

USEPA  
Hazardous Waste Support Branch  
Validating PCB Compounds  
PCBs By Gas Chromatography SW-846 Method 8082A



Prepared by: George Karras Date: 12/8/06  
George Karras, Chemist  
Hazardous Waste Support Section

Prepared by: Russell Arnone Date: 12-8-06  
Russell Arnone, Chemist  
Hazardous Waste Support Section

Concurred by: Linda Mauel Date: 12/11/06  
Linda Mauel, Chief  
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 12/11/06  
Robert Runyon, Chief  
Hazardous Waste Support Branch

Annual Review

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_  
Name

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_  
Name

## **INTRODUCTION**

### **Scope and Applicability**

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8082A" November 2000. Method 8082A is used to determine the concentration of PCB compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8082A, 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.

Yes NO N/A

## DEFINITIONS

### Acronyms

BNA - base neutral acid(another name for Semi Volatiles)  
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

Yes NO N/A

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.
- 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.

Yes NO N/A

**PACKAGE COMPLETENESS AND DELIVERABLES**

CASE NUMBER: \_\_\_\_\_ SDG# \_\_\_\_\_

LAB: \_\_\_\_\_ SITE: \_\_\_\_\_

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? \_\_\_\_\_

**POLYCHLORINATED BIPHENYLS**

1.0 Traffic Reports and Laboratory Narrative

1.1 Are traffic report and chain-of-custody forms present for all samples?  \_\_\_ \_\_\_

Yes NO N/A

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, non detects shall be qualified as unusable, "R."

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 PCB technical holding times, determined from date of collection to date of extraction, been exceeded?

\_\_\_ [ ] \_\_\_

Water and waste samples for PCB 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: If technical holding times are exceeded, flag all positive results as estimated, "J," and sample quantitation limits "UJ" and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be

Yes NO N/A

qualified "J", but the reviewer may determine that non-detects are unusable, "R." (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.

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

[ ] \_\_\_ \_\_\_

Yes NO N/A

b. Soil/Solid

3.2 Are all the PCB 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 recovery limits, if available. In-house criteria should be examined for reasonableness.

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

Note: DCB is used when PCBs are determined as Aroclors. DCB is the internal standard when determining PCB congeners and TCMX the surrogate.

3.4 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis?

ACTION: Follow surrogate criteria, Table 2.

**Table 2. Surrogate Recovery Criteria**

Criteria	Action	
	Detected Target Compounds	Non-detected Target Compounds
%R > 200%	J	Use professional judgement

	Yes	NO	N/A
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.

4.0 Laboratory Control Sample (LCS)

4.1 Are raw data and percent recoveries present for all Laboratory Control samples as required by Method 8000B (section 8.5) and Method 8082A (section 8.4.2)?

Verify that QC check samples were extracted and analyzed by the same procedures used for the actual samples.

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

NOTE: For aqueous samples, an additional QC check sample must be prepared and analyzed when any analyte in a matrix spike fails the required acceptance criteria (see section 5.3 below).

Yes NO N/A

The additional QC check sample must contain each analyte that failed in the MS analysis.

Note: When the results for matrix spike analysis indicates a problem due to sample matrix effects, the LCS results are used to verify the laboratory can perform the analysis in a clean sample.

4.2 Were Laboratory Control Samples analyzed at the required concentration as specified in Method 8000B(sec 8.5) for all analytes as specified in Table 3.

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

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 Were the LCS recoveries within the percent recoveries as specified in Table 3.

**Table 3. LCS Criteria**

Compound	% Recovery
Aroclor 1016	50-150
Aroclor 1260	50-150
Tetrachloro-m-xylene (surrogate)	30-150
decachlorobiphenyl (surrogate)	30-150

4.4 If no, were Laboratory Control Samples re-analyzed?

ACTION: If QC check samples were not re-analyzed, or a general system problem is indicated by repeated failure to meet the QC acceptance criteria specified in the method, make note in the data assessment and use Table 4 recovery actions criteria.

Yes NO N/A

**Table 4. LCS Recovery Actions**

Criteria	Action	
	Detected Associated Compounds	Non-Detected Compounds
%R > Upper Acceptance Limit	J	No qualification
%R < Lower 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 one matrix spike and matrix duplicate (unspiked) pair (MS/Dup) or matrix spike/matrix duplicate (MS/MSD) present and complete for each matrix (Method 8082A Section 8.4.1)?  \_\_\_ \_\_\_

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

5.2 Have MS/Dup or MS/MSD results been summarized on modified CLP Form III?  \_\_\_ \_\_\_

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/Dup, MS/MSD 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 (Method 8000B-39 (section 8.5)).

a. Water  \_\_\_ \_\_\_

- |               |  |
|---------------|--|
|               | <b>Yes NO N/A</b>  |
| b. Waste      | <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |
| c. Soil/Solid | <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |

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

- 5.4 Were Laboratory Control Samples analyzed for all analytes as specified in Table 5, or did the lab use the optional QC acceptance criteria i.e., in-house criteria?

List the criteria used and make note in data assessment.

Criteria used \_\_\_\_\_

**Table 5. MS/MSD Criteria**

Compound	Percent Recovery QC Limits	RPD
Aroclor 1016	29-135	0-15
Aroclor 1260	29-135	0-20

- 5.5 Was the matrix spike prepared at the proper spike concentration? (Method 8000B, section 8.5.1-8.5.2)

For aqueous organic extractable, the spike concentration should be prepared according options in: Method 8000B-40, (section 8.5.1 and 8.5.2).

- 5.6 Were the matrix spike and matrix spike duplicate recovery and RPD limits met as specified in Table 5. Note: No qualification of the data is necessary on MS and MSD data alone. Use professional judgement to use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. If any MS and MSD, percent recovery, or RPD results in the Aroclor fraction is out of specification (Table 5), qualify data to include the consideration of the existence interference in the raw 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. Use professional judgement to determine the need for qualifications of detects of non-spiked compounds.

Yes NO N/A

**Table 6. MS/MSD Actions for Analysis**

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

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on CLP equivalent 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 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

Yes NO N/A

printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for PCBs?

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 PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary.

7.2 Do any field/rinse blanks have positive PCB 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 7 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

**Table 7. Blank Contamination Criteria**

Blank Type	Blank Result	Sample Result	Action for Samples
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Yes NO N/A

	Detects	Not detected	No qualification
Method, Clean up, Instrument, Field	< 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 for calibration criteria.

Note: When applied as described in Table 7 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.

Yes NO N/A

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 capillary column used for the analysis of PCBs?

Action: Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (Method 8082, section 4.2)

8.2 Indicate the specific type of narrow bore or wide bore (.53 mm ID, fused silica GC columns, such as DB-608 and DB-1701 or equivalent).

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.

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. Samples

b. All blanks

c. Matrix spike samples

d. 5 pt. initial calibration standards

e. calibration verification standards

f. Laboratory Control samples (LCS)

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

9.2 Are data summary forms (containing calibration factors or response factors) for the initial 5

Yes NO N/A

pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence?

Note: Calibration Aroclor mixtures other than 1016/1260 may be used (as per approved project QA plan)

NOTE: If internal standard calibration procedure is used (Method 8000B-15(section 7.4.2.2)), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (Method 8000B-16 (section 7.4.2.1)), then calibration factors must be used. The internal standard approach is highly recommended for PCB congener analysis.

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.

9.3 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.

9.4 Are standard retention time (RT) windows for each PCB peak 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.

NOTE: Retention time windows for all PCBs are established using retention times from three calibration standards analyzed during the entire analytical sequence (Method 8000B, section 7.6). Best results are obtained

Yes NO N/A

using retention times which span the entire sequence; i.e., using the calibration verification/continuing calibration standards analyzed every 12 hours.

9.5 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 9.4 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.

9.6 Do all standard retention times in each level of the initial 5 pt. calibrations for PCBs 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 standard spanning the entire sequence were used to obtain 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 Method 8081B-15 (section 7.4.6).

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".

9.7 Has the linearity criteria for the initial calibration standards been satisfied for both

columns? (% RSD for the calibration factors (CFs) for the three to five major peaks of each of the Aroclor compounds must be < 20.0%). Yes NO N/A

ACTION: If no, follow Table 8 criteria.

**Table 8. Initial Calibration CF Action for Aroclor Analysis**

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
% RSD > 20%	J	UJ
% RSD within allowable limits	No qualifications	

9.8 Does the calibration verification/continuing calibration standard contain the PCB peaks of interest, analyzed on each working day, prior to sample analyses (Method 8082, sections 7.6.2)?

9.9 Has a calibration verification/continuing calibration standard been analyzed after every 10 samples and at the end of each analytical sequence (Method 8082A, section 7.6.2).

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

9.10 Has the percent difference (%D) between the Calibration Factor (CF) of each of the three to five peaks used to identify the Aroclor in the CCV and the CF from these peaks in the initial calibration exceeded ± 15%.

9.11 Has a new 5 pt. initial calibration curve been generated for those PCB analytes which failed in the calibration verification/continuing calibration standard (8000B, section 7.7.3), and all samples which followed the out-of-control

calibration verification/standard continuing calibration  
Standard? Yes NO N/A  
[ ] [ ] [ ]

ACTION: If the %D for any analyte exceeded the  $\pm 15\%$  criterion and the instrument was not recalibrated for those analytes, qualify positive results for all associated samples (those which followed the out-of-control standard) "J" and sample quantitation limits "UJ". (see Table 9)

9.12 Have retention time (RT) windows been properly calculated for each analyte of interest (Method 8000B, section 7.6), using RTs from the associated calibration verification/continuing standard? [ ] [ ] [ ]

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

9.13 Do all standard retention times for each calibration verification/continuing calibration standard fall within the windows established during the initial calibration sequence? [ ] [ ] [ ]

9.14 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows. [ ] [ ] [ ]

ACTION: For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use paragraph B below. If the answer to either 9.13 or 9.14 above is no, check the chromatograms of all samples which followed the last in-control standard. If samples were not re-analyzed, all samples analyzed after the last in-control standard must be evaluated using professional judgement.

(A) For non-detected target compounds, check to see if the sample chromatograms contain any peaks that are close to the expected RT window of the Arcolor of interest. If no peaks are present, no qualification of data is necessary. If peaks are present close th RT window of the Aroclor of interest, qualify the non-detected values as presumptively present "N".

Yes NO N/A

(B) For detected compounds in the affected samples, if peaks within the RT window, no qualification necessary. If peaks are close to the expected RT window of the Aroclor of interest, the reviewer can examine the data package for the presence of three or more standards the Aroclor 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 reevaluated using the Mean Retention Times of the standards. If the peaks in the affected sample fall within the revised window, qualify the detected target compounds "NJ". If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable "R". (Table 9)

9.15 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence sequence (closing CCV). (Table 9)

**Table 9. CCV Criteria**

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT window	Use professional judgement (Sec 9.14)	
%D not within +/- 15%	J	UJ
Time elapsed greater than section 9.15 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

9.16 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".

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?

Yes NO N/A

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.

10.3 Were the TCMX/DCB surrogate RTs for the samples within the mean surrogate RT from the initial calibration?

Action: If no, see "Action" in section 9.14 above

11.0 Extraction Techniques for Sample Preparation

Method 8082A permits a variety of extraction techniques to be used for sample preparation. Check 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

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)

Yes NO N/A

6. Supercritical fluid (Method 3562)

7. Other

11.1 Extract Cleanup - Efficiency Verification (Form IX/Equivalent)

11.1.1 Method 8082 (section 7.2) references method 3660 (sulfur) and 3665A (sulfuric acid) to use for cleaning extracts. Were one or both method used?

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

NOTE: Method 3620A, Florisil, may be used per approved project QA plan. The method does not list which analytes and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct PCB list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.2 Are all samples listed on modified CLP PCBs Florisil/Cartridge Check Form?

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

11.3 Was GPC Cleanup (method 3640A) performed?

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

11.4 Were the same PCB analytes used in calibration used to check the efficiency of the cleanup procedures?

11.5 Are percent recoveries (% R) of the PCBs and surrogate compounds used to check the efficiency of the cleanup procedures within lab's in-house QC limits (use 70-130% if not available).

Yes NO N/A

70-130% for GPC calibration?

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

ACTION: If % R are < 70%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for PCBs. Use professional judgement to qualify positive results if recoveries are greater than the upper limit.

12.0 PCB Identification

12.1 Has CLP Form X or equivalent, showing retention time data for positive results on the two GC columns, been completed for every sample in which a PCB 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 cleanup verification forms)?

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

12.3 Are retention times (RT) of sample compounds within the established RT windows for both columns/analyses?

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.

Yes NO N/A

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently.

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 provided when sample concentration was sufficient (> 10 ug/ml) in the final extract?

ACTION: Indicate with red pencil which Form I results were confirmed by GC/MS and also note in data assessment. GC/MS confirmation is an option, see section 7.10 of Method 8082A-20. If GC/MS confirmation is not available, follow action in section 3.2.

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

NOTE: The method requires quantitation from one column. The second column is to confirm the presence of an analyte. 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.

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

% Difference

Qualifier

Yes NO N/A

0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected)	"NJ"
>50% (PCBs value is <CRQL)	"U"
>200%	"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 PCBs 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 PCB 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

**Yes NO N/A**

to determine the proper identification of  
field duplicates.