kg	1.0	U	U	Yes	S3VEM
Dinoseb	19	ug/kg	1.0	U	U	Yes	S3VEM
MCPA	390	ug/kg	1.0	U	UJ	Yes	S3VEM
MCPP	390	ug/kg	1.0	U	UJ	Yes	S3VEM
Pentachlorophenol	3.9	ug/kg	1.0	U	U	Yes	S3VEM
4-Nitrophenol	39	ug/kg	1.0	U	U	Yes	S3VEM

APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR_DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR_DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		