State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Polychlorinated Biphenyls by Method 8082, SW-846

Version 3.0

May 2024

Written by the Connecticut DEEP QA/QC Workgroup

Revision	Comments	Date
1.0	First version for publication	7/11/05
2.0	Final version based upon public comments	July 2006
3.0	Updates to reflect CAM method updates to improve consistency between different states.	May 2024

Table of Contents

Table of Tables

Acronym List

ACRONYM	DEFINITION
BZ198	Ballschmiter and Zell (for congeners)
CASN	Chemical Abstracts Service Number
CCV	Continuing calibration verification
%D	Percent difference or percent drift
DBOFB	4,4'-Dibromooctafluorobiphenyl
DCB	Decachlorobiphenyl
DDD	Dichlorodipheyldichloroethane
DDE	Dichlorodiphenylethane
DDT	Dichlorodiphenyltrichloroethane
DEEP	CT Department of Energy and Environmental Protection
ECD	Electron capture detector
ELCD	Electrolytic conductivity detector
GC	Gas chromatograph
ICV	Initial calibration verification
LCS	Laboratory control sample
LLOQ	Lower limit of quantitation
MS	Matrix spike
MSD	Matrix spike duplicate
MSE	Microscale Solvent Extraction
NA	Not applicable
PCB	Polychlorinated biphenyl
PFE	Pressurized Fluid Extraction
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QC	Quality control
r/r^2	Correlation coefficient/determination
RCP	Reasonable Confidence Protocol
RL	Reporting limit
RPD	Relative percent difference
%RSD	Percent relative standard deviation
RSR/RSRs	Remediation Standard Regulations
SPE	Solid Phase Extraction
TCMX	Tetrachloro-m-xylene
µg/kg	micrograms per kilogram
µg/L	micrograms per liter

1.0 Quality Assurance and Quality Control Requirements for SW-846 Method 8082

1.1 Method Overview

SW-846 Method 8082 is a gas chromatography ("GC") procedure used to determine polychlorinated biphenyls ("PCBs"), as Aroclors or as individual congeners, in a variety of matrices including waters, soils, sediments, wastes, etc. This procedure requires an experienced GC analyst familiar with the Quality Assurance and Quality Control ("QA/QC") requirements of the method. The sample introduction procedure requires the use of a solvent extraction procedure (see Table 1.0).

Open-tubular, capillary columns are employed with electron capture detectors ("ECD") or electrolytic conductivity detectors ("ELCD"). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target analytes may be determined with a dual-column chromatographic system. The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

All method references are to the latest promulgated version of the method found in *Test Methods for Evaluating Solid Waste, SW-846*.

1.2 Summary of SW-846 Method 8082

1.2.1 Sample Extraction and Cleanup

Samples for analysis by SW-846 Method 8082 require extraction by one of the following methods (see Table 1.0). The use of a hydrophilic solvent mixture, either 1:1 Acetone/Hexane or 1:1 Acetone/Methylene chloride, is recommended for soil and sediment samples.

Table 1.0: Extraction Methods

SW-846 Method	Matrix	Description
3542	Air Samples	Extraction of Analytes Collected Using a Modified Method 5 Sampling Train
3510	Aqueous	Separatory Funnel Liquid-Liquid Extraction
3520	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3535	Aqueous	Solid-Phase Extraction ("SPE")
3540	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545	Soil/Sediment	Pressurized Fluid Extraction ("PFE")
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction ("MSE")
3550	Contaminated Solids [1]	Ultrasonic Extraction
3580	NAPL	Solvent Dilution
[1] Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed		

It is highly recommended that extracts for PCB analysis be routinely subjected to a sulfuric acid cleanup using SW-846 Method 3665. This cleanup technique will remove (i.e., destroy) most other organic compounds including many single component organochlorine or organophosphorus pesticides as well as phthalate compounds, which could potentially interfere with the quantitation of PCB Aroclors or congeners. Other optional extraction cleanup methods are included in Table 2.0.

Table 2.0: Optional Extraction Cleanup Methods

SW-846 Method	Description
3600	General Cleanup Selection
3610	Alumina
3620	Florisil
3630	Silica Gel
3640	Gel Permeation Chromatography
3660	Sulfur Cleanup

1.2.2 GC Analysis

The PCBs are extracted from the sample using the appropriate method. The solvent extract is concentrated in hexane or other appropriate solvent. The extract is then subjected to the sulfuric acid cleanup. This cleanup will destroy many single response pesticides and therefore this procedure cannot be used to determine other pesticide compounds. Aliquots of the cleaned-up extract are injected onto the GC column in the gas chromatograph. The GC oven is temperature programmed to facilitate separation of the analytes that are then detected by an ECD or ELCD interfaced to the column.

Preliminary identification of target analytes is accomplished by comparing the retention time of the chromatographic peaks of the sample to known PCBs analyzed under the exact same conditions. Confirmation is accomplished either by analysis of the same extract on a dissimilar column, again comparing the retention times of the chromatographic peaks of the sample to known PCBs analyzed under the exact same conditions, or by using at least one other independent qualitative technique such as GC/MS. Quantitation is accomplished by constructing a minimum 5-point calibration curve of PCB concentration vs. peak area. Identification of PCBs on a single column must be confirmed on a second column or must be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput. Dual column confirmation is not required in the case where PCBs are not detected above their specific reporting limit.

The chromatographic data produced may then be used to identify and quantify the nine (9) Aroclors listed in Table 1B, individual PCB congeners, or to determine total PCBs as the cumulative sum of the individual Aroclors or congeners.

1.3 Method Interferences

Refer to SW-846 Methods 3500, 3600, and 8000 for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

- Contaminated solvents, reagents, or sample processing hardware;
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces;
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response; and
- Co-elution of target analytes

An in-depth discussion of the causes and corrective actions for all these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

1.3.1 Chemical Contaminants

Major contaminant sources for SW-846 Method 8082 include, but are not limited to, plastics, impurities in laboratory chemicals, contaminated laboratory ware, etc. The use of non-polytetrafluoroethylene (“PTFE”) thread

sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials may contaminate the analytical system.

Analysis of blanks provides information about the presence of contaminants. When potential interfering peaks or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the contamination and eliminate it. **Subtracting blank values from sample results is not permitted.** Any method blank exceedances should be fully documented in the laboratory report narrative.

Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations by SW-846 Method 8082. Common flexible plastics contain varying amounts of phthalate esters, as plasticizers, which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination. These materials may be removed prior to analysis using SW-846 Method 3665 (Sulfuric Acid/Permanganate Cleanup).

1.3.2 Cross-contamination/ Carryover

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of PCBs or other compounds which cause a detector response, such as phthalates. Syringes on the autosampler may also become contaminated in the same manner. Concentrations of chlorinated pesticides which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. Low-level samples that immediately follow high-level samples need to be inspected for possible carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis.

1.3.3 Sulfur Interferences

The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting PCB Aroclors. Sulfur contamination should be expected with sediment samples and can be removed using SW-846 Method 3660. This cleanup technique will remove (destroy) most other organic compounds including many single component organochlorine or organophosphorus pesticides as well as phthalate contaminants which could potentially interfere with the quantitation of PCB Aroclors or congeners.

1.3.4 Co-elution

As described in SW-846 Method 8082, co-elution among the many target analytes or other compounds can cause interference problems. The GC analyst should experiment with varying chromatographic conditions to obtain the most efficient compound separation.

1.3.5 Special Precautions

Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized at laboratory drying oven temperatures and spread to other glassware. Due caution should be exercised when drying glassware used for the analysis of samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

1.4 Quality Control Requirements for SW-846 Method 8082

1.4.1 Reporting Limits/Lower Limits of Quantitation for Method 8082

The reporting limit ("RL"), or lower limit of quantitation ("LLOQ"), for a compound is dependent on the concentration of the lowest non-zero standard in the initial calibration, detector type, sample weight/volume, extraction procedure, and moisture content. Table 3.0 lists approximate RL/LLOQs for various matrices utilizing a gas chromatograph with an electron capture detector ("GC/ECD"). ELCDs will have slightly higher RL/LLOQs. Solid matrices in this table assume 100% solids.

Table 3.0: Typical Reporting Limits / Lower Limits of Quantitation[1]

Matrix	Typical Reporting Limit
Water	0.25 to 1.0 µg/L
Soil	50 to 100 µg/Kg
[1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.	

Moisture content of soils and sediments will raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RL/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet the project Data Quality Objectives ("DQOs"). To meet the RLs/LLOQs applicable to project DQOs, it may be necessary to modify the analytical method by using increased sample volume or mass. In such cases the modifications must be noted in the laboratory report narrative.

1.4.2 General Quality Control Requirements

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC instrumentation as a quantitative tool and skilled in the interpretation of chromatograms for PCBs.

Refer to SW-846 Method 8000 for general QC requirements for all chromatographic methods, including SW-846 Method 8082. These requirements ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all chromatographic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the GC system operation may be found in SW-846 Method 8000. Instrument QC and method performance requirements for the GC system may be found in SW-846 Method 8082.

The minimum requirements for a formal QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and LCS duplicates ("LCSD") and matrix spike duplicates ("MSD") to assess precision. The use of site-specific MS/MSD's is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Field, rinsate, or other blanks should not be used for MS/MSD's.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative method being used. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform this method. These data must meet or fall within the performance standards as presented in Section 1.4 and Table 1A of this RCP. See SW-846 Method 8000 for the procedure. The IDOC must include the following elements provided in Table 4.0:

Table 4.0: IDOC Requirements

QC Element	Performance Criteria

Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Average Recovery	Table 1A
% Relative Standard Deviation	Table 1A
Surrogate Recovery	Table 1A
Internal Standards (if employed)	Table 1A

Because of the extensive analyte list and number of QC elements associated with the IDOC, it should be expected that one or more analytes may not meet the performance standards for one or more QC elements. The laboratory should make every effort to find and correct the problem and repeat the analysis. All non-conforming analytes along with the laboratory acceptance criteria should be noted in the IDOC data. This information should be kept on-file at the laboratory.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and relative percent difference (“RPD”) limits, and surrogate recovery limits. These limits must be equal to or fall within the limits specified in Table 1A.

1.4.3 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8082

Specific QA/QC requirements and performance standards for SW-846 Method 8082 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with “Reasonable Confidence” regarding the usability of analytical data to support environmental decisions. The concept of "Reasonable Confidence" is explained on the CT Department of Energy and Environmental Protection (“DEEP”) website.

While optional, parties electing to utilize these protocols will be assured that “Reasonable Confidence” data, will be generally accepted by agency reviewers. To achieve “Reasonable Confidence” parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming analytical performance standards; and

3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

Table 1A: Specific QA/QC Requirements and Performance Standards for Method 8082

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Capability ("IDOC")	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (4) Must follow procedure in SW-846 8000.	No	Refer to SW-846 8000 and Section 1.4.2 of this protocol.	N/A
Retention Windows	Laboratory Analytical Accuracy	(1) Prior to or during the initial calibration when a new GC column is installed. (2) Calculate according to Method 8000. (3) If acid clean-up is not performed, also analyze DDT/DDE/DDD standard.	No	(1) For PCB Aroclor analysis, if interference is present for any of the Aroclor peaks used for quantitation with DDT, DDE, or DDD, either adjust GC conditions to obtain better resolution or choose another peak for the Aroclor of interest that does not coelute with DDT, DDE, or DDD. (2) For PCB congener analysis, if interference is present for any of the target congeners with DDT, DDE, or DDD, adjust GC conditions to obtain better resolution.	N/A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed with dual columns at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) *PCB Aroclors*: 5-point calibration for other Aroclors may be warranted based on site-specific conditions (i.e., if nature of PCB contamination known). A minimum of 5 unique peaks must be evaluated for Aroclors 1016 and 1260. *Congeners*: 5-point calibration must include all target PCB congeners. (4) Low standard must be ≤RL/LLOQ (5) % RSD≤20, r ≥0.99 (linear regression) or, r^2≥0.99 (non-linear regression) for each PCB Aroclor or each PCB congener. (6) % RSD >20, linear or non-linear regression must be used. (7) *PCB Aroclors:* For Aroclors which are not calibrated with 5-points, lab must perform single analysis of these Aroclors at the midpoint of the calibration curve with 12-hrs of sample analysis. (8) Calibration must be performed under the same conditions as the sample. (9) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (10) If curves are used, curve MUST NOT be forced through origin. (11) Curves must be verified with independent ICV prior to sample analysis.	No	(1) Required recalibration as by method. (2) If recalculated concentrations from lowest calibration standard are outside of 70-130% recovery range, either: The RL/LLOQ must be reported as an estimated value; OR The RL/LLOQ must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD>20, r <0.99, or r^2 <0.99) in laboratory report narrative. If non-linear regression (e.g., quadratic equation) is used for calibration, this must be noted in the laboratory report narrative along with the congeners or PCB Aroclors affected.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification ("ICV")	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain Aroclors 1016/1260 for PCB Aroclor analysis and all target congeners for PCB congener analysis. (5) Percent recoveries must be between 80-120% for each PCB Aroclor or congener.	No	Locate source of problem; recalibrate if either PCB Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria.	If recovery is outside of 80-120% for any PCB Aroclor or congener, report non-conformances in laboratory report narrative.
Continuing Calibration Verification ("CCV")	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every ≤20 field samples, whichever is more frequent, and at the end of the analytical sequence. NOTE: If internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required. (2) Concentration level near midpoint of curve using AR-1016/1260. *Congeners:* CCV must include all congeners. (3) *PCB Aroclors*: Must contain Aroclors 1016/1260. Aroclors other than 1016/1260 must be verified with a one-point standard within 12 hours of being detected in a sample. C*ongeners*: Must include all target PCB congeners. (4) %D must be ≤20 for each PCB Aroclor or PCB congener. (5) Verify that all analytes fall within retention time windows. (6) Area count of internal standard in continuing calibration must be within ±50% of the average area count in the associated initial calibration.	No	(1) Perform instrument maintenance, reanalyze CCV and/or recalibrate. (2) Reanalyze associated samples if beginning or closing CCV exhibited low response and associated aroclor/congeners not detected in samples. (3) Reanalyze associated samples if beginning or closing CCV high and associated aroclor/congners were detected in samples. NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conformances in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Method Blank ("MB")	Laboratory Method Sensitivity & Contamination Evaluation	(1) Extracted with every batch or every ≤20 field samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL/LLOQ.	Yes	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory report narrative. (2) If contamination of method blanks is suspected or present, the lab, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the lab may report results of re-extraction only. (4) If re-extraction of performed outside of holding time, the lab must report results of both the initial extraction and re-extraction.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample ("LCS")	Laboratory Method Accuracy	(1) Extracted with every batch or every ≤20 field samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors*: 1016/1260 required. Optionally, LCSs may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples). *Congeners*: Must include all target PCB congeners. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol). (7) Standard source different from initial calibration source.	Yes	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of criteria. (2) If ≤10% of PCB congeners are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the acceptance criteria (>140%), re-extraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples. (4) If MS/MSD in same batch compare to determine if problem isolated to LCS.	(1) If sample re-extraction is not possible, report nonconformance in laboratory report narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener, report non-conforming compounds in laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the lab must report results of both the initial extraction and re-extraction.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
LCS Duplicate ("LCSD")	Laboratory Method Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) *PCB Aroclors*: 1016/1260 required. Optionally, LCS Duplicates may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples.). *Congeners*: Must include all target PCB congeners. (4) Matrix specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) RPDs must be ≤20% for waters and ≤30% for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if either Aroclor 1016/1260 or >10% of all PCB congeners are outside of recovery acceptance criteria. (2) If ≤10% of PCB congeners are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of PCB congeners or either Aroclor 1016/1260 are above the recovery acceptance criteria (>140%), re-extraction is not required if the affected congeners or all PCB Aroclors were not detected in associated samples.	(1) If same re-extraction is not possible, report non-conformance laboratory report narrative. (2) If recovery is outside of 40-140% for any PCB Aroclor or congener or if RPD is outside of criteria, report non-conforming compounds in laboratory report narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the lab must report results of both the initial extraction and re-extraction.
Matrix Spike/Matrix Spike Duplicate ("MS/MSD") (Site Specific)	Method Accuracy & Precision in Sample Matrix	(1) Every ≤20 field samples per matrix (at discretion of lab or at request of data user). (2) Matrix specific (e.g., aqueous, soil). (3) Concentration level near midpoint of curve. (4) *PCB Aroclors*: 1016/1260 required. Optionally, MS/MSD may be spiked with other Aroclors which have been fully calibrated, based on site-specific conditions (i.e., if specific Aroclors are known to be present or expected in samples). *Congeners*: Must include all target PCB congeners. (5) Percent recoveries between 40-140%. (6) RPDs ≤20% for waters and ≤30% for solids (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol). (8) Field blanks, trip blanks, etc. cannot be used for MS/MSDs.	Yes ONLY when requested by data user	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note non-conformances in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 2 surrogates, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: *PCB Aroclor Analysis*: TCMX and DCB. *PCB Congener Analysis*: TCMX or DBOFB and BZ198. (2) Percent recoveries must be between 30-150% for both surrogates on both columns.	Yes (report surrogate recoveries from both columns)	(1) If the same surrogate is outside limits on both columns: (a) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (b) Re-extract the sample if surrogate recoveries are high and PCB Aroclor or PCB congeners were detected in the sample. (2) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (3) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), re-analyze sample on dilution. (4) If a surrogate is diluted to a concentration below that of the lowest calibration standard, re-extraction and/or reanalysis is not required.	(1) Report recoveries outside of 30-150% in laboratory report narrative. (2) If re-extraction yields similar surrogate non-conformances, the lab must report results of both the initial extraction and re-extraction. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the lab may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the lab must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the lab must provide the chromatogram in the data report.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Internal Standards (if employed)	Laboratory Analytical Accuracy & Method Accuracy in Sample Matrix	(1) Minimum of 1. (2) Area counts in samples must be between 50-200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	If internal standard is outside of limits, reanalyze sample unless chromatographic interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample dilution.	(1) Report non-conformances in laboratory report narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the lab must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the lab may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the lab must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the lab must provide the chromatogram in the data report.

Identification & Quantitation	Inter-laboratory Consistency	(1) Peak area is the expected default to be used for quantitation of PCB Aroclors and congeners under most circumstances. Regardless if peak area or peak height is used, the same method for quantitation of sample must also be used for calibration standards. (2) *PCB Arolcors*: the lab must quantitate all Aroclors with the same five peaks used for calibration. If interference exists with select peaks, these peaks do not have to be included in the quantitation of the Aroclor; however, a minimum of three peaks is required. All peaks must be ≥25% of the height of the largest PCB Aroclor peak**.** At least one peak must be unique to the PCB Aroclor. (3) *PCB Congeners*: The lab must use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each PCB congener. *PCB Aroclors*: Laboratory should use the average calibration factor, linear or non-linear regression curve for each of three to five peaks from each concentration level to quantitate Aroclors 1016/1260. Lab should use the average calibration factor for each of three to five peaks from single point standard to quantitate remaining Aroclors (when only single-point standard analyzed). If 5-point calibration is performed for other Aroclors, follow procedure for 1016/1260. Calculate concentration of Aroclor using each individual peak and calculate the average concentration of the three to five results to obtain the final Aroclor concentration. (4) Secondary column analysis: lab must utilize a second dissimilar column to confirm positive results above the RL/LLOQ. The lab must report the higher of the two results. All required QA/QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (5) Do no report concentrations below the RL/LLOQ.	No	If the RPD between the dual column results is >40%: (a) determine potential interference; (b) re-analyze sample on dilution; or (c) additional sample cleanup techniques may be warranted; or (d) re-extract sample and re-analyze.	When the RPD between the dual column results is: (1) <40% and there is no obvious matrix interference, the higher value shall be reported. (2) <40% and there is obvious matrix interference, the lower value shall be reported and the results shall be flagged with a “P”. (3) >40% and there is no obvious matrix interference, the higher value shall be reported and the results shall be flagged with a “P”. (4) >40% and there is obvious matrix interference, the lower value shall be reported and the results shall be flagged with a “P”. All non-conformances must be noted in the laboratory report narrative. If avg. CF or RF or linear regression not used (e.g. quadratic equation), must note list of affected compounds in laboratory report narrative.
General Reporting	N/A	(1) The laboratory must only report values ≥ the sample specific reporting limit.	N/A	N/A	(1) The performance of dilutions must be

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
		(2) Dilutions: If diluted and undiluted analyses are performed, the lab should report results for lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc. for each analysis must be reported. NOTE: Lab shall not perform dilutions on samples due to sulfur interference. Labs must employ a clean-up technique to reduce the presence of sulfur interference. It is highly recommended that acid clean-up be performed on all sample extracts prior to analysis. (3) Results for soils/sediments must be reported on a dry-weight basis.			documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory report narrative. (2) Complete analytical documentation for diluted and undiluted analyses must documented in laboratory report narrative and be maintained in laboratory records. (3) If PCB Aroclors are not detected but chromatogram shows evidence of weathered Aroclors or potential presence of PCBs, this must be noted in the laboratory report narrative and a copy of the chromatogram must be provided in the data report. (4) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report narrative. (5) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory report narrative.

1.5 Special Analytical Considerations for Multi-Response Analytes

The following bullets highlight potential issues that may be encountered with the analysis of PCBs using this protocol.

- The identification of multi-component PCB Aroclors is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. If, based on site history, specific PCB Aroclors are contaminants of concern, it is the responsibility of the data user to request that these specific PCB Aroclor spikes be included in the LCSs and MS/MSDs. All PCB Aroclors are not routinely included in LCSs or MS/MSDs.

- A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation ("%RSD") requirements specified in Table 1A.

1.6 Analyte List for SW-846 Method 8082

The DEEP analyte list for SW-846 Method 8082 is presented in Table 1B. The compounds listed are readily determined by Method 8082.

Table 1B: Analyte List For SW-846 Method 8082

Analyte	CAS Number
Aroclor-1016	12674112
Aroclor-1221	11104282
Aroclor-1232	11141165
Aroclor-1242	53469219
Aroclor-1248	12672296
Aroclor-1254	11097691
Aroclor-1260	11096825
Aroclor-1262	37324235
Aroclor-1268	11100144
[1]Aroclors 1262 and 1268 are not normally on the list of PCBs. If the chromatograms indicate these aroclors are present the laboratory is required to quantitate and report the compounds.	

1.6.1 Additional Reporting Requirements for SW-846 Method 8082

While it is not necessary to request and report all the analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEEP strongly recommends that full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.7 Routine Reporting Deliverables for Method 8082

The following table (Table 5.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report all items in special circumstances.

Table 5.0: Report Deliverables

Parameter	Deliverable	Comments
Retention Time Windows	NO	
Initial Calibration	NO	Note non-conformances in laboratory report narrative
Continuing Calibration	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive sample results above RL/LLOQ with “B” flag.
Lab Control Sample / Lab Control Sample Duplicate	YES	Note non-conformances in laboratory report narrative
Site Specific Matrix Spike/ Matrix Spike Duplicate	YES (If analyzed)	Note non-conformances in laboratory report narrative
Internal Standards (if used)	NO	Note non-conformances in laboratory report narrative
Identification and Quantitation	NO	Note non-conformances in laboratory report narrative
Surrogate Recoveries	YES	Note non-conformances in laboratory report narrative
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory.

1.7.1 Reporting and Flagging of Results

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as “ND” (Not Detected at specified RL/LLOQ). The RL/LLOQ for each compound in each sample must be listed on the report, based upon the lowest calibration standard, the exact sample mass, any dilution factors, percent moisture, etc.

- Compounds detected above the RL/LLOQ in blanks and found in samples, also above the reporting limit, shall be flagged with a “B” suffix (e.g., 25B).

- When the results from dual columns have a RPD >40%, the results shall be qualified with a “P” flag as described in Table 1A, see identification and quantitation entry.

- All soil/sediment results shall be reported on a dry weight basis.

1.8 Sample Containers, Preservation, and Holding Times

Table 6.0 identifies the type of containers, preservation requirements, and holding times dependent upon analyte and matrix.

Table 6.0: Sample Containers, Preservation, and Holding Times

Matrix	Container[1]	Preservative[2]	Holding Time
Aqueous with no chlorine present	1-liter amber glass bottle with Teflon line cap.	Store at 4 ± 2º C, but not frozen.	1 year to extraction. 40 days from extraction to analysis.
Aqueous with chlorine present	1-liter amber glass bottle with Teflon line cap.	Neutralize chlorine with either 25 mg ascorbic acid or 3 mg sodium thiosulfate. Store at 4 ± 2º C, but not frozen.	1 year to extraction. 40 days from extraction to analysis.
Soil/Sediment samples.	250 mL amber glass jar with Teflon lined cap.	Cool to 4 ± 2º C.	1 year to extraction. 40 days from extraction to analysis.
High Concentration Waste Samples Excluding transformer oils.	Collect in amber glass jar with Teflon lined cap.	No special preservation requirement.	1 year to extraction. 40 days from extraction to analysis.
Transformer / Waste Oils	Collect in glass jar with Teflon lined cap.	No special preservation requirement.	1 year

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6ºC are acceptable.