USEPA
Hazardous Waste Support Branch
Validating Pesticide Compounds
Organochlorine Pesticides By Gas Chromatography
SW-846 Method 8081B

Prepared by: _____ Date: 12/29/10
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Annual Review

Reviewed by: _____ Date: _____
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Reviewed by: _____ Date: _____
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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8081B November 2000. Method 8081B is used to determine the concentration of pesticide compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8081B, Method 8000C and the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January 2005. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Methods and National Functional Guidelines mentioned above.

DEFINITIONS

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DEFINITIONS

Acronyms

CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB - decachlorobiphenyl
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
ℓ - liter
mℓ - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor

RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCMX -tetrachloro-m-xylene
TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z - Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ SDG# _____
LAB:_____ SITE:_____

	YES	NO	N/A

1.0 Data Completeness and Deliverables

1.1 Has all the data been submitted in CLP deliverable format? [ ] ___ ___

1.2 Have any missing deliverables been received and added to the data package? [ ] ___ ___

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

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [ ] ___ ___

2.2 Are the case number and/or SDG number contained in the narrative or cover letter? [ ] ___ ___

3.0 Data Validation Checklist

3.1 Does this data package contain:

Water data? [ ] ___ ___

Waste data? [ ] ___ ___

Soil/solid data? [ ] ___ ___

ORGANOCHLORINE PESTICIDE

YES NO N/A

1.0 Traffic Reports and Laboratory Narrative

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

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

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

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated, "J." If a soil sample, other than TCLP, contains more than 90% water, all non detects are qualified as unusable, "R", and positive results flagged “J”.

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

2.0 Holding Times

2.1 Have any organochlorine pesticide technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

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

ACTION: Qualify sample results according to Table 1.

Table 1. Holding Time Criteria

Matrix	Preserved	Criteria	Action	
			Detected compounds	Non-detected compounds
Aqueous	No	≤ 7 days(extraction) ≤ 40 days(analysis)	J*	UJ*
	No	> 7 days(extraction) > 40 days(analysis)	J*	UJ
	Yes	≤ 7 days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 7 days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R
Non-aqueous	No	≤ 14days(extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14days(extraction) >40 days(analysis)	J	UJ
	Yes	≤ 14days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 14days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R

* only if cooler temperature exceeds 10°C; no action required if cooler temperature < 10°C.

YES NO N/A

3.0 Surrogate Recovery (Form II/Equivalent)

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

a. Water/Waste

[ ] ___ ___

b. Soil/Solid

[ ] ___ ___

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

a. Water

[ ] ___ ___

b. Waste

[ ] ___ ___

c. Soil/Solid

[ ] ___ ___

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

3.3 Are all recovery limits for the surrogates TCMX and DCB between 30-150% for all samples, including MS and MSDs, LCSs and all blanks?

[ ] ___ ___

Note: Reviewer shall use lab in-house recover limits if available. In-house criteria should be examined for reasonableness.

YES NO N/A

ACTION: Circle all outliers in red. Follow surrogate action Table 2.

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

ACTION: Follow surrogate action, Table 2 below.

Table 2. Surrogate Recovery Criteria

Criteria	Action	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 200%	J	Use professional judgement
150% < %R ≤ 200%	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 judgement	
RT out of RT window	Use professional judgement	
RT within RT window	No qualification	

3.6 Are there any transcription/calculation errors between raw data and Form II? [ ] ___ ___

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

YES NO N/A

4.0 Laboratory Control Sample(LCS)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples. [ ] ___ ___

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

4.2 Were Laboratory Control Samples analyzed at the required concentration for all analytes of interest as specified in Table 3 below. [ ] ___ ___
Note: Use lab in-house criteria, if available.

Table 3. LCS Spiking Criteria

LCS Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml	Recovery Limits (%)
gamma-BHC	0.05	1	50-120
Heptachor epoxide	0.05	1	50-120
Dieldrin	0.01	1	30-130
4,4'-DDE	0.01	1	50-150
Endrin	0.01	1	50-120
Endosulfan sulfate	0.01	1	50-120
gamma-Chloradane	0.05	1	30-130
Tetrachloro-m-xylene(surrogate)	0.20	3	30-150
Decachlorobiphenyl (surrogate)	0.40	3	30-150

YES NO N/A

Note: The LCS might be spiked with the same analytes at the same concentration as the matrix spike.

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

4.3 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in table above? [ ] ___ ___

ACTION: For LCS % recovery not meeting the required recovery, follow the required action in Table 4 below.

Table 4. LCS Recovery Criteria

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

5.0 Matrix Spikes (Form III/Equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix? [ ] ___ ___

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

YES NO N/A

5.2 Have MS/MD or MS/MSD results been summarized on Form III/Equivalent? [ ] ___ ___

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

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000B-39, section 8.5.])

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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

5.4 We Were Matrix Spike Samples analyzed at the required concentration for all analytes of interest as specified in Table 5 below. [ ] ___ ___

Note: Spiking analytes may differ from those in Table 5. Check QA project plan or task order.

YES NO N/A

Table 5. Matrix Spiking Criteria

Matrix Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml
gamma-BHC	0.05	1
Heptachor	0.05	1
Aldrin	0.05	1
Dieldrin	1.0	1
Endrin	1.0	1
4,4'-DDT	1.0	1

Note: For aqueous organic extractable, the spike concentration should be:

1) For regulatory compliance monitoring - the regulatory concentration limit or 1 to 5 times the expected background concentration, whichever is higher;

2) For all other aqueous samples - the larger of either 1 to 5 x times the expected background concentration, or the same as the QC check sample concentration (see section 4 above);

3) For soil/solid and waste samples - the recommended concentration is 20 times the estimated quantitation limit (EQL).

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

5.5 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in Table 6 below. [ ] ___ ___

Note: Use lab in-house criteria, if available.

YES NO N/A

Table 6. Matrix Spike Recovery Criteria

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
gamma-BHC	56-123	0-15	46-127	0-50
Heptachor	40-13	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

NOTE: The actual number of MS analytes depends on the number analytes being measured (e.g., total number of MS plus MSD compounds). If only chlordane or toxaphene are the analytes of interest, the spiked sample should contain the most representative multi-component analyte.

ACTION: Follow the matrix spike actions (Table 7) for pesticide analyses.

Table 7. Matrix Spike Qualifying Criteria

Criteria	Action	
	Detected Associated Compounds	Non-Detected Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% R ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgement
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualifications	

YES NO N/A

Note: When the results of the matrix spike analyses indicates a potential problem due to the sample matrix itself, the LCS results are used to verify the laboratory can perform analyses in a clean matrix.

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on Method Blank Summary form(s) (Form IV)?

6.2 Frequency of Analysis: Has a reagent blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?

[ ] ___ ___

Note: Method blank should be analyzed, either after the calibration standard or at any other time during the analytical shift.

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.3 Chromatography: review the blank raw data -chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for pesticides? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent/cleanup blanks have positive results for organochlorine pesticides? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ] ___

YES NO N/A

7.2 Do any field/rinse blanks have positive organochlorine pesticide results?

___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 8 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 8. Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Clean up, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U
		≥ CRQL and ≥ blank contamination	No qualification
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	Gross contamination	Detects	Qualify results as unusable R

Note: Analytes qualified “U” for blank contamination are treated as “hits” when qualifying the calibration criteria.

YES NO N/A

Note: When applied as described in Table 8 above, the contaminant concentration in the blank is multiplied by the sample dilution factor.

NOTE: If gross blank contamination exists(e.g., saturated peaks, “hump-o-grams”, “junk peaks”), all affected positive compounds in the associated samples should be qualified as unusable “R”, due to interference.

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.

7.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 Gas Chromatography with Electron Capture Detector (GC/ECD)Instrument Performance Check (CLP Form VI and Form VII Equivalent)

8.1 Was the proper gas chromatographic column used for the analysis of organochlorine pesticides? Check raw data, instrument logs, or contact the lab to determine what type of columns were used.(See Method 8081B-8, section 4.2)

[ ] ___ ___

8.2 If capillary columns were used, were they both wide bore (.53 mm ID) fused silica GC columns, such as DB-608 and DB-1701 or equivalent. Indicate the specific type of column used for:

column 1: _____

column 2: _____

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

YES NO N/A

9.0 Calibration and GC Performance

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

a.	DDT/endrin breakdown check	[ ]	___	___
b.	toxaphene	[ ]	___	___
c.	technical chlordane	[ ]	___	___
d.	5 pt. initial calibration standards	[ ]	___	___
e.	calibration verification standards	[ ]	___	___
f.	LCS	[ ]	___	___
g.	Method blanks	[ ]	___	___

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

9.2 Has a DDT/endrin breakdown check standard (at the mid-concentration level) been analyzed at the beginning of each analytical sequence on both columns (page 8081B-24, section 8.2.3)? [ ] ___ ___

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

9.3 Has the individual % breakdown exceeded 20.0% on either column for:- 4,4' - DDT? ___ [ ] ___

ACTION: If any % breakdown has failed the QC criteria in the breakdown check standard, qualify all sample analyses in the entire analytical sequence as described below.

a. If 4,4'-DDT breakdown is greater than 20.%:

i. Qualify all positive results for DDT with 'J". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable ("R").

YES NO N/A

ii. Qualify positive results for DDD and DDE as presumptively present at an approximated quantity ("NJ").

b. If endrin breakdown is greater than 20.0%:

i. Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable ("R").

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity ("NJ").

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

NOTE: If internal standard calibration procedure is used (page 8000B-16, section 7.4.2.2), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (page 8000B-16, section 7.4.2.1), then calibration factors must be used.

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

9.5 Are there any transcription/calculation errors between raw data and data summary forms.

___ [ ] ___

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

9.6 Are standard retention time (RT) windows for each analyte of interest presented on modified CLP summary forms? [ ] ___ ___

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

YES NO N/A

NOTE: Retention time windows for all pesticides are established using retention times from three calibration standards analyzed during the entire analytical sequence (page 8081B-15, section 7.4.6). A 72 hr. sequence is not required with this method, however, the method states that best results are obtained using retention times which span the entire sequence; i.e., using the mid level from the 5 pt. calibration, one of the mid-concentration standards analyzed during mid-sequence and one analyzed at the end.

9.7 Were RT windows on the confirmation column established using three standards as described above? [ ] ___ ___

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.6 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

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

9.8 Do all standard retention times in each level of the initial 5 pt. calibrations for pesticides fall within the windows established during the initial calibration sequence? [ ] ___ ___

ACTION: i. If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standards, spanning the entire sequence were used to obtained RT windows.If the lab used three standards from the 5 pt., RT windows may be too tight. If so, RT windows should be recalculated as per page 8081B-15, section 7.4.6.2

ii. Alternatively, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times.

If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

ACTION: For toxaphene and chlordane, the RT may be outside the RT window, but these analytes may still be identified from their individual patterns.

YES NO N/A

9.9 Has the linearity criteria for the initial calibration standards been satisfied for both columns? (% RSD must be < allowable limits* for all analytes). [ ] ___ ___

ACTION: If no, follow the actions in Table 9 below.

Table 9. Initial Calibration Linearity Criteria

Criteria	Criteria	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
% RSD exceeds allowable limits*	J	No qualification
% RSD within allowable limits*	NO qualifications	

* %RSD ≤ 20% for single component compounds except alpha-BHC and delta- BHC.
%RSD ≤ 25% for alpha-BHC and delta-BHC
%RSD ≤ 30% for Toxaphene peaks
%RSD ≤ 30% for surrogates(tetrachloro-m-xylene and decachlorobiphenyl).

9.10 Has a calibration verification standard containing all analytes of interest been analyzed on each working day, prior to sample analyses (pages 8081B-15,sections 7.5.2)? [ ] ___ ___

9.11 Has a calibration verification standard also been analyzed after every 10 samples and at the end of each analytical sequence (page 8081B-15, section 7.5.2)? [ ] ___ ___

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

9.12 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence (closing CCV). Has no more than 72 hours elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene CCV? [ ] ___ ___

ACTION: See Table 10 below.

9.13 Has the percent difference (%D) exceeded ± 20% for any organochlorine pesticide analyte in any calibration verification standard? ___ [ ] ___

YES NO N/A

9.13 Has a new 5 pt. calibration curve been generated for those analytes which failed in the calibration verification standard (page 8081B-16, section 7.5.2.2), and all samples which followed the out-of-control standard (page 8081B-16, section 7.5.2.3)reinjected? [ ] ___ ___

ACTION: If the %D for any analyte exceeded the ± 20% criterion and the instrument was not recalibrated for those analytes, see table below.

9.15 Have daily retention time windows been properly calculated for each analyte of interest (page 8081B-16, section 7.5.3)), using RTs from the associated mid concentration standard and standard deviation from the initial calibration)? [ ] ___ ___

ACTION: If no, take action specified in section 3.2 above or recalculate RT windows using the procedure outlined in method 8081B-16, section 7.5.3.

9.16 Do all standard retention times for each mid concentration standard fall within the windows established during the initial calibration sequence? [ ] ___ ___

9.17 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows (page 8081B-16, section 7.5.3)? [ ] ___ ___

ACTION: If the answer to either 9.15 or 9.16 above is no, check the chromatograms of all samples which followed the last in-control standard. All samples analyzed after the last in-control standard must be re-injected, if initial analysis indicated the presence of the specific analyte that exceeded the retention time criteria (page 8081B-18, section 7.5.7.). If samples were not re-analyzed, document under Contract Non-compliance in the Data Assessment.

YES NO N/A

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

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

See Table 10 below.

Table 10. CCV Criteria

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT window	Use professional judgement	
%D not within +/- 20%	J	UJ
Time elapsed greater than section 9.12 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

9.18 Are there any transcription/calculation errors between raw data and data summary forms? ___ [ ] ___

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

YES NO N/A

10.0 Analytical Sequence Check (Form VIII-PEST/Equivalent)

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

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

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

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

11.0 Extraction Method Cleanup Efficiency Verification (Form IX/Equivalent)

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

1. Aqueous samples:

1. Separatory funnel (Method 3510)_____

2. Continuous liquid-liquid extraction (Method 3520)_____

3. Solid phase extraction (Method 3535)_____

4. Other _____

YES NO N/A

2. Solid samples:

1. Soxhlet (Method 3540)_____
2. Automated Soxhlet (Method 3541)_____
3. Pressurized fluid (Method 3545) _____
4. Microwave extraction (Method 3546)_____
5. Ultrasonic extraction (Method 3550)_____
6. Supercritical fluid (Method 3562) _____
7. Other _____

11.2 Is Form IX - Pest-1/Equivalent present and complete for each lot of Florisil/Cartridges used? (Florisil Cleanup, Method 3620A, is required for all organochlorine pesticide extracts.)
[ ] ___ ___

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

NOTE: Method 3620A uses Florisil, while the SOW/CLP allows for Florisil cartridges. Method 3620A does not list which pesticides and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct pesticide list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.3 Are all samples listed on modified CLP Pesticide Florisil/Cartridge Check Form?
[ ] ___ ___

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

11.4 If GPC Cleanup was performed, is Form IX - Pest-2/Equivalent present?
[ ] ___ ___

ACTION: If GPC was not performed and sample results indicate significant sulfur interference, make note in the data assessment.

YES NO N/A

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

11.5 Were the same compounds on Form IX used to check the efficiency of the cleanup procedures? [ ] ___ ___

11.6 Are percent recoveries (% R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits listed on Form IX:

80-120% for florisil cartridge check? [ ] ___ ___

80-110% for GPC calibration? [ ] ___ ___

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

ACTION: If % R are < 80%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for pesticide compounds. Qualify positive results “J” (estimated).

NOTE: If 2,4,5-trichlorophenol was used to measure the efficiency of the Florisil cleanup and the recovery was > 5%, sample data should be evaluated for potential interferences.

12.0 Pesticide Identification

12.1 Has CLP Form X, showing retention time data for positive results on the two GC columns, been completed for every sample in which a pesticide was detected? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and Florisil cleanup verification forms)? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error in the data assessment.

YES NO N/A

12.3 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

Note: Confirmation can be supported by other qualitative techniques such as GC/MS (Method 8270), or GC/AED (Method 8085) if sensitivity permits.

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently (see section 9.7 above).Also check for false negatives among the multiple peak compounds toxaphene and chlordane. Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation used as the second column Confirmation? (This is not required). [ ] ___ ___

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns<25.0%? [ ] ___ ___

NOTE: The method 8081B requires quantitation from one column.The second column is to confirm the presence of an analyte. Calibration for the Confirmation column is a one point calibration. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

YES NO N/A

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

% Difference	Qualifier
0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected)	"NJ"
>50%(Pesticide vale is <CRQL)	"U"
>201%	“R”

Note: The lower of the two values is reported on Form I.If using professional judgement,the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found?

___ [ ] ___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___ ___

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

YES NO N/A

ACTION: When a sample is analyzed at more than one dilution, the lowest EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace

concentrations that exceed the calibration range in the original analysis by crossing out the value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable? [ ] ___ ___

14.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for organochlorine pesticide analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative.However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made to determine the proper identification of field duplicates.