State Of Connecticut
Department of Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Polychlorinated Biphenyls by Method 8082, SW-846

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Written by the Connecticut DEP QA/QC Workgroup

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1.0 QA/QC Requirements for Method 8082

1.1 Method Overview

Method 8082 is gas chromatography procedure used to determine polychlorinated biphenyls (PCB's), 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 QA/QC requirements of the method. The sample introduction procedure requires the use of a solvent extraction procedure. All method references are to the latest promulgated version of the method found in Test Methods for Evaluating Solid Waste, SW-846.

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 either a single- or 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.

1.1.1 Reporting Limits for Method 8082

The reporting limit (RL) for a compound is dependent on the concentration of the lowest standard in the initial calibration, detector type, sample weight/volume, extraction procedure, and moisture content. The following table lists approximate reporting limits for various matrices utilizing a gas chromatograph with an electron capture detector (GC/ECD). Electrolytic conductivity detectors will have slightly higher reporting limits. Solid matrices in this table assume 100% solids.

Table 1.0 Typical Reporting Limits

Matrix	Typical Reporting Limit
Water	0.5 to 1.0 ug/L
Soil	50 to 70 ug/Kg

Moisture content of soils and sediments will raise the RL, 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 RLs to be raised.

Sample container type, preservation requirements, and holding times for waters, soils, and sediments are presented in Table 2A of this document.

1.1.2 General Quality Control Requirements

Each laboratory is required to operate a formal quality assurance program and be certified by the Connecticut Department of Public Health for the analysis performed. The minimum requirements include initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples (LCS) to assess precision and accuracy. The use of site specific matrix spikes and matrix spike duplicates is highly recommended. Evaluation of sample matrix effects on compound recovery is key to making good decisions.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Section 1.5 and Table 1A. See Section 8.4 of Method 8000 in SW-846 for the procedure. The Initial Demonstration of Proficiency must include the following elements:

Table 1.1 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	Table 1A

Note: Because of the extensive analyte list and number of QC elements associated with the Initial Demonstration of Proficiency, 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 Initial Demonstration of Proficiency data.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and

precision (RPD) limits, and surrogate recovery limits. These limits must meet or exceed the limits specified in Table 1A.

1.2 Summary of Method 8082

1.2.1 Sample Extraction and Cleanup

Samples for analysis by Method 8082 require extraction by one of the following methods. 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.

SW-846 Method	Matrix	Description
3542	Air Samples	Extraction of Analytes Collected Using a Modified Method 5 Sampling Train
3510C	Aqueous	Separatory Funnel liquid-Liquid Extraction
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction (PFE)
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids [1]	Ultrasonic Extraction
3580A	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

All soil extracts must be cleaned up, using SW-846 Method 3665 Sulfuric Acid/Permanganate Cleanup prior to analysis. Other cleanup methods listed below may also be employed if necessary.

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 PCB's 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 gas chromatograph (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 PCB's 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 PCB's 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 calibration curve of PCB concentration vs. peak area. Confirmation is not required in the case where PCB's 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 PCB's as the cumulative sum of the individual Aroclors or congeners.

1.3 Method Interferences

Refer to SW-846 Methods 3500 (Sec. 3.0, in particular), 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 of 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 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 Method 3665 Sulfuric Acid/Permanganate Cleanup.

1.3.2 Cross-contamination/ Carryover

Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of PCB's or other compounds which cause a detector response, such as phthalates. Syringes on the autosampler may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of solvent blanks. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run (ghost peaks).

1.3.3 Sulfur Interferences

The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting chlorinated pesticides. Sulfur contamination should be expected with sediment samples and can be removed through the use of SW-846 Method 3660.

1.3.4 Co-elution

As described in Section 3.8 and 3.9 of 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 General Quality Control Requirements for Determinative Chromatography Methods

Refer to SW-846 Method 8000 for general quality control requirements for all chromatographic methods, including SW-846 Method 8082. These requirements insure that each laboratory maintain a formal quality assurance program and records to document the quality of all chromatographic data. Quality Control procedures necessary to evaluate the GC system operation may be found in SW-846 Method 8000, Section 7.0, and include evaluation of retention time windows, initial and verification of instrument calibrations and chromatographic performance of sample analyses. Instrument quality

control and method performance requirements for the GC system may be found in SW-846 Method 8082, Sections 8.0 and 9.0, respectively.

1.4.2 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 environmental professional (EP) with "Reasonable Confidence" regarding the usability of analytical data to support DEP decisions.

While optional, parties electing to utilize these protocols will be assured that "Reasonable Confidence" data, will be generally accepted by agency reviewers. In order 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, as necessary, compliance with performance standards prescribed in Table 1A for this test method; and

3. Adopt the reporting formats and elements specified in Section 1.7 of this method.

1.4.3 Site Specific Matrix Spike (MS), Matrix Spike Duplicate (MSD) Samples

It is strongly recommended that site specific MS/MSD samples be analyzed from each site, and each matrix type sampled. Percent recovery data from site specific samples allow the 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 and are in general discouraged. Batch MS/MSD's should not be reported under the RCP's. Additionally trip blanks, field blanks, rinsate blanks, etc. should not be used for MS/MSD's.

1.4.4 Special Analytical Considerations for Multi-Response Analytes

The identification of multi-component mixtures, such as PCB's, 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's are contaminants of concern; it is the responsibility of the EP to request that these site specific PCB's are included in the MS/MSD's.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method 8082*

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Retention Time Windows	Accurate identification of Pesticides	1) Prior to or during the initial calibration when a new column is installed. 2) Calculate according to Method 8000, Section 7.6.	NO	N/A	N/A
Initial Calibration	Laboratory Analytical Accuracy	1) Minimum of 5 stds (Note 1) 2) Low std at or below reporting limit 3) % RSD must be ≤20% or if linear regression used "r" ≥ 0.990 4) 5-point cal for AR-1016/1260. Single point for other Aroclors at mid-point within 12-hrs of sample analysis. If congeners are determined, must use 5-point for each congener. 5) If curves are used, curve must NOT be forced through origin. 6) Curves must be verified with independent ICV prior to sample analysis.	NO	Recalibrate as required by the method.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in narrative. If avg CF or linear regression not used (e.g. quadratic equation), must note list of affected compounds in narrative
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	1) Prior to samples, every 12-hours or 20 samples, whichever is more frequent, and at the end of the analytical sequence. 2) Concentration near mid-point of curve using AR-1016/1260. Congeners; CCAL must include all congeners 3) Percent difference or drift ≤15%. 4) Verify all analytes fall in retention time windows.	NO	1) Perform instrument maintenance, reanalyze CCAL and/or recalibrate. 2) Reanalyze associated samples if beginning or closing CCAL exhibited low response and associated pesticides not detected in samples. 3) Reanalyze associated samples if beginning or closing CCAL high and associated pesticides were detected in samples.	Report exceedances in narrative. Note: Associated samples means all samples analyzed since the last acceptable CCAL.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method 8082* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Contamination Evaluation	1) Extracted every 20 samples or every batch, whichever is greater. 2) Matrix specific 3) Target analytes must be <RL	YES	Locate source of contamination and correct problem. Reanalyze method blank. Re-extract samples if method blank contaminated	1) Report non-conformances in case narrative. 2) All results for compounds present in method blank above RL must be "B" flagged if detected in samples associated with the method blank. 3) If re-extraction performed within holding time, report only compliant data. If re-extraction performed outside holding time report all data.
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	1) Every 20 samples or each batch, whichever is more frequent. 2) Standard source different from initial calibration source. 3) Concentration level must be near or at the mid-point of the initial calibration. 4) LCS with AR-1016/1260. Congeners must contain all target congeners. 5) Matrix specific. 6) Laboratory determined percent recovery limits must be between 40-140%. 7) Labs are required to develop own in-house limits that meet or exceed limits listed above.	YES	Recalculate the percent recoveries Reanalyze the LCS If MS/MSD in same batch compare to determine if problem isolated to LCS Re-extract LCS and samples if >10% compounds outside acceptance criteria and no MS/MSD with acceptable criteria Locate & correct problem, reanalyze associated samples	1) Report non-conformances in case narrative. 2) If re-extraction performed within holding time, report only compliant data. If re-extraction performed outside holding time report all data.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method 8082* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Site Specific Matrix Spike/Matrix Spike Duplicate	Precision and Accuracy in Sample Matrix	1) Every 20 samples per matrix* 2) Spike concentration in lower part of calibration curve. 3) Usually contain AR-1016/1260 unless site specific Aroclor requested. 4) Laboratory determined percent recovery limits for AR-1016/1260 must be between 40-140%. Use 40-140% for other Aroclors. Congeners must contain all target congeners. 5) RPD's ≤ 50% for Aroclors, ≤ 30% for congeners.	Yes* (*If requested by EP)	If compounds out compare to LCS; if LCS recoveries in note in narrative; if LCS compounds out note in narrative probable lab error	Note outliers in narrative
Surrogates	Accuracy in Sample Matrix	1) Minimum 2 compounds across retention times of GC run. Recommended compounds Tetrachloro-m-xylene and decachlorobiphenyl. 2) Recovery limits lab generated and within 30-150% for both compounds on both columns. 3) Labs must develop own in-house limits that fall within 30-150% limits.	Yes	If the same surrogate outside limits on both columns, re-extract sample. If both surrogates outside limits on one column only, reanalyze sample. If surrogate diluted out below lowest calibration std, no recovery criteria.	1) Note exceedances in narrative. 2) If re-extraction or reanalysis confirms matrix interference or if re-extraction outside holding times report all results. 3) If re-extraction or reanalysis results in criteria and in holding time, report only compliant data.

TABLE 1A Specific QA/QC Requirements and Performance Standards for Method 8082* (continued)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Identification and Quantitation	Inter-laboratory Consistency	1) Laboratory must use a minimum of 3 peaks. Peaks selected must be ≥25% of height of largest aroclor peak. 2) Aroclors: Laboratory should use the average calibration factor for each of the peaks from each concentration level to quantitate Aroclors 1016 and 1260. Laboratory should use the average calibration factor for each of the peaks from single point standard to quantitate remaining Aroclors (when only single-point standard analyzed). If 5-point calibration performed for other Aroclors, follow procedure for 1016 and 1260. Calculate concentration of Aroclor using each individual peak and calculate the average concentration of the three results to obtain the final Aroclor concentration. Congeners: Laboratory should use the average response factor of each congener. 3) Second column analysis: Laboratory must utilize a second dissimilar column to confirm all positive results above the RL. Report the higher of the two analyses. The QA/QC parameters in this document must be met for both columns	NO	N/A	1) If the RPD between the results for the two columns exceeds 40%, the laboratory must flag the results with a "P" suffix and note in narrative. 2) If avg Rf or linear regression not used (e.g. quadratic equation), must note list of affected compounds in narrative. Note: If a high RPD between the two columns can be definitely attributed to a matrix interference, report the lower value and note in the narrative with an explanation.

Notes for Table 1A:

* Refers to latest promulgated version of SW-846 Method 8082.
GC/MS = Gas Chromatography/Mass Spectrometry
CCC = Calibration Check Compound
%RSD = Relative Percent Standard Deviation
EP = Environmental Professional
r = Correlation Coefficient
RPD = Relative Percent Difference
N/A = Not Applicable
CF = Calibration Factor

Note 1: Six standards are required for a quadratic equation calibration curve, and seven are required for a polynomial fit. In either case the correlation coefficient must be ≥ 0.990.

1.5 Analyte List for SW-846 Method 8082

The Connecticut DEP (DEP) analyte list for SW-846 Method 8082 is presented in Table 1B. The compounds listed are readily determined by Method 8082. Most of the compounds listed have Connecticut Remediation Standard Criteria or are listed in the Approved Criteria for Additional Polluting Substances.

1.5.1 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. DEP 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.

The Reporting Limit (RL) is based upon the lowest standard in the initial calibration. It is the responsibility of the EP to specify to the laboratory the detection limits required for the samples. In order to meet the detection limits 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 narrative.

1.6 Routine Reporting Deliverables for Method 8082

The following table (Table 1.2) 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.

1.6.1 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 reporting limit). The reporting limit for each compound in each

sample must be listed on the report and take into account the exact sample mass, any dilution factors, percent moisture, etc.

Compounds detected above the reporting limit in blanks and found in samples, also above the reporting limit, shall be flagged with a "B" suffix (e.g. 25B).

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

Table 1.2 Report Deliverables

PARAMETER	DELIVERABLE	COMMENTS
Retention Time Windows	NO	
Initial Calibration	NO	Note non-conformances in narrative
Continuing Calibration	NO	Note non-conformances in narrative
Method Blanks	YES	Note non-conformances in narrative. Flag all positive sample results above RL with "B" flag.
Lab Control Sample (LCS)	YES	Note non-conformances in narrative
Site Specific Matrix Spike/ Matrix Spike Duplicate	YES (If analyzed)	Note non-conformances in narrative
Surrogate Recoveries	YES	Note non-conformances in narrative
General Reporting Issues	YES	Note non-conformances in narrative
QA/QC Certification Form	YES	Signed by laboratory director or his/her designee

Table 1B Analyte List for SW-846 Method 8082

ANALYTE	CAS NUMBER	NOTES
Aroclor-1016	12674112	
Aroclor-1221	11104282	
Aroclor-1232	11141165	
Aroclor-1242	53469219	
Aroclor-1248	12672296	
Aroclor-1254	11097691	
Aroclor-1260	11096825	
Aroclor-1262	37324235	See 1
Aroclor-1268	11100144	See 1

Notes:
1. Arochlors 1262 and 1268 are not normally on the list of PCB's. If the chromatograms indicate these aroclors are present the laboratory is required to quantitate and report the compounds.

Table 2A Sample Containers, Preservation, and Holding Times

MATRIX	CONTAINER	PRESERVATIVE	HOLDING TIME
Aqueous with no chlorine present	1-liter amber glass bottle with Teflon line cap	Store at 4 ± 2º C.	7 days 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.	7 days 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	14 days to extraction. 40 days from extraction to analysis. Up to one year for samples frozen within 48 hours of collection. (Note 1)
High Concentration Waste Samples Excluding transformer oils.	Collect in amber glass jar with Teflon lined cap.	No special preservation requirement	14 days to extraction. 40 days from extraction to analysis.
Transformer / Waste Oils	Collect in glass jar with Teflon lined cap.	No special preservation requirement	One year

Notes:

Note 1: If the freezing option is selected, the sample must be frozen within 48 hours of collection. The holding time recommences when thawing begins. The total holding time is calculated from the time of collection to freezing plus the time allowed for thawing. The total elapsed time must be less than 14 days.

The number of sample containers is optional. Laboratories should supply enough containers to allow for any reanalysis or breakage.