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Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082

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1 Scope and Application

1.1 Method SW846-8082 is used to determine the concentrations of various Polychlorinated Biphenyls (PCBs) in waste. Samples are extracted with methylene chloride/acetone (1:1) using EPA Method 3541 then solvent exchanged with hexane. Samples are optionally cleaned by using EPA method 3640A. The extract is analyzed by injection into a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.

1.2 PCBs 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262 and 1268 are analyzed by this method. The EPD lab uses PCB 1660 (1016 + 1260) as the primary PCB mix for QC purposes. PCBs 1262 and 1268 may be analyzed for screening purposes only for Hazardous Waste projects as they are not included in the WP Performance Test studies.

1.3 This method is restricted to analysts who have completed the requirements of the initial demonstration SOP. Refer to SOP reference 13.1.

2 Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control definitions.

2.2 Refer to GA EPD Laboratory SOP 1-052, Organics Data Validation, online revision.

3 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in chromatograms.

3.2 Glassware must be scrupulously cleaned with hot water detergent followed by de-ionized water then rinsed with methanol followed by acetone. The glassware is rinsed again with extraction solvent, methylene chloride, immediately prior to use.

3.3 The use of high purity reagents and solvents helps to minimize interference problems.

3.4 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes.

3.5 Matrix interferences may be caused by contaminants that are co-extracted from the sample.

4 Safety

4.1 Refer to Georgia EPD Laboratory Chemical Hygiene Plan, online revision.

5 Apparatus and Equipment

5.1 Sample container: 250mL clear, wide-mouthed jar with lid

5.2 Vials: auto-sampler vials, clear and amber, screw top, 2.0mL, caps with septa and 300µL inserts

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5.3 Glass culture tubes: 5mL & 10mL with caps

5.4 Micro-syringes: various sizes

5.5 Syringes: various sizes

5.6 Spatulas: stainless steel or aluminum

5.7 Beakers: 250mL

5.8 Volumetric flasks (Class A): various sizes

5.9 Sample extract vials: minimum 10mL culture tubes with caps

5.10 Disposable pipettes and bulbs

5.11 Detergent: Steris Labklenz or equivalent

5.12 Brushes: various sizes

5.13 Volumetric Pipet, (Class A): 1.0mL & 2.0mL with squeeze bulb

5.14 Balance: Top loading, capable of accurately weighing to the nearest 0.01g

5.15 Balance: Analytical, capable of accurately weighing to the nearest 0.0001g

5.16 Aluminum Foil

5.17 Aluminum weigh boats

5.18 Automated Soxhlet system: Gerdhardt or equivalent

5.19 Soxhlet extraction beaker, 200mL with 6-hole rack

5.20 Cellulose thimbles: Gerdhardt 33x80mm or equivalent

5.20.1 Cellulose thimbles must be solvent rinsed before use.

5.20.1.1 Place thimbles in a tumbler extraction bottle.

5.20.1.2 Cover the thimbles with a solvent mix of 1:1 v/v methylene chloride and acetone.
5.20.1.3 Cap the bottle and allow the thimbles to soak in the solvent mix for 1 hour.
5.20.1.4 After 1 hour, drain the solvent mix off and refill the tumbler extraction bottle again with the 1:1 v/v methylene chloride and acetone solvent mix and soak again for 1 hour a second time.
5.20.1.5 After 1 hour, drain the solvent mix off of the cellulose thimbles and allow the thimbles to dry on aluminum foil completely inside a fume hood, typically overnight.
5.20.1.6 Once the cellulose thimbles are dry, they are ready for use and may be re-boxed for storage until needed.
5.21 Thimble clamps and wire thimble holders
5.22 Boiling chips
5.23 Oven: Fisher Isotemp, 105°C ± 2°C
5.24 GPC instrument (Gel Permeation Chromatography): Optional
5.24.1 *Note: If GPC cleanup is used, MDL studies must be in place with the use of the GPC instrument. If the GPC is not use, MDL studies must be in place without the use of the GPC instrument. MDL studies with and without the use of the GPC instrument may be maintained concurrently. Whichever option is used most frequently must be maintained with on-going MDLs. The alternate, least used option, either with or without GPC, may have MDLs maintained at baseline level. If either one of the options is not used, MDLs are not required for that option.*
5.25 Luer-Lock syringe: 10mL
5.26 Syringe filter: Whatman 0.45µm PTFE w/GMF or equivalent
5.27 TurboVap or similar concentrator with nitrogen blow down and controlled heating capabilities
5.28 TurboVap or similar concentration tubes with at least 50mL volume
5.29 RapidVap or similar concentrator with nitrogen blow down and controlled heating capabilities
5.30 RapidVap or similar concentration tubes with at least 300mL volume

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6 Reagents and Standards

6.1 Methylene chloride: pesticide grade or equivalent
6.2 Hexane: pesticide grade or equivalent
6.3 Acetone: pesticide grade or equivalent
6.4 Isooctane: pesticide grade or equivalent
6.5 Sand: purified, baked at 450°C for 4 hours
6.6 Sodium sulfate: granular, anhydrous, certified ACS grade suitable for pesticide residue analysis or equivalent
6.6.1 Sodium sulfate is baked for 4 hours at 450°C then stored in a glass container
6.7 Calibration Standard Solutions

6.7.1 Prepare five different concentrations equivalent to the concentration levels in Section 8.2 by dilution of the stock standard solutions. Standard stock solutions are usually at a concentration of 200µg/mL or 1000µg/mL in various solvents or from neat concentration. Calculations or amounts will vary depending on the stock standard concentration. Prepare the primary dilution standard of 200XP at 20µg/mL concentration for PCBs and 0.8-1.6µg/mL for Surrogates.

6.7.2 Calibration Standards for each PCB must have a minimum of 3 peaks selected but preferably 5 or more peaks with the exception of PCB 1221 which only has 3 peaks.

6.8 Initial Calibration Verification Standard Solutions (ICV)

6.8.1 Stock standard solutions prepared from a second source vendor's standards or a different lot from the same vendor as the calibration standards containing all of the analytes listed in Section 8.2, diluted in Hexane.

6.8.2 ICV standards are equivalent to Level 3 calibration standard in concentration listed in Section 8, Tables 8.2.2.

6.9 QC Spiking Solution

6.9.1 The spiking solution for SW846-8082 samples is typically a mix of PCB 1016 and PCB 1260 (PCB 1660). The typical volumes of standards used for preparing spikes are given in Section 6.9.2. These may be adjusted if necessary to meet the final concentration if the concentration of the vendor stock changes. An alternate PCB may be used for QC purposes if required for a special project.

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6.9.2 PCB 1660 Spike: The PCB 1660 100XP is made from a 4-8µg/mL SS:Surrogate Stock mix and 200µg/mL PCB 1660 Stock mix in Acetone. The PCBs may be added individually as PCB 1016 and PCB 1260 if necessary or alternate PCBs may be substituted if required. If the initial concentration is different from 200µg/mL, the volumes may be adjusted to meet the final concentration in Table 6.9.2.1. See Tables 6.9.2.1 – 6.9.2.2.

Table 6.9.2.1 – 8082 PCB 1660 100XP Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4.0	5.0	0.40
SS:DCPB	8.0		0.80
PCB 1660 (1016 + 1260)	200	2.5	10
Total Volume of Standard Aliquot		7.5mL	
Addition of Acetone to Standard Aliquot		42.5mL	
Final Volume of PCB 1660 100XP Spiking Standard		50mL	

Table 6.9.2.2 – 8082 PCB 1660 10XP Spiking Standard Final Concentration in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.40	1.0	0.04
SS:DCPB	0.80		0.08
PCB 1660 (1016 + 1260)	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of PCB 1660 10XP Spiking Standard in Sample Extract		10mL	

6.10 Surrogate Spiking Solution

6.10.1 The Surrogate Spiking solution is made from a 100-200µg/mL mix in Acetone. Note: Surrogates may be added individually if a mix is not available. Volumes may be adjusted if necessary to meet final concentration of 4-8µg/mL. The surrogates are spiked at 1.0mL per sample with a sample extract final volume of 10mL.

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Table 6.10.1 – 8082 SS: Surrogate Stamdard Spiking Solution 1000XPSS Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	100	2.0	4.0
SS:DCBP	200		8.0
Total Volume of Standard Aliquot		2.0mL	
Addition of Acetone to Standard Aliquot		48mL	
Final Volume of SS Spiking Solution in Acetone		50mL	

6.11 MDL Spike

6.11.1 The MDL Spike is made by diluting the PCB 1660 100XP by 1:10 in Acetone. The PCB MDL spike is spiked at 0.5mL per MDL with a 10mL sample extract final volume, see Tables 6.11.1.1 & 6.11.1.2.

Table 6.11.1.1 – 8082 PCB 1660 MDL Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.40	1.0	0.04
SS:DCPB	0.80		0.08
PCB 1660 (1016 + 1260)	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of PCB 1660 MDL Spiking Standard		10mL	

Table 6.11.1.2 – 8082 PCB 1660 MDL Spiking Standard Final Concentration in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.04	0.50	0.002
SS:DCPB	0.08		0.004
PCB 1660 (1016 + 1260)	1.0		0.05
Total Volume of Standard Aliquot		0.50mL	
Addition of Hexane to Standard Aliquot		9.5mL	
Final Volume of PCB 1660 MDL Spiking Standard in Extract		10mL	

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6.12 Expiration Dates

6.12.1 All standards that are made for SW846-8082 analysis have an expiration date of six months from the opening of the vendor stock ampule or the manufacturer's expiration date if less than six months from opening.

7 Sample Collection

7.1 Waste samples for Method SW846-8082 are collected in 8oz wide-mouth glass sample jars, typically 1-3 containers.

7.2 Samples are cooled to 0-6ºC (not frozen) after sample collection. Samples must be extracted within 14 days from collection and analyzed within 40 days of extraction.

8 Calibration

8.1 Calibration Curve

8.1.1 A five-point calibration is performed for PCB 1016 and PCB 1260 in a PCB 1660 mix. Five-point calibrations for alternate PCBs are performed only if necessary. The calibration system uses traceable certified standards. The calibration is an external standard calibration with an average of response factor linear curve fit and should result in a percent relative standard deviation < 20% between calibration levels of each analyte. The origin may not be forced.

8.2 Calibration Standards

8.2.1 All PCB calibration curves consist of calibration standards at the following concentrations (µg/mL): The PCB 1660 curve will be used as reference. All other PCBs (1221, 1232, 1242, 1248, 1254, 1262 & 1268) will be made in the same way unless the vendor stock is different than 200µg/mL and will have the same concentration levels as PCB 1660 in Table 8.2.2. Volumes may be adjusted to meet the final concentrations in Table 8.2.2. The PCB 1660 calibration curve is made from a 4000-8000µg/mL SS: Surrogate Stock mix and 200µg/mL PCB 1660 Stock.

Table 8.2.1 – 8082 PCB 1660 200XP Calibration Stock Standard in Hexane

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Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4000	5.0	800
SS:DCPB	8000		1600
PCB 1660 (1016 + 1260)	200	2.5	20
Total Volume of Standard Aliquot		7.5mL	
Addition of Hexane to Standard Aliquot		17.5mL	
Final Volume of PCB 1660 200XP Stock Standard		25mL	

Table 8.2.2 PCB 1660 Calibration Curve Levels (µg/mL)

Compound	Level 1 1XP	Level 2 5XP	Level 3 10XP	Level 4 15XP	Level 5 20XP
SS:TCMX	0.002	0.02	0.04	0.06	0.08
SS:DCBP	0.004	0.04	0.08	0.12	0.16
PCB 1660 (1016 + 1260)	0.05	0.50	1.0	1.5	2.0

Table 8.2.3 Aliquots of PCB 1660 Calibration Stock to make up all the levels in Table 8.2.2

(Aliquots corresponds to each level directly above each column)

	Level 1 1XP	Level 2 5XP	Level 3 10XP	Level 4 15XP	Level 5 20XP
Aliquot of PCB 1660 Calibration Stock 200XP (see Table 8.2.1)	0.050mL (50µL)	0.25mL (250µL)	0.50mL (500µL)	0.75mL (750µL)	1.0mL (1000µL)

Note: Bring all levels (points of the curve) up to 10mL by using **Hexane**

8.3 Calibration Verification

8.3.1 Second source calibration verification (ICV) must be analyzed after each initial calibration. All analytes must be within ± 15% of the expected value.

8.3.2 The ICVs for all PCBs are equivalent in concentration to Level 3 of the calibration curves.

8.3.3 The PCB 1660 ICV is made from individual PCB 1016 and PCB 1260 100µg/mL PCB Stocks. If the ICV vender stock is the same concentration as the Primary standard, then the ICV is made exactly like the primary calibration curve at Level 3 in Section 8.2. Surrogates are not included. All other PCBs (1221, 1232, 1242, 1248, 1254, 1262 and 1268) will be made in the same way unless the vendor stock is different than 100µg/mL.

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Table 8.3.3.1 – 8082 PCB 1660 ICV 100XP-ICV Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
PCB 1016	100	1.0	10
PCB 1260	100	1.0	10
Total Volume of Standard Aliquot		2.0mL	
Addition of Hexane to Standard Aliquot		8.0mL	
Final Volume of PCB 1660 ICV 100XP-ICV Stock Standard		10mL	

Table 8.3.3.2 – 8082 PCB 1660 ICV 10XP-ICV Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
PCB 1016	10	1.0	1.0
PCB 1260	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of PCB 1660 ICV 10XP-ICV Standard		10mL	

8.4 Record Keeping

8.4.1 Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with project records.

8.5 Daily Calibration Verification and Continuing Calibration

8.5.1 A continuing calibration standard (CCC) ensures the instruments target compound retention times and quantitation parameters meet method performance criteria. For any 12-hour analysis period, prior to sample analysis, a mid-point daily continuing calibration verification is performed for each pesticide and multi-component mix. Continuing calibration standards are analyzed during the analysis period to verify that instrument calibration accuracy does not exceed ±15% of the initial calibration, i.e. %Drift ≤ 15% (calculation 11.7). If the continuing calibration does not meet method performance criteria, then the instrument must be re-calibrated. A CCC is required after running the standard curve and initial calibration verification. After performing an initial calibration, an ICV may be substituted for a CCC if it meets method criteria for a CCC.

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8.6 Average Response Factor Calibration

8.6.1 To evaluate the linearity of the initial calibration, calculate the mean response factor (RF), the standard deviation (σ_{n-1}) and the relative standard deviation expressed as a percentage (%RSD). If the %RSD of the response factors is ≤ 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response may be used to determine sample concentrations. See Calculations 11.2.

8.7 Linear Calibration using First Order Least Squares Regression

8.7.1 Linearity through the origin is not assumed in a least squares fit. The instrument responses versus the concentration of the standards for the 5 points are evaluated using the instrument data analysis software. The regression will produce the slope and intercept terms for a linear equation. The regression calculation will regenerate a correlation, r, a measure of goodness of fit of the regression line to the data. A value

of 1.0 is a perfect fit. An acceptable correlation of coefficient should be $r \geq 0.990$ (or $r^2 \geq 0.980$). See Calculations 11.4.

8.7.2 Alternatively, second order quadratic fit may be used with an acceptable correlation of coefficient of $r \geq 0.990$ (or $r^2 \geq 0.980$). Note: quadratic fit will be calculated by chromatographic software. See Calculation 11.5.

8.8 Retention Time Windows

8.8.1 The width of the retention time window for each analyte, surrogate and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time of CCCs established over a 72 hour period from beginning injection to final injection over four days, with final injection occurring at a time earlier than the first injection so as to not exceed 72 hours. See Calculation 11.6.

8.8.2 CCCs used for RT Studies only are not required to meet continuing calibration criteria.

8.9 Daily Retention Time Update

8.9.1 Retention Times (RT) are updated once every 12 hours when ran on a GC for 8082 analysis. Each CCC is processed using Totalchrom software and the subsequent new RTs are saved in a copy of the Totalchrom method used for analyzing this batch of samples. To the existing Totalchrom method an extension is added by using "Month-Day-Year." The vial number where the update occurred may also be added to prevent confusion as there may be up to three or more RT updates in a single sequence. Hard copies of the calibration parameters are included with the data package for that batch of samples.

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8.10 Verification of Linear Calibrations

8.10.1 Calibration verification for linear calibrations involves the calculations of % drift of the instrument response between the initial calibration and each subsequent analysis of the verification standard. The % drift may be no more than ± 15%. See Calculation 11.7.

8.11 Sample Concentration

8.11.1 Sample results are expressed in mg/kg. See Calculation 11.9.

8.11.2 If an analyte response is calibrated by Average Response Factor, $\overline{RF}$, the chromatographic software calculates the concentration of the extract per equation 11.8, Calculations in µg/mL.

8.11.3 If an analyte response is calibrated by linear regression, the chromatographic software calculates the concentration of the extract solving for x per equation 11.4, Calculations in µg/mL.

8.11.4 If an initial volume of other than 2g is used or a dilution of the extract is analyzed, the final sample result is multiplied by the factor determined per equation 11.10.

9 Quality Control

9.1 Refer to Table 14.1 for Reporting Limits (RLs), Appendix A, Table A.1 for Quality Assurance criteria and Table 14.2 for a summary of Quality Control procedures associated with this method.

9.2 A Method Detection Limit Study for all analytes must be performed once per year. Refer to SOP Reference 13.4.

9.3. Method Detection Limit Study (MDL):

9.3.1. MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

9.3.2. The actual MDL varies depending on instrument and matrix.

9.3.3. The MDL must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.3.4. An MDL study may be done two different ways. The two different ways are considered and initial MDL study and a continuous MDL study. Both ways will be explained below.

9.4. Initial MDL study:

9.4.1. An initial MDL study may occur when a new instrument is brought online, changes to the method (which affect the compound of interest's peak area), and lastly major instrument repairs have been made.

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9.4.2. An initial MDL study will consist of the following operating parameters, 7 MDL samples and 7 MDL blanks. The 7 MDL samples study is performed by preparing 7 spiked vials, MDLSpike, spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDLBlank. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there may or may not be a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blanks.

9.5. Continuous MDL study:

9.5.1. A Continuous MDL study is preferred over the initial except in a few cases. For a continuous MDL study to be used on an instrument it must have a minimum of 7 MDL samples and 7 MDL blanks extracted over the course of multiple batches over a year. It is required that at a minimum 2 MDL samples and 2 MDL blanks must be ran per quarter per instrument. If this requirement is not met, then the initial MDL study must be performed for that instrument. (See section 9.4.2 for requirements.)

9.5.2. A continuous format MDL study is performed where one vial is spiked as an MDLSpike, at the lowest point of the calibration curve and analyzed with every batch of samples along with the method blank vial as an MDLBlank.

9.5.3. The results of the MDLBlank will be entered into Labworks using the Method Blank test code, $B_8082W. The MDLSpike result will be entered using the $ML8082W. The MDL Spiked Amount will be entered into the test code $MA8082W. The

instrument used for the MDL and Blank analysis will be selected using the test code INSTR-8082W.

9.6. MDL studies must be pulled on a yearly basis or an initial MDL study must be performed before the current MDL's for the instrument expire.

9.7. Refer to SOP Reference 13.1 for training and certification procedures.

9.8. Refer to SOP Reference 13.2 for control charting procedures.

9.9. LCS control limits are used to monitor LCSD recovery. LCSD recovery is not used to validate batch data; however, the LCS/LCSD precision (%RPD) is used for batch validation.

9.10. MS/MSD pairs are analyzed at a minimum of 5% of all samples analyzed.

9.11. Control Limits

9.12. Note: Analysts must use the control limits presented in Appendix A, Table A.1 for LCS/LCSDs. Those limits cannot exceed the default limits presented in Table 9.7.1.

Table 9.7.1: Default QC Limits*

	Compound	Default LCL %Recovery	Default UCL %Recovery	Default Precision %RPD
LCS/LCSD				
	PCB 1016	10	200	30
	PCB 1221	10	200	30
	PCB 1232	10	200	30
	PCB 1242	10	200	30
	PCB 1248	10	200	30
	PCB 1254	10	200	30
	PCB 1260	10	200	30
	PCB 1262	10	200	30
	PCB 1268	10	200	30
Surrogate				
	TCMX (Surrogate)	10 (0.02mg/kg)	200 (0.40mg/kg)	NA
	DCBP (Surrogate	10 (0.04mg/kg)	200 (0.80mg/kg)	NA
MS/MSD	Same as LCS/LCSD*			

*Methods 8000B and 8082 do not specify a range limit for Surrogate, LCS or MS recoveries or precisions. LCS recoveries are derived from Control Charting. The EPD lab will use the LCS/LCSD limits for the MS/MSD recovery limits. No recovery may be less than 10% or higher than 200%. The EPD Lab sets a default of no higher than 70% for the LCL and no less than 130% for the UCL. Precision RPD will be set at 30% default.

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9 Procedure

10.1 Refer to GA EPD Laboratory SOP – Automated Soxhlet Extraction – Method SW846-3541, SOP 1-029, Rev. 8 or later and GA EPD Laboratory SOP – Gel Permeation Chromatography (GPC) Cleanup – Method 3640A, SOP 1-005, Rev. 6 or later for the sample prep, extraction and optional cleanup procedures.

10.2 Upon completion of the extraction procedure, samples are diluted if necessary and vialed in 2mL autosampler vials using 300µL inserts to preserve sample volume if desired.

10.3 Analyze all sample extracts and QC using a gas chromatograph equipped with an electron capture detector.

10.4 Sample response is measured against the calibration curves. If the response exceeds the upper limit of the curve, the sample extract is diluted and re-analyzed.

10.4.1 Dilutions: Upon analysis of the extract, if a target compound response is greater than that of the highest standard of the calibration curve, the sample must be diluted with the final extraction solvent (Hexane) so that, upon analyzing the dilution (in a valid analysis sequence), the target response is between the lowest concentration standard (or the reporting limit, whichever is higher) and the highest concentration standard.

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10.5 A detect is considered to be positive if the quantitation amount is greater than the Reporting Limit for that compound. When a positive detect is found, the sample must be re-analyzed on a second, dissimilar confirmation column. If the difference between the quantitation amount found for the detected compound on the primary column and the confirmation column is greater than 40%, the detected compound is considered to be not confirmed. The Blanks, LCS and MS values are taken from the primary column. If the results of this column are out of acceptable range due to matrix interferences or other problems, the results may be reported from the confirmation column provided the calibration criteria are met.

10.5.1 If a detect for a PCB other than PCB 1660 occurs, the instrument will be calibrated with a five point curve for the alternate PCB and the Blank and affected sample(s) will be reanalyzed against this curve for concentration, retention time and pattern match.

10.6 For all PCB mixes, a fingerprint pattern and retention time match is required.

10.6.1 The chosen peaks for each PCB mix should not be disproportionately larger or smaller in the sample compared to the standard. The areas of the four (or more) peaks should be summed and averaged for use in determining the PCB concentration.

10 Calculations

11.1 Response Factor, RF, for a peak

$$RF = \frac{Area_{Analyte}}{Concentration_{Analyte}}$$

11.1.1 Where:

RF = Response Factor
Area $_{Analyte}$ = Area of the peak of the analyte of interest
Concentration $_{Analyte}$ = Concentration of the analyte of interest in µg/ml

11.2 Average Response Factor, $\overline{RF}$

$$\overline{RF} = \sum \frac{RF_i}{n}$$

11.2.1 Where:

$\overline{RF}$ = Mean response factor
RF_i = Response factor of compound at each level *i*
n = Number of calibration standards

11.3 Sample Standard Deviation (n – 1) (σ_{n-1}) of response factors

$$\sigma_{n-1} = \sqrt{\sum_{i=1}^{n} \frac{(RF_i - \overline{RF})^2}{n-1}}$$

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11.3.1 Where:

σ_{n-1} = Sample Standard Deviation
$\overline{RF}$ = Mean response factor
RF_i = Response factor of compound at each level *i*
n = Number of calibration standards

11.4 First Order Linear Regression Response Equation

$$Y = ax + b$$

This rearranges to:

$$x = Y - b/a$$

11.4.1 Where:

Y = Instrument response
a = Slope of the line
b = Intercept

x = Concentration in the extract or standard

11.5 Second Order Quadratic Fit Equation

11.5.1 $Y = ax^2 + bx + c$

11.5.2 Where:
Y = Instrument response
a = Slope of the line
b = Intercept
c = constant
x = Concentration in the extract or standard

11.5.3 Subtract Y from c to get modified equation $0 = ax^2 + bx + c$

11.5.4 Solve for x using the quadratic formula:

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

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11.5.5 A positive and negative value will be generated. Use positive value.

11.6 Average Retention Time, $\overline{RT}$

$$\overline{RT} = \sum \frac{RT}{n}$$

11.6.1 Where:
$\overline{RT}$ = Mean retention time for the target compound
RT = Retention time for the target compound
n = Number of values

11.7 Percent Drift, %Drift

$$\%Drift = \frac{(Concentration_{Calculated} - Concentration_{Expected})}{Concentration_{Expected}} * 100$$

11.7.1 Where:
$Concentration_{Calculated}$ = Concentration calculated from result

Concentration $_{Expected}$ = Theoretical concentration of the standard

11.8 Extract Concentration Calculation (µg/mL)

$$\mu g/mL = \frac{(A_s)}{(\overline{RF})}$$

11.8.1 Where:
A_s = Peak area of analyte
$\overline{RF}$ = Average Response Factor

11.9 Sample Concentration Calculation (mg/kg)

$$mg/kg = \frac{(A_s)(V_t)(D)}{(RF)(V_i)(W_s)}$$

11.9.1 Where:
A_s = Area of peak for analyte in sample
V_t = Extract volume in mL
D = Dilution factor
RF = Mean response factor (area per mg)
V_i = Volume of sample injected in µL
W_s = Original sample weight in kg

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11.10 Sample Concentration Adjustment for Varying Initial Volume and Dilutions

$$mg/kg_{Corrected} = mg/kg_{Uncorrected} * \frac{(0.002kg)(DF)}{W_s}$$

11.10.1 Where:
DF = Dilution Factor
W_s = Original sample weight in kg

11.11 Quality Control Calculations

$$\text{LCS/LCSD/ICV \% Recovery} = \frac{R_{spike}}{\text{Expected Result}} X\,100$$

$$\%\,\mathrm{RPD(precision)} = \frac{\left|R_{sample} - R_{duplicate}\right|}{\left(\frac{R_{sample} + R_{duplicate}}{2}\right)} X\,100$$

11.11.1 Where:

R_{spike} =% recovery of spiked sample
R_{sample} =% recovery of sample
$R_{duplicate}$ =% recovery of duplicate sample

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures, SOP6-015, online revision.

13 References

13.1 GA EPD Laboratory SOP's- Initial Demonstration of Capability SOP 6-001, online revision or later and/or Continuing Demonstration of Capability SOP 6-002, online revision.

13.2 GA EPD Laboratory SOP- EPD Laboratory Procedures for Control Charting and Control and Control Limits SOP, SOP 6-025, online revision.

13.3 GA EPD Laboratory SOP- EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

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13.4 GA EPD Laboratory SOP- Determination of Method Detection Limit, Method Detection Limit SOP 6-007, online revision.

13.5 GA EPD Laboratory SOP, Organics Data Validation, SOP 1-052, online revision.

13.6 GA EPD Laboratory SOP – Automated Soxhlet Extraction – EPA Method SW846-3541, SOP 1-029, online revision.

13.7 GA EPD Laboratory SOP, Percent Solids Determination – EPA Method 3541, SOP 1-042, online revision.

13.8 GA EPD Laboratory SOP, Gel Permeation Chromatography (GPC) Cleanup – Method 3640A, SOP1-005, online revision.

13.9 EPA Method SW846-8000B – Determinative Chromatographic Separation, Rev. 2, December 1996.

13.10 EPA Method SW846-8081A – Organochlorine Pesticides by Gas Chromatography, Rev. 1, December 1996.

13.11 EPA Method SW846-3541 – Automated Soxhlet Extraction, Rev. 0, September 1994.

13.12 EPA Method SW846-3640A – Gel-Permeation Cleanup, Rev. 1, September 1994.

13.13 GA EPD Laboratory Chemical Hygiene Plan, online revision.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

14.1 Refer to Appendix A, Table A.1 for precision and accuracy criteria.

Table 14.1 RLs for EPA Method SW846-8082 in Waste

Parameter/Method	Analyte	Matrix (Waste)	
		RL	Unit
SW846-8082 (Waste)	PCB 1016	1.65	mg/kg
	PCB 1221	1.65	mg/kg
	PCB 1232	1.65	mg/kg
	PCB 1242	1.65	mg/kg
	PCB 1248	1.65	mg/kg
	PCB 1254	1.65	mg/kg
	PCB 1260	1.65	mg/kg
	PCB 1262	1.65	mg/kg
	PCB 1268	1.65	mg/kg

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Table 14.2 Summary of Calibration and QC Procedures for EPA Method SW846-8082 in Waste

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method SW846-8082 (Waste)	Polychlorinated Biphenyls (PCBs)	5-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD for all analytes ≤ 20% linear-least squares regression r ≥ 0.990 or $r^2 \geq 0.980$	Correct problem then repeat initial calibration	

Table 14.2 Summary of Calibration and QC Procedures for EPA Method SW846-8082 in Waste

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method SW846-8082 (Waste)	Polychlorinated Biphenyls (PCBs)	Initial calibration verification (CCC)	Beginning each analysis sequence prior to the analysis of samples, after every 12 hours, and at the end of the analysis sequence	All analytes within ± 15% of expected values	If out of range high, high bias with no detects, generate a corrective action and use data. If low bias or with detects, rerun CCC and affected samples. If rerun passes, use data. If reruns do not pass, correct problem, repeat initial calibration verification and re-analyze all samples since last successful calibration verification	
		Second source calibration verification (ICV)	Once per initial calibration	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration	
		Retention Time window calculated for each analyte	Once per year or after major maintenance that would affect RTs	± 3 times standard deviation for each analyte retention time for standard analytical batch sequence	Correct problem then re-analyze all samples analyzed since the last retention time check	
		Retention time window update	Must be done every 12 hours with each CCC and prior to sample analysis	First CCC of each sequence and then every 12 hours	None	
		IDC-Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample, a Blind and a Blank	Once per analyst	QC acceptance criteria Table A.1, Appendix A	Locate and fix problem then re-run or re-extract demonstration for those analytes that did not meet criteria	

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Table 14.2 Summary of Calibration and QC Procedures for EPA Method SW846-8082 in Waste

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method SW846-8082 (Waste)	Polychlorinated Biphenyls (PCBs)	Surrogate spike	Every sample, spiked sample, standard and method blank	QC acceptance criteria Table A.1, Appendix A	Analyze second extract aliquot, if this does not pass, correct problem then re-extract and re-analyze the sample	
		Method Blank Solvent Blank	One per analytical batch of 20 or less samples	No analytes detected >RL	Analyze second extract aliquot, if this does not pass, correct problem then re-analyze or re-extract the blank and all samples in the affected batch	
		LCS/LCSD for all analytes	One per analytical batch of 20 or less samples	QC acceptance criteria Table A.1, Appendix A	Reanalyze once. If they fail a second time, correct problem the reanalyze or re-extract the LCS/LCSD and all samples in the affected batch	Flag QC sample report if LCSD exceeds upper acceptable control limits with passing RPD when high bias with no detects
		MS/MSD	Minimum of 5% of all samples analyzed	QC acceptance criteria Table A.1, Appendix A	Flag QC sample report	
		Second-column confirmation	100% for all positive results, ≤ 40% RPD for confirmation	If used for quantitation, same as for initial or primary column analysis	Same as for initial or primary column analysis	
		MDL study	Once per year or after major maintenance of the instrument	All Spiked MDLs must have a value greater than 0. Minimum Detection Limits established shall be < the RLs in Table 14.1	Re-do MDL Study	None

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Table 14.2 Summary of Calibration and QC Procedures for EPA Method SW846-8082 in Waste

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
		MDL analysis	Once per batch or as needed to acquire data points per SOP 6-007, online revision	All Spiked MDLs must have a value greater than 0. All other QC in the MDL blank and MDL sample (i.e. Surrogate Spike or Internal Standard, etc. if included) must meet established criteria	Correct problem and re-run the MDL sample or MDL blank once and initiate a corrective action. If the re-run fails a second time, do not use MDL data. Update corrective action, and use associated sample data	None
		Results reported between MDL and RL	None	None	None	

15 Associated LabWorks Test Codes

15.1 Parent Test Code
15.1.1 $8082W
15.2 Extraction Test Code
15.2.1 EXTN_PWT
15.3 QC Test Codes
15.3.1 $B_8082W – Extraction Blank Results
15.3.2 $LA8082W – LCS/LCSD Spike Amount
15.3.3 $LS8082W – LCS Results
15.3.4 $LD8082W – LCSD Results
15.3.5 $LR8082W – LCS Percent Recovery
15.3.6 $L28082W – LCSD Percent Recovery
15.3.7 $LP8082W – LCS/LCSD Precision
15.3.8 $A_8082W – MS/MSD Spike Amount
15.3.9 $S_8082W – MS Results
15.3.10 $D_8082W – MSD Results
15.3.11 $R_8082W – MS Percent Recovery
15.3.12 $RD8082W – MSD Percent Recovery
15.3.13 $P_8082W – MS/MSD Precision
15.3.14 $MA8082W – MDL Spike Amount
15.3.15 $ML8082W – MDL Results

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Appendix A – Quality Assurance Criteria for EPA Method SW846-8082 in Waste

Table A.1					
QC Type	Analyte	Accuracy (%R) LCL		UCL	Precision (%RPD)
LCS/LCSD*	PCB 1016	50	-	150	50
	PCB 1221	50	-	150	50
	PCB 1232	50	-	150	50
	PCB 1242	50	-	150	50
	PCB 1248	50	-	150	50
	PCB 1254	50	-	150	50
	PCB 1260	50	-	150	50
	PCB 1262	50	-	150	50
	PCB 1268	50	-	150	50
Surrogate*	TCMX	50	-	150	NA
	TCMX (as mg/kg)	0.10	-	0.30	NA
	DCBP	50	-	150	NA
	DCBP (as mg/kg)	0.20	-	0.60	NA
MS/MSD**	Same as LCS Recoveries	See Above			50

*Surrogate and LCS/LCSD recoveries are based on EPD set defaults due to the absence of a minimum of 20 data points for Control Charting. The EPD lab will use default limits of 50-150% recoveries for wastes.

** The EPD lab sets the MS/MSD recoveries and precisions as the same as the LCS/LCSDs.

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Updates: Appendix A added. Updated for online revision.