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Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Wastewater by Gas Chromatography – EPA Method 608.3

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1 Scope and Application

1.1 EPA Method 608.3 is used to determine the concentrations of various chlorinated hydrocarbon pesticides and polychlorinated biphenyls (PCBs) in wastewater. Samples are extracted at neutral pH with methylene chloride then solvent exchanged with hexane. 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 and 1260 are analyzed by this method. The EPD lab uses PCB 1660 (1016 + 1260) as the primary PCB mix for QC purposes.

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

1.3.1 Initial and continuing demonstrations for EPA Methods SW846-8081A and SW846-8082 in Water will be used for EPA Method 608.3 IDCs and CDCs.

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: 1.0L amber bottle with Teflon-lined caps
5.2 Vials: auto-sampler vials, clear, screw top, 2.0mL and 300µL inserts
5.3 Volumetric flasks (Class A): various sizes
5.4 Micro-syringes: various sizes
5.5 Syringes: various sizes
5.6 Drying column: Sodium sulfate
5.7 Glasswool: Baked at 400°C for 4 hours
5.8 Gas chromatograph: capable of temperature programming equipped for split/splitless injection
5.9 Mega bore 30m X 0.53mm, Rtx-CLP1 or equivalent (0.32mm may be used)
5.10 Mega bore 30m X 0.53mm, Rtx-CLP2 or equivalent (0.32mm may be used)
5.11 Electron capture detector
5.12 Chromatography software
5.13 Separatory Funnel: 2.0L with PTFE stopcock
5.14 Separatory Funnel Shaker
5.15 Graduated cylinders (Class A): 100mL & 1000mL
5.16 Erlenmeyer flasks: 250-300mL
5.17 Beakers: various sizes
5.18 pH indicator paper: pH range 0-14
5.19 Balance: Analytical, capable of accurately weighing to the nearest 0.0001g
5.20 Balance: Top-loading, capable of accurately weighing to the nearest 0.01g
5.21 RapidVap or similar concentrator with nitrogen blow down and controlled heating capabilities
5.22 RapidVap or similar concentration tubes with at least 300mL volume
5.23 TurboVap or similar concentrator with nitrogen blow down and controlled heating capabilities
5.24 TurboVap or similar concentration tubes with at least 50mL volume

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5.25 Sample extract vials: 10mL culture tubes with caps
5.26 Disposable pipettes and bulbs
5.27 Detergent: Steris Labklenz or equivalent

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 Reagent water: Purified water which does not contain any measureable quantities of target analytes or interfering compounds for each compound of interest (deionized, HPLC, Milli-Q or equivalent). Milli-Q water has a resistivity of 18 MΩ·cm or greater at 25°C and a TOC of 50µg/L or less.
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 100µg/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 at 1µg/mL concentration.
6.7.2 Calibration Standards for Chlordane will have 3-5 (or more) peaks chosen for calibration and Toxaphene will have 4-6 (or more) peaks chosen for calibration.
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 Sections 8.3 – 8.7, diluted in Hexane.
6.8.2 ICV standards are equivalent to Level 3 calibration standard in concentration listed in Section 8, Tables 8.3.2, 8.4.2, 8.5.2, 8.6.2 & 8.7.2.
6.9 QC Spiking Solutions
6.9.1 There are four separate spiking solutions for SW846-8081A samples. A Mix A spike, Mix B spike, Chlordane Spike and Toxaphene Spike. The typical volumes of standards used for preparing spikes are given in Sections 6.9.2 – 6.9.5. These may be adjusted if necessary to meet the final concentration if the concentration of the vendor stock changes.
6.9.2 Mix A Spike: The Mix A 100XA is made from a 10µg/mL Primary Stock #1A, a 10µg/mL Primary Stock #2A and an 8-80µg/mL Mix A mix in Acetone. The surrogates are included in the mix. The Mix A spike is spiked at 1.0mL per sample with a sample extract final volume of 10mL. See Tables 6.9.2.1 – 6.9.2.4.

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6.9.2.1 *Note: Chlorpyrifos is not reported by 608.3 analysis.*

6.9.2.2 *Note: Spikes and Standards for EPA Methods SW846-8081A and SW846-8082 will be used for 608.3 analysis. The names of the standards will remain as 8081A and 8082 throughout this SOP.*

Table 6.9.2.1 – 8081A Mix A Spiking Primary Stock #1A Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Chlorpyrifos (Dursban)	1000	0.25	10
Total Volume of Standard Aliquot		0.25mL	
Addition of Acetone to Standard Aliquot		24.75mL	
Final Volume of Mix A Primary Stock #1A		25mL	

Table 6.9.2.2 – 8081A Mix A Spiking Primary Stock #2A Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Mirex	1000	0.25	10
Total Volume of Standard Aliquot		0.25mL	
Addition of Acetone to Standard Aliquot		24.75mL	
Final Volume of Mix A Primary Stock #2A		25mL	

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Table 6.9.2.3 – 8081A Mix A 100XA Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	8.0	1.25	0.40
SS:DCBP	16		0.80
α-BHC	8.0		0.40
γ-BHC (Lindane)	8.0		0.40
p,p'-DDD	16		0.80
p,p'-DDT	16		0.80
Dieldrin	16		0.80
Endosulfan I	8.0		0.40
Endrin	16		0.80
Heptachlor	8.0		0.40

Table 6.9.2.3 – 8081A Mix A 100XA Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Methoxychlor	80		4.0
Chlorpyrifos (Dursban)	10	2.0	0.80
Mirex	10	2.0	0.80
Total Volume of Standard Aliquots		5.25mL	
Addition of Acetone to Standard Aliquots		19.75mL	
Final Volume of Mix A 100XA Spiking Standard		25mL	

Table 6.9.2.4 – 8081A Mix A 100XA 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:DCBP	0.80		0.08
α-BHC	0.40		0.04
γ-BHC (Lindane)	0.40		0.04
p,p'-DDD	0.80		0.08
p,p'-DDT	0.80		0.08
Dieldrin	0.80		0.08
Endosulfan I	0.40		0.04
Endrin	0.80		0.08
Heptachlor	0.40		0.04
Methoxychlor	4.0		0.40
Chlorpyrifos (Dursban)	0.80		0.08
Mirex	0.80		0.08
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Mix A Spiking Standard in Sample Extract		10mL	

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6.9.3 Mix B Spike: The Mix B 100XB is made from a 10µg/mL Primary Stock #1B and an 8-16µg/mL Mix B mix in Acetone. The surrogates are included in the mix. The Mix B spike is spiked at 1.0mL per sample with a sample extract final volume of 10mL. See Tables 6.9.3.1 – 6.9.3.3.

Table 6.9.3.1 – 8081A Mix B Spiking Primary Stock #1B Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Hexachlorobenzene	1000	0.25	10
Total Volume of Standard Aliquot		0.25mL	
Addition of Acetone to Standard Aliquot		24.75mL	
Final Volume of Mix B Primary Stock #1B		25mL	

Table 6.9.3.2 – 8081A Mix B 100XB Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	8.0	1.25	0.40
SS:DCBP	16		0.80
Aldrin	8.0		0.40
β-BHC	8.0		0.40
δ-BHC	8.0		0.40
α-Chlordane	8.0		0.40
γ-Chlordane	8.0		0.40
p,p'-DDE	16		0.80
Endosulfan II	16		0.80
Endosulfan Sulfate	16		0.80
Endrin Aldehyde	16		0.80
Endrin Ketone	16		0.80
Heptachlor Epoxide	16		0.80
Hexachlorobenzene	10	1.0	0.40
Total Volume of Standard Aliquots		2.25mL	
Addition of Acetone to Standard Aliquots		22.75mL	
Final Volume of Mix B 100XB Spiking Standard		25mL	

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Table 6.9.3.3 – 8081A Mix B 100XB 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:DCBP	0.80		0.08
Aldrin	0.40		0.04
β-BHC	0.40		0.04
δ-BHC	0.40		0.04
α-Chlordane	0.40		0.04
γ-Chlordane	0.40		0.04
p,p'-DDE	0.80		0.04
Endosulfan II	0.80		0.04
Endosulfan Sulfate	0.80		0.08
Endrin Aldehyde	0.80		0.08
Endrin Ketone	0.80		0.08
Heptachlor Epoxide	0.80		0.08
Hexachlorobenzene	0.40		0.04
Total Volume of Standard Aliquots		1.0mL	
Addition of Hexane to Standard Aliquots		9.0mL	
Final Volume of Mix B 100XB Spiking Standard in Sample Extract		10mL	

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6.9.4 Chlordane Spike: The Chlordane 100XC Spike is made from a 4-8µg/mL SS: Surrogate Stock mix and 1000µg/mL Chlordane Stock in Acetone. The Chlordane spike is spiked at 1.0mL per sample with a sample extract final volume of 10mL. See Tables 6.9.4.1, 6.9.4.2 & 6.10.1.

Table 6.9.4.1 – 8081A Chlordane 100XC Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4.0	2.5	0.40
SS:DCPB	8.0		0.80
Chlordane	1000	0.25	10
Total Volume of Standard Aliquot		2.75mL	

Table 6.9.4.1 – 8081A Chlordane 100XC Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Addition of Acetone to Standard Aliquot		22.25mL	
Final Volume of Chlordane 100XC Spiking Standard		25mL	

Table 6.9.4.2 – 8081A Chlordane 100XC 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
Chlordane	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Chlordane 100XC Spiking Standard in Standard Extract		10mL	

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6.9.5 Toxaphene Spike: The Toxaphene 100XT Spike is made from a 4-8µg/mL SS: Surrogate Stock mix and 1000µg/mL Toxaphene Stock in Acetone. The Toxaphene spike is spiked at 1.0mL per sample with a sample extract final volume of 10mL. See Tables 6.9.5.1, 6.9.5.2 & 6.10.1.

Table 6.9.5.1 – 8081A Toxaphene 100XT Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4.0	2.5	0.40
SS:DCPB	8.0		0.80
Toxaphene	1000	0.25	10
Total Volume of Standard Aliquot		2.75mL	
Addition of Acetone to Standard Aliquot		22.25mL	
Final Volume of Toxaphene 100XT Spiking Standard		25mL	

Table 6.9.5.2 – 8081A Toxaphene 100XT 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
Toxaphene	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Toxaphene 100XT Spiking Standard in Sample Extract		10mL	

6.9.6 PCB 1660 Spike: There 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 6.9.6.1 & 6.9.6.2. 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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6.9.6.1 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 6.9.6.1. See Tables 6.9.6.1 – 6.9.6.2.

Table 6.9.6.1 – 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	

Table 6.9.6.1 – 8082 PCB 1660 100XP Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Final Volume of PCB 1660 100XP Spiking Standard		50mL	

Table 6.9.6.2 – 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	

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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.

Table 6.10.1 – 8081A/8082 SS: Surrogate 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 Spikes

6.11.1 MDL Spikes are made by diluting the Mix A 100XA, Mix B 100XB, Chlordane 100XC, Toxaphene 100XT and PCB 1660 100XP by 1:10 in Acetone. They are not mixed.

6.11.2 The Mix A MDL spike and Mix B MDL spikes are each spiked at 0.5mL per MDL with a 10mL sample extract final volume. For Mix A MDL Spikes, see Tables 6.11.2.1 & 6.11.2.2. For Mix B MDL Spikes, see Tables 6.11.2.3 & 6.11.2.4.

Table 6.11.2.1 – 8081A Mix A 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:DCBP	0.80		0.08
α-BHC	0.40		0.04
γ-BHC (Lindane)	0.40		0.04
p,p'-DDD	0.80		0.08
p,p'-DDT	0.80		0.08
Dieldrin	0.80		0.08
Endosulfan I	0.40		0.04
Endrin	0.80		0.08
Heptachlor	0.40		0.04
Methoxychlor	4.0		0.40
Chlorpyrifos (Dursban)	0.40		0.04
Mirex	0.80		0.08
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of Mix A MDL Spiking Standard		10mL	

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Table 6.11.2.2 – 8081A Mix A 100XA Spiking Standard Final Concentration in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.04		0.002
SS:DCBP	0.08		0.004
α-BHC	0.04		0.002
γ-BHC (Lindane)	0.04		0.002

Table 6.11.2.2 – 8081A Mix A 100XA Spiking Standard Final Concentration in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
p,p'-DDD	0.08	0.50	0.004
p,p'-DDT	0.08		0.004
Dieldrin	0.08		0.004
Endosulfan I	0.04		0.002
Endrin	0.08		0.004
Heptachlor	0.04		0.002
Methoxychlor	0.40		0.02
Chlorpyrifos (Dursban)	0.04		0.002
Mirex	0.08	0.50	0.004
Total Volume of Standard Aliquot		0.50mL	
Addition of Hexane to Standard Aliquot		9.5mL	
Final Volume of Mix A MDL Spiking Standard in Sample Extract		10mL	

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Table 6.11.2.3 – 8081A Mix B 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:DCBP	0.80		0.08
Aldrin	0.40		0.04
β-BHC	0.40		0.04
δ-BHC	0.40		0.04
α-Chlordane	0.40		0.04
γ-Chlordane	0.40		0.04
p,p'-DDE	0.80		0.08
Endosulfan II	0.80		0.08
Endosulfan Sulfate	0.80		0.08
Endrin Aldehyde	0.80		0.08
Endrin Ketone	0.80		0.08
Heptachlor Epoxide	0.40		0.04
Hexachlorobenzene	0.80		0.08

Table 6.11.2.3 – 8081A Mix B MDL Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Total Volume of Standard Aliquots		1.0mL	
Addition of Acetone to Standard Aliquots		9.0mL	
Final Volume of Mix B MDL Spiking Standard		10mL	

Table 6.11.2.4 – 8081A Mix B 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:DCBP	0.08		0.004
Aldrin	0.04		0.002
β-BHC	0.04		0.002
δ-BHC	0.04		0.002
α-Chlordane	0.04		0.002
γ-Chlordane	0.04		0.002
p,p'-DDE	0.08		0.004
Endosulfan II	0.08		0.004
Endosulfan Sulfate	0.08		0.004
Endrin Aldehyde	0.08		0.004
Endrin Ketone	0.08		0.004
Heptachlor Epoxide	0.04		0.002
Hexachlorobenzene	0.08		0.004
Total Volume of Standard Aliquots		0.50mL	
Addition of Hexane to Standard Aliquots		9.5mL	
Final Volume of Mix B MDL Spiking Standard in Sample Extract		10mL	

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6.11.3 The Chlordane and Toxaphene MDL spikes are each spiked at 1.0mL per MDL with a 10mL sample extract final volume. For Chlordane MDL Spikes, see Tables 6.11.3.1 & 6.11.3.2. For Toxaphene MDL Spikes, see Tables 6.11.3.3 & 6.11.3.4.

Table 6.11.3.1 – 8081A Chlordane 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
Chlordane	10		1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of Chlordane MDL Spiking Standard		10mL	

Table 6.11.3.2 – 8081A Chlordane MDL Spiking Standard Final Concentration in Hexane

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Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.04	1.0	0.004*
SS:DCPB	0.08		0.008*
Chlordane	1.0		0.10
*Surrogates not at lowest point on the curve. Surrogates not used for MDL study.			
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Chlordane MDL Spiking Standard in Standard Extract		10mL	

Table 6.11.3.3 – 8081A Toxaphene 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
Toxaphene	10		1.0

Table 6.11.3.3 – 8081A Toxaphene MDL Spiking Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of Toxaphene MDL Spiking Standard		10mL	

Table 6.11.3.4 – 8081A Toxaphene MDL Spiking Standard Final Concentration in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.04	1.0	0.004*
SS:DCPB	0.08		0.008*
Toxaphene	1.0		0.10
*Surrogates not at lowest point on the curve. Surrogates not used for MDL study.			
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Toxaphene MDL Spiking Standard in Sample Extract		10mL	

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6.11.4 The PCB MDL spike is spiked at 0.5mL per MDL with a 10mL sample extract final volume, see Tables 6.11.4.1 & 6.11.4.2.

Table 6.11.4.1 – 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 6.11.4.2 – 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

6.12 Breakdown Standard Solution

6.12.1 A standard solution containing Endrin and DDT diluted in Hexane, used to calculate the breakdown of these compounds within the GC before and during the analysis of samples.

6.12.2 The 0.08µg/mL Breakdown Solution is made by diluting 80µL of 100µg/mL p,p'-DDT and 80µL of 100µg/mL Endrin into 100mL final volume Hexane.

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Table 6.12.2.1 – 8081A Breakdown Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
DDT	100	0.08	0.08
Endrin	100		0.08
Total Volume of Standard Aliquot			0.08mL
Addition of Hexane to Standard Aliquot			99.92mL
Final Volume of Breakdown Standard			100mL

6.13 Expiration Dates

6.13.1 All standards that are made for SW846-8081A/SW846-8082 and 608.3 analyses 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 Aqueous samples for Method 608.3 are collected in two to four amber, pre-certified 1000mL glass bottles with Teflon lined screw caps.

7.2 Samples are cooled to 0-6ºC (not frozen) after sample collection. Samples must be extracted within 7 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 all single and multi-peak components. 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

Note: It will be necessary to make separate curves for Mix A, Mix B, Chlordane and Toxaphene and PCB analyses. These are alternated in QA/QC batching; for instance, one batch will have Chlordane criteria and the next will have Toxaphene until all five have been used over five successive batches. CCCs for all five will be analyzed with each sample batch.

8.3 The Mix A calibration curve consists of the calibration standards at the following concentrations (µg/mL): *A vendor stock of 8-80µg/mL is used to make the Mix A stock at 200XA concentration with Chlorpyrifos and Mirex being at 1000µg/mL. A Primary Stock #1A and #2A is used to dilute Chlorpyrifos and Mirex to 10µg/mL exactly like Section 6.9.2, Tables 6.9.2.1 & 6.9.2.2. While the final solvent of Primary Stock #1A and Primary Stock #2A is still acetone, the final solvent for the Mix A 200XA calibration stock standard is hexane.*

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Table 8.3.1 – Mix A 200XA Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	8.0	1.0	0.80
SS:DCBP	16		1.6
α-BHC	8.0		0.80
γ-BHC (Lindane)	8.0		0.80
p,p'-DDD	16		1.6
p,p'-DDT	16		1.6
Dieldrin	16		1.6
Endosulfan I	8.0		0.80
Endrin	16		1.6
Heptachlor	8.0	1.0	0.80
Methoxychlor	80		8.0
Chlorpyrifos (Dursban)	10	1.6	1.6

Table 8.3.1 – Mix A 200XA Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Mirex	10	1.6	1.6
Total Volume of Standard Aliquots		4.2mL	
Addition of Hexane to Standard Aliquots		5.8mL	
Final Volume of Mix A 200XA Stock Standard		10mL	

Table 8.3.2 Mix A Calibration Curve Levels (µg/mL)

Compound	Level 1 0.5XA	Level 2 5XA	Level 3 10XA	Level 4 15XA	Level 5 20XA
SS:TCMX	0.002	0.02	0.04	0.06	0.08
SS:DCBP	0.004	0.04	0.08	0.12	0.16
α-BHC	0.002	0.02	0.04	0.06	0.08
γ-BHC (Lindane)	0.002	0.02	0.04	0.06	0.08
p,p'-DDD	0.004	0.04	0.08	0.12	0.16
p,p'-DDT	0.004	0.04	0.08	0.12	0.16
Dieldrin	0.004	0.04	0.08	0.12	0.16
Endosulfan I	0.002	0.02	0.04	0.06	0.08
Endrin	0.004	0.04	0.08	0.12	0.16
Heptachlor	0.002	0.02	0.04	0.06	0.08
Methoxychlor	0.02	0.20	0.40	0.60	0.80
Chlorpyrifos (Dursban)	0.004	0.04	0.08	0.12	0.16
Mirex	0.004	0.04	0.08	0.12	0.16

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Table 8.3.3 Aliquots of Mix A Calibration Stock to make up all the levels in Table 8.3.2

(Aliquots corresponds to each level directly above each column)

	Level 1 0.5XA	Level 2 5XA	Level 3 10XA	Level 4 15XA	Level 5 20XA
Aliquot of Mix A Calibration Stock 200XA (see Table 8.3.1)	0.025mL (25µL)	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**

8.4 The Mix B calibration curve consists of the calibration standards at the following concentrations (µg/mL): *A vendor stock of 8-16µg/mL is used to make the Mix B*

stock at 200XB concentration with Hexachlorobenzene being at 1000µg/mL. A Primary Stock #1B is used to dilute Hexachlorobenzene to 10µg/mL exactly like Section 6.9.3, Table 6.9.3.1. While the final solvent of Primary Stock #1B is still acetone, the final solvent for the Mix B 200XB calibration stock standard is hexane.

Table 8.4.1 – Mix B 200XB Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	8.0	1.0	0.80
SS:DCBP	16		1.6
Aldrin	8.0		0.80
β-BHC	8.0		0.80
δ-BHC	8.0		0.80
α-Chlordane	8.0		0.80
γ-Chlordane	8.0		0.80
p,p'-DDE	16		1.6
Endosulfan II	16		1.6
Endosulfan Sulfate	16		1.6
Endrin Aldehyde	16		1.6
Endrin Ketone	16		1.6
Heptachlor Epoxide	16		1.6
Hexachlorobenzene	10	0.80	0.80
Total Volume of Standard Aliquots		1.8mL	
Addition of Hexane to Standard Aliquots		8.2mL	
Final Volume of Mix B 200XB Stock Std		10mL	

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Table 8.4.2 Mix B Calibration Curve Levels (µg/mL)

Compound	Level 1 0.5XB	Level 2 5XB	Level 3 10XB	Level 4 15XB	Level 5 20XB
SS:TCMX	0.002	0.02	0.04	0.06	0.08
SS:DCBP	0.004	0.04	0.08	0.12	0.16
Aldrin	0.002	0.02	0.04	0.06	0.08
β-BHC	0.002	0.02	0.04	0.06	0.08
δ-BHC	0.002	0.02	0.04	0.06	0.08
α-Chlordane	0.002	0.02	0.04	0.06	0.08
γ-Chlordane	0.002	0.02	0.04	0.06	0.08
p,p'-DDE	0.004	0.02	0.04	0.06	0.08
Endosulfan II	0.004	0.04	0.08	0.12	0.16

Table 8.4.2 Mix B Calibration Curve Levels (µg/mL)

Compound	Level 1 0.5XB	Level 2 5XB	Level 3 10XB	Level 4 15XB	Level 5 20XB
Endosulfan Sulfate	0.004	0.04	0.08	0.12	0.16
Endrin Aldehyde	0.004	0.04	0.08	0.12	0.16
Endrin Ketone	0.004	0.04	0.08	0.12	0.16
Heptachlor Epoxide	0.004	0.04	0.08	0.12	0.16
Hexachlorobenzene	0.002	0.02	0.04	0.06	0.08

Table 8.4.3 Aliquots of Mix A Calibration Stock to make up all the levels in Table 8.4.2

(Aliquots corresponds to each level directly above each column)

	Level 1 0.5XB	Level 2 5XB	Level 3 10XB	Level 4 15XB	Level 5 20XB
Aliquot of Mix B Calibration Stock 200XB (see Table 8.4.1)	0.025mL (25µL)	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**

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8.5 The Chlordane calibration curve is made from a 4000-8000µg/mL SS: Surrogate Stock mix and 1000µg/mL Chlordane Stock.

Table 8.5.1 – 8081A Chlordane 200XC Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4000	5.0	0.80
SS:DCPB	8000		1.6
Chlordane	1000	0.50	20
Total Volume of Standard Aliquot		5.5mL	
Addition of Hexane to Standard Aliquot		19.5mL	
Final Volume of Chlordane 200XC Stock Standard		25mL	

Table 8.5.2 Chlordane Calibration Curve Levels (µg/mL)

Compound	Level 1 1XC	Level 2 5XC	Level 3 10XC	Level 4 15XC	Level 5 20XC
SS:TCMX	0.002	0.02	0.04	0.06	0.08
SS:DCBP	0.004	0.04	0.08	0.12	0.16
Chlordane	0.10	0.50	1.0	1.5	2.0

Table 8.5.3 Aliquots of Chlordane Calibration Stock to make up all the levels in Table 8.5.2

(Aliquots corresponds to each level directly above each column)

	Level 1 1XC	Level 2 5XC	Level 3 10XC	Level 4 15XC	Level 5 20XC
Aliquot of Chlordane Calibration Stock 200XC (see Table 8.5.1)	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**

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8.6 The Toxaphene calibration curve is made from a 4000-8000µg/mL SS: Surrogate Stock mix and 1000µg/mL Toxaphene Stock.

Table 8.6.1 – 8081A Toxaphene 200XT Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	4000	5.0	0.80
SS:DCPB	8000		1.6
Toxaphene	1000	0.50	20
Total Volume of Standard Aliquot		5.5mL	
Addition of Hexane to Standard Aliquot		19.5mL	
Final Volume of Toxaphene 200XT Stock Standard		25mL	

Table 8.6.2 Toxaphene Calibration Curve Levels (µg/mL)

Compound	Level 1 1XT	Level 2 5XT	Level 3 10XT	Level 4 15XT	Level 5 20XT
SS:TCMX	0.002	0.02	0.04	0.06	0.08

Table 8.6.2 Toxaphene Calibration Curve Levels (µg/mL)

Compound	Level 1 1XT	Level 2 5XT	Level 3 10XT	Level 4 15XT	Level 5 20XT
SS:DCBP	0.004	0.04	0.08	0.12	0.16
Toxaphene	0.10	0.50	1.0	1.5	2.0

Table 8.6.3 Aliquots of Toxaphene Calibration Stock to make up all the levels in Table 8.6.2

(Aliquots corresponds to each level directly above each column)

	Level 1 1XT	Level 2 5XT	Level 3 10XT	Level 4 15XT	Level 5 20XT
Aliquot of Toxaphene Calibration Stock 200XT (see Table 8.6.1)	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**

8.7 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 and 1254) 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.

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Table 8.7.1 – 8082 PCB 1660 200XP Calibration Stock Standard in Hexane

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 8.7.2 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 8.7.3 Aliquots of PCB 1660 Calibration Stock to make up all the levels in Table 8.7.2

(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 8.7.1)	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**

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8.8 Calibration Verification

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

8.8.2 The ICVs for all pesticide mixes are equivalent in concentration to Level 3 of the corresponding calibration curve.

8.8.3 The Mix A ICV consists of the calibration standards at the following concentrations (µg/L): *A vendor stock of 5-50µg/mL is used to make the Mix A stock at 125XA concentration with Chlorpyrifos at 1000µg/mL and Mirex at 100µg/mL. A Primary Stock #1A-ICV and #2A-ICV is used to dilute Chlorpyrifos and Mirex to 10µg/mL. See Tables 8.3.3.1 & 8.3.3.2. If the Vendor Stock is the same concentration as the Primary Standard, then the Mix A ICV will be made exactly as the primary calibration curve at Level 3 in Section 8.3.*

Table 8.8.3.1 – 8081A Mix A Spiking Primary Stock #1A-ICV Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Chlorpyrifos (Dursban)	1000	0.25	10
Total Volume of Standard Aliquot		0.25mL	
Addition of Acetone to Standard Aliquot		24.75mL	

Table 8.8.3.1 – 8081A Mix A Spiking Primary Stock #1A-ICV Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Final Volume of Mix A ICV Stock #1A-ICV		25mL	

Table 8.8.3.2 – 8081A Mix A Spiking Primary Stock #2A-ICV Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Mirex	100	1.0	10
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of Mix A ICV Stock #2A-ICV		10mL	

Table 8.8.3.3 – Mix A ICV 125XA-ICV Calibration Stock Standard in Hexane

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Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	5.0	1.0	0.50
SS:DCBP	10		1.0
α-BHC	5.0		0.50
γ-BHC (Lindane)	5.0		0.50
p,p'-DDD	10		1.0
p,p'-DDT	10		1.0
Dieldrin	10		1.0
Endosulfan I	5.0		0.50
Endrin	10		1.0
Heptachlor	5.0		0.50
Methoxychlor	50		5.0
Chlorpyrifos (Dursban)	10	1.0	1.0
Mirex	10	1.0	1.0
Total Volume of Standard Aliquots		3.0mL	
Addition of Hexane to Standard Aliquots		7.0mL	
Final Volume of Mix A ICV 125XA-ICV Stock Standard		10mL	

Table 8.8.3.4 – Mix A ICV 10XA-ICV Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.50	0.80	0.04
SS:DCBP	1.0		0.08
α-BHC	0.50		0.04
γ-BHC (Lindane)	0.50		0.04
p,p'-DDD	1.0		0.08
p,p'-DDT	1.0		0.08
Dieldrin	1.0		0.08
Endosulfan I	0.50		0.04
Endrin	1.0		0.08
Heptachlor	0.50		0.04
Methoxychlor	5.0		0.40
Chlorpyrifos (Dursban)	1.0		0.08
Mirex	1.0		0.08
Total Volume of Standard Aliquots		0.80mL	
Addition of Hexane to Standard Aliquots		9.2mL	
Final Volume of Mix A ICV 10XA-ICV Standard		10mL	

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8.8.4 The Mix B ICV consists of the calibration standards at the following concentrations (µg/L): *A vendor stock of 5-10µg/mL is used to make the Mix B stock at 125XB concentration with Hexachlorobenzene at 100µg/mL. A Primary Stock #1B-ICV is used to dilute Hexachlorobenzene to 10µg/mL. See Table 8.4.4.1. If the Vendor Stock is the same concentration as the Primary Standard, then the Mix B ICV will be made exactly as the primary calibration curve at Level 3 in Section 8.4.*

Table 8.8.4.1 – 8081A Mix B Spiking Primary Stock #1B-ICV Standard in Acetone

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Hexachlorobenzene	100	1.0	10
Total Volume of Standard Aliquot		1.0mL	
Addition of Acetone to Standard Aliquot		9.0mL	
Final Volume of Mix B ICV Stock #1B-ICV		10mL	

Table 8.8.4.2 – Mix B ICV 125XB-ICV Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	5.0	1.0	0.50
SS:DCBP	10		1.0
Aldrin	5.0		0.50
β-BHC	5.0		0.50
δ-BHC	5.0		0.50
α-Chlordane	5.0		0.50
γ-Chlordane	5.0		0.50
p,p'-DDE	5.0		0.50
Endosulfan II	10		1.0
Endosulfan Sulfate	10		1.0
Endrin Aldehyde	10		1.0
Endrin Ketone	10		1.0
Heptachlor Epoxide	10		1.0
Hexachlorobenzene	10	0.50	0.50
Total Volume of Standard Aliquots		1.5mL	
Addition of Hexane to Standard Aliquots		8.5mL	
Final Volume of Mix B ICV 125XB-ICV Stock Standard		10mL	

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Table 8.8.4.3 – Mix B ICV 10XB-ICV Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
SS:TCMX	0.50	0.80	0.04
SS:DCBP	1.0		0.08
Aldrin	0.50		0.04
β-BHC	0.50		0.04
δ-BHC	0.50		0.04
α-Chlordane	0.50		0.04

Table 8.8.4.3 – Mix B ICV 10XB-ICV Calibration Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
γ-Chlordane	0.50		0.04
p,p'-DDE	0.50		0.04
Endosulfan II	1.0		0.08
Endosulfan Sulfate	1.0		0.08
Endrin Aldehyde	1.0		0.08
Endrin Ketone	1.0		0.08
Heptachlor Epoxide	1.0		0.08
Hexachlorobenzene	0.50		0.04
Total Volume of Standard Aliquots		0.80mL	
Addition of Hexane to Standard Aliquots		9.2mL	
Final Volume of Mix B ICV 10XB-ICV Standard		10mL	

8.8.5 The Chlordane ICV is made from a 100µg/mL Chlordane Stock. 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 8.4. Surrogates are not included.

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Table 8.8.5.1 – 8081A Chlordane ICV 100XC-ICV Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Chlordane	100	1.0	10
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Chlordane ICV 100XC-ICV Stock Standard		10mL	

Table 8.8.5.2 – 8081A Chlordane ICV 10XC-ICV Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Chlordane	10	1.0	1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Chlordane ICV 10XC-ICV Standard		10mL	

8.8.6 The Toxaphene ICV is made from a 100µg/mL Toxaphene Stock. 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 8.5. Surrogates are not included.

Table 8.8.6.1 – 8081A Toxaphene ICV 100XT-ICV Stock Standard in Hexane

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Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Toxaphene	100	1.0	10
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Toxaphene ICV 100XT-ICV Stock Standard		10mL	

Table 8.8.6.2 – 8081A Toxaphene ICV 10XT-ICV Stock Standard in Hexane

Compound	Initial Concentration (µg/mL)	Aliquot (mL)	Final Concentration (µg/mL)
Toxaphene	10	1.0	1.0
Total Volume of Standard Aliquot		1.0mL	
Addition of Hexane to Standard Aliquot		9.0mL	
Final Volume of Toxaphene ICV 10XT-ICV Standard		10mL	

8.8.7 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 8.7. Surrogates are not included. All other PCBs (1221, 1232, 1242, 1248, 1254) will be made in the same way unless the vendor stock is different than 100µg/mL.

Table 8.8.7.1 – 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	

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Table 8.8.7.2 – 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	

8.9 Record Keeping

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

8.10 Daily Calibration Verification and Continuing Calibration

8.10.1 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.

8.11 Average Response Factor Calibration

8.11.1 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.

8.12 Linear Calibration using First Order Least Squares Regression

8.12.1 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.

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8.12.2 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.

8.13 Retention Time Windows

8.13.1 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.

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

8.14 Daily Retention Time Update

8.14.1 Retention Times (RT) are updated once every 12 hours when ran on a GC for 8081A 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.

8.15 Verification of Linear Calibrations

8.15.1 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.

8.16 Sample Concentration

8.16.1 Sample results are expressed in µg/L. See Calculation 11.9.

8.16.2 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.

8.16.3 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.

8.16.4 If an initial volume of other than 1000mL is used or a dilution of the extract is analyzed, the final sample result is multiplied by the factor determined per equation 11.10.

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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.

9.2.1 A Method Detection Limit study for all analytes must be performed initially, after major instrument repairs or changes to extraction procedures. MDL studies performed for these purposes can be done by the extraction and analysis of 7 samples and 7 blanks over 3 separate days.

9.2.2 The 7 MDL sample study is performed by extracting 7 spiked MDL samples, MDL_{Spike}, spiked at the lowest point of the curve and extracted along with 7 blank MDL samples, MDL_{Blank}. These sets of spiked and blank samples are extracted over 3 separate days and analyzed over a period of 3 separate days. There is a non-analysis day between each of the 3 days. A total of 14 samples are extracted, 7 spiked and 7 blank.

9.2.3 **On a continuous basis,** MDLs are performed by extraction and analysis of one sample spiked as an MDL_{Spike}, at the lowest point of the curve and extracted with every batch of samples along with the method blank, MDL_{Blank}, per each batch of

samples. The results of the MDL_{Spike} and MDL_{Blank} will be entered into LabWorks using the blank test code $B_8081H, and the MDL test code, $ML8081H, and the MDL Spiked Amount, $MA8081H. MDL reports will be pulled from LabWorks at a minimum of once per year (See SOP reference 13.4).

9.2.4 The higher value of the 2 MDLs, MDL_{Blank} or MDL_{Spike} will be used as the reporting MDL.

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

9.4 Refer to SOP Reference 13.2 for control charting procedures.

9.5 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.

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

9.7 Control Limits

9.7.1 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				
	Aldrin	60	140	30
	α-BHC	60	140	30
	β-BHC	60	140	30
	δ-BHC	60	140	30
	γ-BHC (Lindane)	60	140	30
	Chlordane	60	140	30
	α-Chlordane	60	140	30
	γ-Chlordane	60	140	30
	p,p'-DDD	60	140	30
	p-p'-DDE	60	140	30
	p,p'-DDT	60	140	30
	Dieldrin	60	140	30
	Endosulfan I	60	140	30
	Endosulfan II	60	140	30
	Endosulfan Sulfate	60	140	30
	Endrin	60	140	30
	Endrin Aldehyde	60	140	30
	Endrin Ketone	60	140	30
	Heptachlor	60	140	30

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Table 9.7.1: Default QC Limits*

	Compound	Default LCL %Recovery	Default UCL %Recovery	Default Precision %RPD
	Heptachlor Epoxide	60	140	30
	Hexachlorobenzene	60	140	30
	Methoxychlor	60	140	30
	Mirex	60	140	30
	Toxaphene	60	140	30
	PCB 1016	60	140	30
	PCB 1221	60	140	30
	PCB 1232	60	140	30
	PCB 1242	60	140	30
	PCB 1248	60	140	30
	PCB 1254	60	140	30
	PCB 1260	60	140	30
Surrogate				
	TCMX (Surrogate)	60 (0.24µg/L)	140 (0.56µg/L)	NA
	DCBP (Surrogate	60 (0.48µg/L)	140 (1.12µg/L)	NA
MS/MSD	Same as LCS/LCSD*			

*In the absence of 20 data points, method 608.3 specifies 60-140% recoveries for Surrogates, LCS and MS recoveries. The EPD laboratory sets a default 30% RPD for all compounds.

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9.8. Method Detection Limit Study (MDL):

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

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

9.8.3. 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.

9.8.4. 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.

9.9. Initial MDL study:

9.9.1. 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.

9.9.2. 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.

9.10. Continuous MDL study:

9.10.1. 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 9.9.2 for requirements.)

9.10.2. 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.

9.10.3. The results of the MDLBlank will be entered into Labworks using the Method Blank test code, $B_8081S. The MDLSpike result will be entered using the $ML8081S. The MDL Spiked Amount will be entered into the test code $MA8081S. The instrument used for the MDL and Blank analysis will be selected using the test code INSTR-8081S.

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

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10 Procedure

10.1 608.3 samples are immediately checked for neutral pH and Chlorine upon arrival at the laboratory or within 72 hours from sampling. Refer to GA EPD Laboratory SOP – pH and Chlorine Check – EPA 608.3 and 625.1, SOP 1-001, Rev. 1 or later.

10.2 Refer to GA EPD Laboratory SOP – Separatory Funnel Liquid-Liquid Extraction – EPA Method 3510C, SOP 1-028, Rev. 7 or later for the sample prep and extraction procedure.

10.3 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.

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

10.5 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.

10.5.1 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.

10.6 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.

10.7 Single peak analytes are identified as positive if detected within its appropriate retention time window on both columns. For multi-component analytes, a fingerprint pattern and retention time match is required.

10.7.1 Chlordane will be quantitated when the pattern in the sample reasonably matches that of the standard. Heptachlor, Heptachlor Epoxide, α-Chlordane and γ-Chlordane are calculated separately. The area of a minimum of three peaks, but preferably five or more peaks, should be summed and averaged for use in determining the Chlordane concentration. Weathered Chlordane no longer showing the characteristic pattern will be qualified as estimated (J).

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10.7.2 Toxaphene concentration is determined using four to six (or more) peaks. When front end degradation of the Toxaphene is apparent on the chromatogram, then the peaks should be taken from the latter half of the Toxaphene pattern. The chosen peaks should not be disproportionately larger or smaller in the sample compared to the standard. The areas of the four to six peaks should be summed and averaged for use in determining the Toxaphene concentration. Weathered Toxaphene no longer showing the characteristic pattern will be qualified as estimated (J).

10.7.3 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.

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

10.7.5 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.

11 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

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

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

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 (µg/L)

$$\mu g/L = \frac{(A_s)(V_t)(D)}{(RF)(V_i)(V_s)}$$

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 µg)
V_i = Volume of sample injected in µL
V_s = Original sample volume in mL

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11.10 Sample Concentration Adjustment for Varying Initial Volume and Dilutions

$$\mu g/L_{Corrected} = \mu g/L_{Uncorrected} * \frac{(1000\ mL)(DF)}{V_s}$$

11.10.1 Where:
DF = Dilution Factor
V_s = Original sample volume in mL

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

11.12 Breakdown Calculations

11.12.1 Endrin and DDT breakdown due to active sites in the injector or on the column with Endrin being oxidized and DDT being subjected to dechlorination. In addition, Endrin is subject to oxidation as a result of air leaking into the system or not being adequately scrubbed from the gases used for flow and makeup.

11.12.2 Breakdown for each main compound is calculated by determining the % recovery of each compound with respect to the total amount of main compound plus derivatives.

11.12.3 Endrin Breakdown:

$$\%\mathrm{Recovery\ of\ Endrin} = \left(\frac{\mathrm{Area_E}}{\mathrm{Area_E} + \mathrm{Area_{EA}} + \mathrm{Area_{EK}}}\right) * 100$$

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11.12.4 DDT Breakdown:

$$\%\mathrm{Recovery\ of\ DDT} = \left(\frac{\mathrm{Area_{DDT}}}{\mathrm{Area_{DDT}} + \mathrm{Area_{DDE}} + \mathrm{Area_{DDD}}}\right) * 100$$

11.12.5 Where:

$\mathrm{Area_E}$ = Area of Endrin peak in breakdown chromatogram
$\mathrm{Area_{EA}}$ = Area of Endrin aldehyde
$\mathrm{Area_{EK}}$ = Area of Endrin Ketone
$\mathrm{Area_{DDT}}$ = Area 4,4'-DDT
$\mathrm{Area_{DDE}}$ = Area 4,4'-DDE
$\mathrm{Area_{DDD}}$ = Area 4,4'-DDD

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, Rev. 3, online revision 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.

13.4 GA EPD Laboratory SOP – Determination of Method Detection Limit, Method Detection Limit SOP 6007, online revision.

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

13.6 GA EPD Laboratory SOP – Separatory Funnel Liquid-Liquid Extraction – EPA Method 3510C, SOP 1-028, online revision.

13.7 GA EPD Laboratory SOP – pH and Chlorine Check – EPA 608.3 and 625.1, SOP 1-001, online revision.

13.8 EPA Method 608.3 – Organochlorine Pesticides and PCBs By GC/HSD, December 2014.

13.9 EPA Method SW846-3510C – Separatory Funnel Liquid-Liquid Extraction, Rev. 3, December 1996.

13.10 GA EPD Laboratory Chemical Hygiene Plan, online revision.

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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 and MDL derived RLs.

Table 14.1 EPA Method 608.3 Default RLs in Water

Parameter/Method	Analyte	Matrix (Water)	
		RL	Unit
608.3 (Water)	Aldrin	0.024	µg/L
	α-BHC	0.018	µg/L
	β-BHC	0.021	µg/L
	δ-BHC	0.015	µg/L
	γ-BHC (Lindane)	0.033	µg/L
	Chlordane*	20.0	µg/L
	α-Chlordane	0.027	µg/L
	γ-Chlordane	0.024	µg/L
	p,p'-DDD	0.015	µg/L
	p-p'-DDE	0.030	µg/L
	p,p'-DDT	0.036	µg/L
	Dieldrin	0.018	µg/L

Table 14.1 EPA Method 608.3 Default RLs in Water

Parameter/Method	Analyte	Matrix (Water)	
		RL	Unit
608.3 (Water)	Endosulfan I	0.033	µg/L
	Endosulfan II	0.024	µg/L
	Endosulfan Sulfate	0.021	µg/L
	Endrin	0.012	µg/L
	Endrin Aldehyde	0.033	µg/L
	Endrin Ketone	0.024	µg/L
	Heptachlor	0.015	µg/L
	Heptachlor Epoxide	0.036	µg/L
	Hexachlorobenzene*	0.030	µg/L
	Methoxychlor	0.090	µg/L
	Mirex	0.012	µg/L
	Toxaphene	27.3	µg/L
	PCB 1016	4.50	µg/L
	PCB 1221	4.50	µg/L
	PCB 1232	4.50	µg/L
	PCB 1242	4.50	µg/L
	PCB 1248	4.50	µg/L
	PCB 1254	4.50	µg/L
	PCB 1260	4.20	µg/L

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***Compound RL (Method ML) not given in EPA Method 608.3. The EPD laboratory sets the default RL for Chlordane at 20.0µg/L and the default RL for Hexachlorobenzene at 0.030µg/L.**

Table 14.2 Summary of Calibration and QC Procedures for EPA Method 608.3 in Water

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 608.3 (Water)	Chlorinated hydrocarbon pesticides and Polychorinated 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	
		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 ± 20% 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	
		Breakdown check (Endrin & DDT)	Prior to analysis then every 12 hours	Degradation < 20% for either Endrin or DDT	Correct problem and re-analyze	

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Table 14.2 Summary of Calibration and QC Procedures for EPA Method 608.3 in Water

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 608.3 (Water)	Chlorinated hydrocarbon pesticides and Polychorinated Biphenyls (PCBs)	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	
		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	

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Table 14.2 Summary of Calibration and QC Procedures for EPA Method 608.3 in Water

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 608.3 (Water)	Chlorinated hydrocarbon pesticides and Polychorinated Biphenyls (PCBs)	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
		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	

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15 Associated LabWorks Test Codes

15.1 Parent Test Code

15.1.1 $608H

15.2 Extraction Test Code

15.2.1 608HE

15.3 QC Test Codes

15.3.1 $B_608H – Extraction Blank Results

15.3.2 $LA608H – LCS/LCSD Spike Amount

15.3.3 $LS608H – LCS Results

15.3.4 $LD608H – LCSD Results

15.3.5 $LR608H – LCS Percent Recovery

15.3.6 $L2608H – LCSD Percent Recovery

15.3.7 $LP608H – LCS/LCSD Precision

15.3.8 $A_608H – MS/MSD Spike Amount

15.3.9 $S_608H – MS Results

15.3.10 $D_608H – MSD Results

15.3.11 $R_608H – MS Percent Recovery

15.3.12 $RD608H – MSD Percent Recovery
15.3.13 $P_608H – MS/MSD Precision
15.3.14 $MA608H – MDL Spike Amount
15.3.15 $ML608H – MDL Results

Appendix A – Quality Assurance Criteria & RLs for EPA Method 608.3 in Wastewater*

Table A.1 Quality Assurance Criteria for EPA Method 608.3 in Wastewater

QC Type	Analyte	Accuracy (%R) LCL	UCL	Precision (%RPD)
LCS/LCSD*	a-BHC	60 -	140	30
	LINDANE (g-BHC)	60 -	140	30
	HEPTACHLOR	60 -	140	30
	ENDOSULFAN I	60 -	140	30
	DIELDRIN	60 -	140	30
	ENDRIN	60 -	140	30
	4,4-DDD	60 -	140	30
	4,4-DDT	60 -	140	30
	MIREX	60 -	140	30
	METHOXYCHLOR	60 -	140	30
	HEXACHLOROBENZENE	60 -	140	30
	b-BHC	60 -	140	30
	d-BHC	60 -	140	30
	ALDRIN	60 -	140	30
	HEPTACHLOR EPOXIDE	60 -	140	30
	gamma-CHLORDANE	60 -	140	30
	alpha-CHLORDANE	60 -	140	30
LCS/LCSD*	4,4-DDE	60 -	140	30
	ENDOSULFAN II	60 -	140	30
	ENDRIN ALDEHYDE	60 -	140	30
	ENDOSULFAN SULFATE	60 -	140	30
	ENDRIN KETONE	60 -	140	30
	CHLORDANE	60 -	140	30
	TOXAPHENE	60 -	140	30
	PCB 1016*	60 -	140	30
	PCB 1221*	60 -	140	30
	PCB 1232*	60 -	140	30
	PCB 1242*	60 -	140	30
	PCB 1248*	60 -	140	30

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QC Type	Analyte	Accuracy (%R) LCL	UCL	Precision (%RPD)
	PCB 1254*	60	140	30
	PCB 1260*	60	140	30
Surrogate*	TCMX	60	140	NA
	TCMX (as ug/L)	0.24	0.56	NA
	DCBP	60	140	NA
	DCBP (as µg/L)	0.48	1.12	NA
MS/MSD*	Same as LCS Recoveries	60	140	30

*Surrogate/LCS/LCSD/MS/MSD recoveries are based on Method Default limits. The EPD laboratory has < 20 data points for 608.3 Control Charting. The EPD lab sets a default of 30% RPD for all compounds.

Table A.2 MDLs and RLs for EPA Method 608.3 in Wastewater

Parameter	Analyte	MDLs	RLs (µg/L)
608.3 (Water)	a-BHC	0.01399	0.044
	LINDANE (g-BHC)	0.01410	0.045
	HEPTACHLOR	0.01006	0.032
	ENDOSULFAN I	0.00813	0.026
	DIELDRIN	0.01305	0.041
	ENDRIN	0.01989	0.063
	4,4-DDD	0.01747	0.056
	4,4-DDT	0.03097	0.10
	MIREX	0.01101	0.035
	METHOXYCHLOR	0.10730	0.34
	HEXACHLOROBENZENE	0.01343	0.043
	b-BHC	0.01144	0.036
	d-BHC	0.01411	0.045
	ALDRIN	0.01399	0.044
	HEPTACHLOR EPOXIDE	0.01062	0.034
	gamma-CHLORDANE	0.01259	0.040
	alpha-CHLORDANE	0.01263	0.040
	4,4-DDE	0.02139	0.068
	ENDOSULFAN II	0.02230	0.071
	ENDRIN ALDEHYDE	0.02312	0.074
	ENDOSULFAN SULFATE	0.01897	0.060
	ENDRIN KETONE	0.02302	0.073
	CHLORDANE	0.48330	1.5

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Parameter	Analyte	MDLs	RLs (µg/L)
	TOXAPHENE	0.42750	1.4
	PCB 1016*	0.21720	0.69
	PCB 1221*	NA	0.69
	PCB 1232*	NA	0.69
	PCB 1242*	NA	0.69
	PCB 1248*	NA	0.69
	PCB 1254*	NA	0.69
	PCB 1260*	0.21720	0.69

608.3 RLs are based on 3.18 x Lowest MDL values between instruments and columns. The method specifies 3.18 x Lowest MDL value plus rounding to the nearest whole number which is presented as the whole number in Tables 1 & 2 in EPA Method 608.3, December 2014. MDLs from SW846-8081A and SW846-8082 in Water are used for 608.3 MDLs.
*PCB MDLs will be derived from PCB 1016 and PCB 1260 only (PCB 1660). The lowest calculated MDL between PCB 1016 and PCB 1260 will be used to calculate all PCB RLs.
See Spreadsheet Below for Lowest MDL and RL calculations between instruments and columns.

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608.3 MDLx3 RLs	16A	16B	20A	20B	Lowest	608.3 RL	608.3
Compound	MDL	MDL	MDL	MDL	MDL	3.18 X MDL	Final RL
a-BHC	0.04054	0.04480	0.01399	0.02135	0.01399	**0.01399**	**0.044**
LINDANE (g-BHC)	0.01410	0.02213	0.01514	0.02043	0.01410	**0.01410**	**0.045**
HEPTACHLOR	0.01445	0.01006	0.01320	0.01210	0.01006	**0.01006**	**0.032**
ENDOSULFAN I	0.02368	0.04757	0.00813	0.00843	0.00813	**0.00813**	**0.026**
DIELDRIN	0.02935	0.02955	0.01305	0.02280	0.01305	**0.01305**	**0.041**
ENDRIN	0.02940	0.05642	0.01989	0.02331	0.01989	**0.01989**	**0.063**
4,4-DDD	0.04859	0.02965	0.01747	0.02577	0.01747	**0.01747**	**0.056**
4,4-DDT	0.04240	0.08756	0.03310	0.03097	0.03097	**0.03097**	**0.10**
MIREX	0.02242	0.06362	0.01101	0.03307	0.01101	**0.01101**	**0.035**
METHOXYCHLOR	0.13540	0.14120	0.10730	0.14090	0.10730	**0.10730**	**0.34**
HEXACHLOROBENZENE	0.01343	0.01898	0.01698	0.01515	0.01343	**0.01343**	**0.043**
b-BHC	0.02312	0.03298	0.01390	0.01144	0.01144	**0.01144**	**0.036**
d-BHC	0.01730	0.03597	0.01411	0.02165	0.01411	**0.01411**	**0.045**
608.3 MDLx3 RLs	16A	16B	21A	21B	Lowest	608.3 RL	608.3
Compound	MDL	MDL	MDL	MDL	MDL	3.18 X MDL	Final RL
ALDRIN	0.01930	0.02669	0.01858	0.01057	0.01057	**0.01057**	**0.034**
HEPTACHLOR EPOXIDE	0.02584	0.02750	0.01182	0.01062	0.01062	**0.01062**	**0.034**
gamma-CHLORDANE	0.02782	0.03950	0.01259	0.01343	0.01259	**0.01259**	**0.040**
alpha-CHLORDANE	0.03160	0.02847	0.01263	0.01300	0.01263	**0.01263**	**0.040**
4,4-DDE	0.73270	0.04803	0.02408	0.02139	0.02139	**0.02139**	**0.068**
ENDOSULFAN II	0.02871	0.05920	0.02323	0.02230	0.02230	**0.02230**	**0.071**

ENDRIN ALDEHYDE	0.02893	0.03890	0.02312	0.03105	0.02312	**0.02312**	**0.074**
ENDOSULFAN SULFATE	0.07084	0.03449	0.05710	0.01897	0.01897	**0.01897**	**0.060**
ENDRIN KETONE	0.04723	0.04708	0.02888	0.02302	0.02302	**0.02302**	**0.073**
CHLORDANE	0.64780	0.48990	0.56260	0.48330	0.48330	**0.48330**	**1.5**
TOXAPHENE	0.60930	0.83200	0.52250	0.42750	0.42750	**0.42750**	**1.4**
PCB 1016	0.54460	0.48560	0.47810	0.29700	0.29700	**0.29700**	**0.94**
PCB 1260	0.75060	0.66020	0.45640	0.21720	0.21720	**0.21720**	**0.69**

Updates: Appendix A added. Updated for online revision.

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