Global Operations, Environment, Health & Safety

159 Plastics Avenue
Pittsfield, MA 01201

July 21, 2017

Dean Tagliaferro
EPA Project Coordinator
U.S. Environmental Protection Agency
c/o Avatar Environmental
10 Lyman Street, Suite 2
Pittsfield, MA 01201

Re: GE-Pittsfield/Housatonic River Site (GECD900)
Addendum to Field Sampling Plan / Quality Assurance Project Plan; Biota Sampling and Analysis Procedures

Dear Mr. Tagliaferro:

On July 2, 2013, the General Electric Company (GE) submitted to the U.S. Environmental Protection Agency (EPA) a revised Field Sampling Plan/Quality Assurance Project Plan (FSP/QAPP). EPA approved the FSP/QAPP in a letter to GE dated July 23, 2013. Appendix H to the FSP/QAPP, titled Biota Sampling and Analysis Procedures, includes the standard operating procedures (SOPs) to be used for sampling and analyses of biota.

Some of the SOPs provided in Appendix H were provided by Pace Analytical Services, Inc. (Pace) and included information related to their specific methods for preparation and analysis of biota tissue for congener-specific PCB analysis or analysis of lipids. Specifically, the following SOPs were referenced:

- Attachment H-7, which provides the SOP for the preparation and homogenization of biota tissue samples for chemical analysis.
- Attachment H-8, which provides the SOP for the extraction and cleanup of PCBs from fish and biota material.
- Attachment H-9, which provides the SOP for congener-specific PCB analysis using the Green Bay method.
- Attachment H-10, which provides the SOP for the extraction and analysis of lipids from fish and biota material.

However, the Pace laboratory in Schenectady, New York, which performed these PCB and lipid analyses historically, was closed at the end of 2016. As a result, GE identified SGS AXYS located in British Columbia, Canada, as a laboratory qualified to perform the required analyses. Accordingly, GE proposes to use SGS AXYS for future analyses of biota samples.

SGS AXYS has provided GE with SOPs for their Method MLA-007 (Attachment A). This method is modified from EPA 8270C. The SOP from SGS AXYS differs slightly from the SOPs listed above that were previously provided by Pace; however, the substantive portions of the of the procedures are consistent with prior methods provided and approved by EPA for performance of preparation and analyses of biota material for PCB congeners and lipids.

SGS AXYS is accredited for analysis of PCB congeners by method MLA-007 through the Canadian Association for Laboratory Accreditation (CALA) (Attachment B). The complete scope of the accreditation from CALA is provided as Attachment C; page 37 of that document is specific to method MLA-007.

Please provide EPA approval of the use of the SGS AXYS and its attached SOPs for the analyses of biota for PCBs and lipids. In the meantime, if you have any questions about the information presented in this letter, please feel free to contact me at 413-553-6610 or Kevin.Mooney@ge.com.

Sincerely,

Lauren Putnam /for

Kevin G. Mooney
Senior Project Manager – Environmental Remediation

Attachments:
Attachment A – Standard Operating Procedures for SGS AXYS Laboratory Analyses of Biota
Attachment B – Accreditations
Attachment C – Scope of Accreditation

cc: John Kilborn, EPA*
Chris Ferry, ASRC Primus*
Robert Leitch, USACE*
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Public Information Repositories
GE Internal Repositories

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ATTACHMENT A

SGS AXYS Analytical Services Ltd.

SUMMARY OF SGS AXYS METHOD MLA-007 REV 13.09:

DETERMINATION OF PCB AROCLORS, TOTAL PCBs, CHLORINATED PESTICIDES, PCB CONGENERS, TECHNICAL TOXAPHENE, TOXAPHENE CONGENERS/PARLARS AND CHLOROBENZENES

MLA-007 describes the analytical procedures for the quantitative determination by GC and low resolution MS/ECD detection of any of the following analytical compound groups or combinations thereof:

- Individual PCB congeners
- PCBs as Aroclor equivalents, determined by weighted summing of individual PCB congeners
- Total PCBs by congener group, determined by summing of individual PCB congeners
- Total PCB, determined by summing of individual PCB congeners
- Chlorinated pesticides, including Technical Toxaphene and Chlorobenzenes
- Toxaphene as individual congeners/Parlars

The following matrices are covered by the method: Solids (sediment/soil/sludge/ash/ pulp), particulate filters, tissue (including blood and milk), aqueous samples, XAD-2 columns (resin and filters), ambient air samples (PUF and filter), and solvent extracts.

This method may be used for analysis of samples where USEPA Methods 608, 625, 1625B, 8081A, 8081B, 8270C, 8270D, or Method SM18 6630B have been requested **provided that the modifications described in this document are permitted by contract**. Key attributes of methods MLA-007, EPA 8270C/D, EPA 625, EPA 608 and EPA 8081A/B are summarized in the Appendix.

TARGET ANALYTES

PCBs as Aroclor Equivalents [1] and PCBs as Congener Groups [2]

Compound	CAS No.
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242/1016	53469-21-9/12674-11-2
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Total monochloro-PCBs	27323-18-8
Total dichloro-PCBs	25512-42-9
Total trichloro-PCBs	25323-68-6
Total tetrachloro-PCBs	26914-33-0
Total pentachloro-PCBs	25429-29-2
Total hexachloro-PCBs	26601-64-9
Total heptachloro-PCBs	28655-71-2
Total octachloro-PCBs	55722-26-4
Total nonachloro-PCBs	53742-07-7
Total decachloro-PCBs	2051-24-3
Total PCBs	1336-36-3

[1] Aroclor equivalents are quantified by weighed summing of a number of characteristic marker PCB congeners.

[2] Total PCBs are calculated as the sum of the individual PCB congener.

Chorobenzenes

Compound	CAS No.
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
1,3,5-Trichlorobenzene	108-70-3
1,2,4-Trichlorobenzene	120-82-1
1,2,3-Trichlorobenzene	87-61-6
1,2,3,5/1,2,4,5-Tetrachlorobenzene	634-90-2/95-94-3
1,2,3,4-Tetrachlorobenzene	634-66-2
Pentachlorobenzene	608-93-5
Hexachlorobenzene	118-74-1

Dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes are non-routine compounds and must be specifically requested by clients if required. The recoveries of these may be low due to loss through volatilization during the analytical work-up and they can be reported only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.

Pesticides

Compound	CAS No.
E1 Pesticides by GC/LRMS	
Hexachlorobenzene	118-74-1
alpha-HCH	319-84-6
beta-HCH	319-85-7
gamma-HCH	58-89-9
Heptachlor	76-44-8
Aldrin	309-00-2
Oxychlordane	27304-13-8
trans-Chlordane	5103-74-2
cis-Chlordane	5103-71-9
o,p'-DDE	3424-82-6
p,p'-DDE	72-55-9
trans-Nonachlor	39765-80-5
cis-Nonachlor	5103-73-1
o,p'-DDD	53-19-0
p,p'-DDD	72-54-8
o,p'-DDT	789-02-6
p,p'-DDT	50-29-3
Mirex	2385-85-5
Hexachlorobutadiene (upon request)	87-68-3
Octachlorostyrene (upon request)	29082-74-4
Technical Toxaphene (upon request)	8001-35-2

E2 Pesticides by GC/ECD	
cis-Heptachlor epoxide	1024-57-3
alpha-Endosulfan	959-98-8
delta-HCH	319-86-8
Dieldrin	60-57-1
Endrin	72-20-8
Methoxychlor	72-43-5
Endosulfan sulfate	1031-07-8
Endrin ketone	53494-70-5
beta-Endosulfan	33213-65-9
Endrin aldehyde [1]	7421-93-4

[1] Recovery of Endrin aldehyde in tissue matrix may be very low and the accuracy of results is not defined. Endrin aldehyde results for tissue samples are reported as "Information Values" only and represent estimated concentrations.

PCB Congeners

Results for all 209 PCB congeners may be reported by MLA-007.

Loss of one or more chlorines from a highly chlorinated congener may inflate or produce a false response for a less chlorinated congener of much lower abundance eluting at the same time, and analyst judgement applies to the determination of any potentially affected congener. This interference may be significant for PCBs 77, 81, 123, and 126, which are "toxic" congeners assigned TEF values by the World Health Organization and any concentrations reported for PCBs 77, 81, 123, and 126 should be interpreted as maximum values. Results for other low abundance PCB congeners may also be affected by this type of interference but the effect on results will be negligible in relation to the total PCB concentration.

Technical Toxaphene - (sum of major peaks)

Technical Toxaphene is estimated to contain 600 to 900 chlorinated bornanes, or chlorinated camphene compounds. The Technical Toxaphene product sold by Hercules and others is used as the analytical reference standard for measurement of the total amount of Technical Toxaphene in environmental samples. The analysis is performed by GC/LRMS (EI), involves comparison of 5-7 major peaks in the Technical Toxaphene calibration standard to those same peaks detectable in samples, and results in a single value.

Toxaphene Congeners/Parlars (selected chlorobornanes, Parlar II Suite)

Toxaphene congeners/Parlars consists of 600 – 900 individual chlorinated bornane or camphene components. Toxaphene congener/Parlars analysis quantifies a number of specific congeners present in the greatest concentration in samples. The individual components determined are:

Compound		**CAS No.**
2-exo,3-endo,6-exo,8,9,10-HexaCB	Hex sed (2)	57981-29-0
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	Hept sed (3)	208049-58-5
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	(Peak 5)	163390-24-7

2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	Parlar 32 (6)	254969-81-8 51775-36-1
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	(Peak 7)	206360-10-3 254969-83-0
2-exo,5-exo,6-endo,8,9,10,10-HeptaCB (aka 2-endo,3-exo,6-exo,8,9,10,10-HeptaCB)	(Peak 9)	163390-25-8
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	Parlar 26 (4)	142534-71-2
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	Parlar 39 (10)	64618-67-3
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	Par 40/41 (11,12)	166021-27-8 165820-16-6
2,2,5-endo,6-exo,8,8,9,10-OctaCB	Parlar 42a (13)	58002-18-9
2-exo,5,5,8,9,9,10,10-OctaCB	Parlar 44 (14)	165820-17-7
2,2,5-endo,6-exo,8,9,10,10-OctaCB	(Peak 15)	64618-69-5
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	(Peak 18)	254969-88-5
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	Parlar 50 (16)	66860-80-8
2,2,3-exo,5,5,9,9,10,10-NonaCB	(Peak 17)	
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	Parlar 56 (19/20)	64618-70-8 253340-63-5 64618-71-9
2,2,3-exo,5,5,8,9,10,10-NonaCB	Parlar 58 (21)	165820-20-2
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	Parlar 59	155750-49-5
2,2,5,5,8,9,9,10,10-NonaCB	Parlar 62 (22)	154159-06-5
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	Parlar 63	151214-79-8
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	Parlar 69	151183-19-6

Optional Analysis of Coplanar PCBs by GC/HRMS

PCB 81 PCB 77
PCB 126 PCB 169

1.0 EXTRACTION AND CLEANUP PROCEDURES

Prior to sample extraction, isotopically labeled surrogate standards are added to the sample. Solid (soil, sediment, sludge, ash, pulp) and tissue samples are dried with sodium sulfate and then Soxhlet extracted with dichloromethane. Fly ash samples that will not be analyzed for pesticides are pre-treated by sonication with dilute hydrochloric acid prior to extraction. XAD-2 samples are Soxhlet extracted with 80:20 toluene:acetone. Wet filters are Dean-Stark Soxhlet extracted with toluene. PUF and dry filter samples are Soxhlet co-extracted together with 80:20 toluene:acetone. Aqueous samples are liquid-liquid extracted with dichloromethane. Aqueous samples with more than 1% suspended solids are centrifuged prior to extraction; if the separated solids are to be included with the analysis, the Soxhlet extract of the solids is combined with the aqueous phase extract prior to clean-up. Milk samples are liquid-liquid extracted with acetone and hexane. Blood

samples (whole blood, serum, plasma) are liquid-liquid extracted with ethanol:hexane:saturated ammonium sulfate solution.

During cleanup tissue extracts are always first eluted through a gel permeation column to remove lipids. All extracts are fractionated on a Florisil column. To each of the two Florisil fractions aliquots of the appropriate ^{13}C-labeled recovery (internal) standards are added. The E1 fraction is analyzed for low polarity pesticides, Toxaphene, chlorobenzenes and PCBs using GC/LRMS. The E2 fraction is analyzed for polar pesticides by GC/ECD. The E2 fraction of blood samples must always undergo additional cleanup on a gel permeation column.

If analysis of coplanar PCBs by GC/HRMS is requested, cleanup of the E1 fraction is performed on a carbon/Celite column.

If necessary and depending on analytes, optional cleanup may be performed using acid washing, alumina column chromatography, gel permeation chromatography and/or layered acid/base silica column chromatography. Cleanup by alumina column chromatography or layered acid/base silica column chromatography must however not be performed on extracts analyzed for pesticides. Cleanup by acid washing must not be performed on pesticides in the Florisil E2 fraction.

2.0 INSTRUMENTATION

Analysis of Fraction E1

Gas chromatography/low resolution mass spectrometry (GC/LRMS) analysis is performed on a gas chromatograph (GC) coupled to a quadrupole mass spectrometer (MS). A DB-5 capillary chromatography column is connected directly to the MS source. The mass spectrometer is operated at a unit mass resolution in the electron ionization (EI) mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. A splitless/split injection sequence is used.

Analysis of Fraction E2

Gas chromatography/electron capture detection (GC/ECD) analysis is conducted on a gas chromatograph with a $^{63}Nickel$ electron capture detector and an integrator. A DB-5 capillary column is connected directly to the ECD source. Where required, analyte confirmation is performed by simultaneous analysis on a DB-17 capillary column.

Analysis of Toxaphene congeners/Parlars in Fraction E1

Gas chromatography/low resolution mass spectrometry (GC/LRMS) analysis of the E1 fraction is performed on a DB-5 capillary column. The MS is operated at unit mass resolution in the electron capture negative ionization mode (ECNI) using multiple ion detection (MID) acquiring at least two characteristic ions for each target analyte and surrogate standard. A splitless/split injection sequence is used.

Optional analysis of coplanar PCBs in Fraction E1

Gas chromatography/high resolution mass spectrometry (GC/HRMS) analysis is performed using a DB-5 or an Octyl capillary gas chromatography column.

3.0 CALIBRATION

Initial calibration (default procedure) is performed using a series of five calibration solutions that encompass the working concentration range of the instrument. The initial calibration solutions contain surrogates, recovery standards and native analytes. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution.

SGS AXYS Analytical Services Ltd.

Nominal Concentrations of Fraction E1 Calibration Standard Solutions for Chlorobenzenes, Pesticides and PCBs

Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
1,3-dichlorobenzene	10	20	40	160	400	2000	
1,4-dichlorobenzene	10	20	40	160	400	2000	
1,2-dichlorobenzene	10	20	40	160	400	2000	
1,3,5-trichlorobenzene	10	20	40	160	400	2000	
1,2,4-trichlorobenzene	10	20	40	160	400	2000	
1,2,3-trichlorobenzene	10	20	40	160	400	2000	
1,2,3,5- /1,2,4,5-tetrachlorobenzene	20	40	80	320	800	4000	
1,2,3,4-tetrachlorobenzene	10	20	40	160	400	2000	
Pentachlorobenzene	10	20	40	160	400	2000	
Hexachlorobenzene	10	20	40	160	400	2000	
alpha-HCH	20	40	80	320	800	4000	
beta-HCH	20	40	80	320	800	4000	
gamma-HCH	20	40	80	320	800	4000	
delta-HCH	20	40	80	320	800	4000	
Heptachlor	10	20	40	160	400	2000	
Aldrin	20	40	80	320	800	4000	
trans-Chlordane	20	40	80	320	800	4000	
cis-Chlordane	20	40	80	320	800	4000	
trans-Nonachlor	20	40	80	320	800	4000	
cis-Nonachlor	20	40	80	320	800	4000	
o,p'-DDE	10	20	40	160	400	2000	
p,p'-DDE	10	20	40	160	400	2000	10000
o,p'-DDT	10	20	40	160	400	2000	
p,p'-DDT	10	20	40	160	400	2000	
Octachlorostyrene	10	20	40	160	400	2000	
Oxychlordane	20	40	80	320	800	4000	
op'-DDD	10	20	40	160	400	2000	
pp'-DDD	10	20	40	160	400	2000	
Mirex	10	20	40	160	400	2000	
PCB 1	10	20	40	160	400	2000	
PCB 3	10	20	40	160	400	2000	
PCB 4	10	20	40	160	400	2000	
PCB 8	10	20	40	160	400	2000	
PCB 15	10	20	40	160	400	2000	
PCB 18	10	20	40	160	400	2000	
PCB 19	10	20	40	160	400	2000	
PCB 34/23	10	40	80	320	800	4000	
PCB 28	10	20	40	160	400	2000	

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Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
PCB 31	10	20	40	160	400	2000	
PCB 34	10	20	40	160	400	2000	
PCB 37	10	20	40	160	400	2000	
PCB 40	10	20	40	160	400	2000	
PCB 44	10	20	40	160	400	2000	
PCB 49	10	20	40	160	400	2000	
PCB 52	10	20	40	160	400	2000	
PCB 54	10	20	40	160	400	2000	
PCB 56	10	20	40	160	400	2000	
PCB 66	10	20	40	160	400	2000	
PCB 77	10	20	40	160	400	2000	
PCB 81	10	20	40	160	400	2000	
PCB 87	10	20	40	160	400	2000	
PCB 95	10	20	40	160	400	2000	
PCB 99	10	20	40	160	400	2000	
PCB 101	10	20	40	160	400	2000	
PCB 104	10	20	40	160	400	2000	
PCB 105	10	20	40	160	400	2000	
PCB 110	10	20	40	160	400	2000	
PCB 114	10	20	40	160	400	2000	
PCB 118	10	20	40	160	400	2000	
PCB 123	10	20	40	160	400	2000	
PCB 126	10	20	40	160	400	2000	
PCB 138	10	20	40	160	400	2000	
PCB 149	10	20	40	160	400	2000	
PCB 151	10	20	40	160	400	2000	
PCB 153	10	20	40	160	400	2000	
PCB 155	10	20	40	160	400	2000	
PCB 156	10	20	40	160	400	2000	
PCB 157	10	20	40	160	400	2000	
PCB 167	10	20	40	160	400	2000	
PCB 169	10	20	40	160	400	2000	
PCB 170	10	20	40	160	400	2000	
PCB 180	10	20	40	160	400	2000	
PCB 183	10	20	40	160	400	2000	
PCB 187/182	20	40	80	320	800	4000	
PCB 188	10	20	40	160	400	2000	
PCB 189	10	20	40	160	400	2000	
PCB 194	10	20	40	160	400	2000	
PCB 196	10	20	40	160	400	2000	

SGS AXYS Analytical Services Ltd.

Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
PCB 202	10	20	40	160	400	2000	
PCB 204	10	20	40	160	400	2000	
PCB 205	10	20	40	160	400	2000	
PCB 206	10	20	40	160	400	2000	
PCB 208	10	20	40	160	400	2000	
PCB 209	10	20	40	160	400	2000	
Labeled Surrogates							
$^{13}C_6$-1,4-dichlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-1,2,3-trichlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-1,2,3,4-tetrachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-Pentachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-Hexachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-beta-HCH	400	400	400	400	400	400	400
$^{13}C_6$-gamma-HCH	400	400	400	400	400	400	400
$^{13}C_{10}$-Heptachlor	400	400	400	400	400	400	400
$^{13}C_{12}$-Aldrin	400	400	400	400	400	400	400
$^{13}C_{10}$-trans-Chlordane	400	400	400	400	400	400	400
$^{13}C_{10}$-trans-Nonachlor	400	400	400	400	400	400	400
$^{13}C_{12}$-p,p'-DDE	400	400	400	400	400	400	400
$^{13}C_{12}$-p,p'-DDT	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 3	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 8	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 28	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 101	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 118	400	400	400	400	400	400	400
$^{13}C_{12}$ PCB 180	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 202	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 206	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 209	400	400	400	400	400	400	400
Recovery Standards							
$^{13}C_{12}$-PCB 52	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 138	400	400	400	400	400	400	400

Note: Level A is a sensitivity standard only.

SGS AXYS Analytical Services Ltd.

Nominal Concentrations of Calibration Solutions for Technical Toxaphene

Compound Name	Calibration Standards					
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (ng/mL)	Level F CS-5 (ng/mL)
Technical Toxaphene	2500	5000	25000	100000	250000	800000
Surrogate Standard						
$^{13}C_{12}$-PCB 159	400	400	400	400	400	400
Recovery Standard						
$^{13}C_{12}$-PCB 138	400	400	400	400	400	400

Notes: Level A is a sensitivity standard only.
Level D CAL VER or bracketing CAL level.

Nominal Concentrations of E2 Calibration Solutions for GC/ECD Analysis of E2 Pesticides

Compound Name	Level A CS-0 (ng/mL)	Calibration Standards				
		Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (Mid-Level) (ng/mL)	Level E CS-4 (ng/mL)	Level G CS-5 (ng/mL)
Dieldrin	4	8	40	80	160	800
alpha-Endosulfan	4	8	40	80	160	800
beta-Endosulfan	4	8	40	80	160	800
Endosulfan Sulfate	4	8	40	80	160	800
Endrin	4	8	40	80	160	800
Methoxychlor	4	8	40	80	160	800
Endrin Aldehyde	4	8	40	80	160	800
Endrin Ketone	4	8	40	80	160	800
cis-Heptachlor Epoxide	4	8	40	80	160	800
delta-HCH	5	10	50	100	200	500
Surrogates						
d_4-alpha-Endosulfan	75	75	75	75	75	75
d_4-beta-Endosulfan	75	75	75	75	75	75
Recovery Standard						
$^{13}C_{12}$-PCB 153	75	75	75	75	75	75

Note: Level A is a sensitivity standard only.

4.0 QUANTIFICATION PROCEDURES

Target concentrations are determined with respect to a labeled surrogate. Mean relative response factors (RRF), determined from either a multi-level initial calibration series or a mid-level calibration standard run at the beginning and end of the samples, are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\underset{\text{(ng/g or ng/L)}}{\text{Concentration of Target}} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{concentration of Qt Std}}{\text{concentration of Target}}\right)$

and the Qt Std is either the surrogate or the internal standard

Concentration results for target compounds are recovery corrected by the method of quantification. Surrogate recoveries are determined similarly against the recovery (internal) standard and are used as general indicators of overall analytical quality.

Aroclor equivalent concentrations may be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclor mixtures[1].

Aroclor 1016[2] = the sum of PCB 8/18/31/28 concentrations multiplied by 3.0;
Aroclor 1221 = the sum of PCB 3/4/8 concentrations multiplied by 3.0;
Aroclor 1232 = the sum of PCB 8/18/28/31 concentrations multiplied by 5.0;
Aroclor 1242[2] = the sum of PCB 8/18/31/28 concentrations multiplied by 3.8;
Aroclor 1248 = the sum of PCB 66/44/49 concentrations multiplied by 5.5;
Aroclor 1254 = the sum of PCB 87/97/99 concentrations multiplied by 10;
Aroclor 1260 = the sum of PCB 183/180/170 concentrations multiplied by 6.0;

[1] The congeners listed may have additional co-eluting congeners but these are insignificant with respect to the sums.

[2] Aroclors 1016 and 1242 may be reported as combined 1016/1242 using the 1242 factor if allowed by contract.

Environmental samples with no clearly identified Aroclor signature are quantified as 1242/1254/1260 mixtures. Results may be reported as Aroclor 1248 instead of Aroclor 1242 and 1254 where the congener pattern clearly indicates this formulation. Other Aroclor formulations may be reported by calibration against the specific Aroclor solutions.

An additional high level calibration standard containing p,p'-DDE is analyzed during the initial calibration of the instrument to extend the calibration range for that compound.

The target analyte delta-HCH elutes primarily in the E2 fraction and is quantified from the E2 fraction analysis data using the recovery corrected procedure described in the method.

Technical Toxaphene is determined by summing the responses of five peaks and quantifying by internal standard quantification procedures.

Technical chlordane is quantified by summing the concentrations of Oxychlordane, cis- and trans-Chlordane, and cis- and trans-Nonachlor, and multiplying by 2.5.

4.1 Reporting Limits

Sample specific detection limits (SDL) are determined from the analysis data by converting the minimum detectable area to a concentration following the same procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as three times the height of the noise in the m/z channel of interest, converted to an area using the area height ratio of the corresponding labeled surrogate peak.

SGS AXYS Analytical Services Ltd.

Analyte Ions Monitored, Surrogates Used, and RRF Determination for E1 Pesticides and PCB Congeners by GC/LRMS

(Target compounds are listed below the labeled standard used for quantification. No entry in the "RRF Used" field designates an RRF derived from that same compound.)

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
^{13}C-PCB 52 -REC[1]	**23.27**	**302**	**304**	**130**	
$^{13}C_6$-1,4-DiCB	**7.57**	**154**	**152**	**156**	
1,3-DiCB	7.52	146	148	64	
1,4-DiCB	7.57	146	148	64	
1,2-DiCB	7.77	146	148	64	
$^{13}C_6$-1,2,3-TriCB	**9.63**	**190**	**188**	**312**	
1,3,5-TriCB	8.71	180	182	95.9	
1,2,4-TriCB	9.2	180	182	95.9	
1,2,3-TriCB	9.63	180	182	95.9	
$^{13}C_6$-1,2,3,4-TetCB	**11.77**	**224**	**222**	**208**	
1,2,3,5-/1,2,4,5-TetCB	11.09	216	214	78.2	
1,2,3,4-TetCB	11.77	216	214	78.2	
Hexachlorobutadiene	9.65	225	260	38.4	1,2,3,4-TetCB
$^{13}C_6$-PentaCB	**14.07**	**256**	**260**	**20.4**	
PentaCB	14.07	250	252	64	
$^{13}C_6$-HexaCB	**17.87**	**292**	**294**	**42.6**	
HexaCB	17.87	284	286	80	
$^{13}C_6$-beta HCH	**18.7**	**225**	**227**	**48**	
beta-HCH	18.7	219	217	78.2	
$^{13}C_6$-gamma HCH	**18.94**	**225**	**227**	**48**	
alpha-HCH	17.52	219	217	78.2	
gamma-HCH	18.94	219	217	78.2	
delta-HCH	20.01	219	217	78.2	
$^{13}C_{10}$-Heptachlor	**22.25**	**277**	**279**	**80**	
Heptachlor	22.28	272	270	50	
$^{13}C_{12}$-Aldrin	**23.92**	**270**	**272**	**64**	
Aldrin	23.94	263	261	62.5	
$^{13}C_{10}$-trans-Chlordane	**27.19**	**383**	**385**	**96**	
trans-Chlordane	27.2	373	375	95.9	
cis-Chlordane	28	373	375	95.9	
Octachlorostyrene	25.86	380	378	89.4	
Oxychlordane	26.04	185	115	120	
$^{13}C_{10}$-trans-Nonachlor	**28.26**	**419**	**421**	**64**	
trans-Nonachlor	28.28	409	411	64	
cis-Nonachlor	31.5	409	411	64	

[1] REC = Recovery Standard, added just prior to Instrument analysis

SGS AXYS Analytical Services Ltd.

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
Mirex	37.98	272	270	52.1	
$^{13}C_{12}$-pp'-DDE	**29.21**	**330**	**328**	**78.2**	
o,p'-DDE	27.56	246	248	64	
p,p'-DDE	29.22	246	248	64	
$^{13}C_{12}$-PCB 138-REC[1]	**33.52**	**372**	**376**	**34**	
$^{13}C_{12}$-pp'-DDT	**33.32**	**247**	**249**	**64**	
o,p'-DDD	29.63	235	237	64	
p,p'-DDD	31.37	235	237	64	
o,p'-DDT	31.53	235	237	64.6	
p,p'-DDT	33.32	235	237	64	
$^{13}C_{12}$-PCB 52-REC[2]	**23.27**	**302**	**304**	**130**	
$^{13}C_{12}$-PCB 3	**15.03**	**200**	**202**	**31.9**	
PCB 1	13.63	188	190	32.1	
PCB 2	14.91	188	190	32.1	PCB 3
PCB 3	15.04	188	190	32.1	
$^{13}C_{12}$-PCB 8	**17.51**	**234**	**236**	**64**	
PCB 4/10	15.86	222	224	64.1	
PCB 7/9	16.84	222	224	64.1	PCB 8/5
PCB 6	17.28	222	224	64.1	PCB 8/5
PCB 8/5	17.52	222	224	64.1	
PCB 14	18.12	222	224	64.1	PCB 8/5
PCB 11	18.98	222	224	64.1	PCB 8/5
PCB-12/13	19.25	222	224	64.1	PCB 8/5
PCB 15	19.5	222	224	64.1	
$^{13}C_{12}$-PCB 28	**21.57**	**268**	**270**	**96**	
PCB 19	18.4	256	258	96.2	
PCB 30	18.77	256	258	96.2	PCB 18
PCB 18	19.4	256	258	96.2	
PCB 17	19.5	256	258	96.2	PCB 18
PCB 24/27	19.88	256	258	96.2	PCB 18
PCB 16/32	20.27	256	258	96.2	PCB 18
PCB 34/23	20.73	256	258	96.2	PCB 31
PCB 29	20.9	256	258	96.2	PCB 31
PCB 26	21.13	256	258	96.2	PCB 31
PCB 25	21.51	256	258	96.2	PCB 31
PCB 31	21.58	256	258	96.2	
PCB 28	22.03	256	258	96.2	
PCB 33/20/21	22.4	256	258	96.2	PCB 31

[1] REC = Recovery Standard, added just prior to Instrument analysis
[2] REC = Recovery Standard, added just prior to Instrument analysis

SGS AXYS Analytical Services Ltd.

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 22	22.69	256	258	96.2	PCB 31
PCB 36	23.12	256	258	96.2	PCB 31
PCB 39	23.58	256	258	96.2	PCB 31
PCB 38	24.08	256	258	96.2	PCB 37
PCB 35	24.45	256	258	96.2	PCB 37
PCB 37	21.5	256	258	96.2	
$^{13}C_{12}$PCB 31- FS[1]	**21.51**	**270**	**272**	**33.2**	**PCB 31**
$^{13}C_{12}$-PCB 138-REC[2]	**33.52**	**372**	**376**	**34**	
$^{13}C_{12}$-PCB 101	**27.69**	**338**	**340**	**64**	
PCB 54	20.91	290	292	129.9	
PCB 50	21.53	290	292	129.9	PCB 54
PCB 53	22.09	290	292	129.9	PCB 52/73
PCB 51	22.32	290	292	129.9	PCB 52/73
PCB 45	22.66	290	292	129.9	PCB 52/73
PCB 46	23.05	290	292	129.9	PCB 52/73
PCB 69	23.18	290	292	129.9	PCB 52/73
PCB 52/73	23.29	290	292	129.9	
PCB 49/43	23.49	290	292	129.9	
PCB 47/48/75	23.65	290	292	129.9	PCB 52/73
PCB 65/62	23.82	290	292	129.9	PCB 52/73
PCB 44	24.3	290	292	129.9	
PCB 42/59	24.4	290	292	129.9	PCB 44
PCB 72	24.73	290	292	129.9	PCB 44
PCB 41/71/64/68	24.92	290	292	129.9	PCB 44
PCB 40	25.31	290	292	129.9	
PCB 57	25.39	290	292	129.9	PCB 40
PCB 67	25.62	290	292	129.9	PCB 40
PCB 58	25.79	290	292	129.9	PCB 40
PCB 63	25.91	290	292	129.9	PCB 66/80
PCB 74/61	26.11	290	292	129.9	PCB 66/80
PCB 70/76	26.28	290	292	129.9	PCB 66/80
PCB 66/80	26.45	290	292	129.9	
PCB 55	26.9	290	292	129.9	PCB 56/60
PCB 56/60	27.32	290	292	129.9	
PCB 79	28.03	290	292	129.9	PCB 56/60
PCB 78	28.55	290	292	129.9	PCB 56/60
PCB 81	29.06	290	292	129.9	PCB 56/60
PCB 77	29.56	290	292	129.9	PCB 56/60

[1] FS = Field Standard
[2] REC = Recovery Standard, added just prior to Instrument analysis

SGS AXYS Analytical Services Ltd.

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 104	24.08	326	328	64.5	
PCB 96	25.14	326	328	64.5	PCB 95/93
PCB 103	25.34	326	328	64.5	PCB 95/93
PCB 100	25.62	326	328	64.5	PCB 95/93
PCB 94	26.03	326	328	64.5	PCB 95/93
PCB 98/102	26.39	326	328	64.5	PCB 95/93
PCB 95/93	26.51	326	328	64.5	
PCB 88/121	26.66	326	328	64.5	PCB 95/93
PCB 91	26.83	326	328	64.5	PCB 95/93
PCB 92	27.41	326	328	64.5	PCB 90/101/89
PCB 84	27.58	326	328	64.5	PCB 90/101/89
PCB 90/101/89	27.7	326	328	64.5	
PCB 113	27.88	326	328	64.5	PCB 90/101/89
PCB 99	27.97	326	328	64.5	
PCB 119	28.3	326	328	64.5	PCB 99
PCB 112	28.41	326	328	64.5	PCB 87/115/116
PCB 83/108	28.54	326	328	64.5	PCB 87/115/116
PCB 97/86	28.8	326	328	64.5	PCB 87/115/116
PCB 125	28.92	326	328	64.5	PCB 87/115/116
PCB 111/117	29	326	328	64.5	PCB 87/115/116
PCB 87/115/116	29.08	326	328	64.5	
PCB 85/120	29.28	326	328	64.5	PCB 87/115/116
PCB 110	29.58	326	328	64.5	
PCB 82	30.14	326	328	64.5	PCB 87/115/116
PCB 124	30.49	326	328	64.5	PCB 110
PCB 107/109	30.67	326	328	64.5	PCB 110
PCB 114	31.5	326	328	64.5	
PCB 122	31.63	326	328	64.5	PCB 114
PCB 105/127	32.3	326	328	64.5	
PCB 126	33.99	326	328	64.5	
$^{13}C_{12}$PCB 95- FS[1]	**26.52**	**338**	**340**	**64**	**PCB 95/93**
PCB 155	27.23	360	362	80.6	
PCB 150	28.33	360	362	80.6	PCB 149/139
PCB 152	28.71	360	362	80.6	PCB 149/139
PCB 145	29.04	360	362	80.6	PCB 149/139
PCB 148	29.26	360	362	80.6	PCB 149/139
PCB 136	29.41	360	362	80.6	PCB 149/139
PCB 154	29.64	360	362	80.6	PCB 149/139

[1] FS = Field Standard

SGS AXYS Analytical Services Ltd.

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 151	30.22	360	362	80.6	
PCB 144/135	30.46	360	362	80.6	PCB 149/139
PCB 147	30.64	360	362	80.6	PCB 149/139
PCB 149/139	30.84	360	362	80.6	
PCB 140	30.99	360	362	80.6	PCB 149/139
PCB 134/143	31.31	360	362	80.6	PCB 149/139
PCB 133	31.5	360	362	80.6	PCB 149/139
PCB 131/142	31.63	360	362	80.6	PCB 149/139
PCB 165	31.72	360	362	80.6	PCB 153
PCB 146	31.82	360	362	80.6	PCB 153
PCB 161	31.91	360	362	80.6	PCB 153
$^{13}C_{12}$-PCB 118	**30.92**	**338**	**340**	**64**	
PCB 123	30.8	326	328	64.5	PCB 118/106
PCB 118/106	30.92	326	328	64.5	
$^{13}C_{12}$-PCB 180	**37.05**	**406**	**408**	**95**	
PCB 153	32.12	360	362	80.6	
PCB 132/168	32.25	360	362	80.6	PCB 153
PCB 141	32.77	360	362	80.6	PCB 138/163/164
PCB 137	33.12	360	362	80.6	PCB 138/163/164
PCB 130	33.26	360	362	80.6	PCB 138/163/164
PCB 138/163/164	33.52	360	362	80.6	
PCB 158/160	33.66	360	362	80.6	PCB 138/163/164
PCB 129	33.95	360	362	80.6	PCB 138/163/164
PCB 166	34.27	360	362	80.6	PCB 138/163/164
PCB 159	34.44	360	362	80.6	PCB 138/163/164
PCB 162	34.71	360	362	80.6	PCB 138/163/164
PCB 128	34.93	360	362	80.6	PCB 138/163/164
PCB 167	35.01	360	362	80.6	
PCB 156	36.17	360	362	80.6	
PCB 157	36.46	360	362	80.6	
PCB 169	37.91	360	362	80.6	
PCB 188	31.77	394	396	95.2	
PCB 184	32.12	394	396	95.2	PCB 188
PCB 179	32.85	394	396	95.2	PCB 188
PCB 176	33.22	394	396	95.2	PCB 188
PCB 186	33.67	394	396	95.2	PCB 187/182
PCB 178	34	394	396	95.2	PCB 187/182
PCB 175	34.29	394	396	95.2	PCB 187/182
PCB 187/182	34.42	394	396	95.2	
PCB 183	34.68	394	396	95.2	

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Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 185	35.23	394	396	95.2	PCB 183
PCB 174/181	35.68	394	396	95.2	PCB 183
PCB 177	35.89	394	396	95.2	PCB 183
PCB 171	36.12	394	396	95.2	PCB 180
PCB 173	36.42	394	396	95.2	PCB 180
PCB 172/192	36.75	394	396	95.2	PCB 180
PCB 180	37.06	394	396	95.2	
PCB 193	37.19	394	396	95.2	PCB 180
PCB 191	37.39	394	396	95.2	PCB 180
PCB 170/190	38.22	394	396	95.2	
PCB 189	38.98	394	396	95.2	
$^{13}C_{12}$-PCB 153- FS[1]	**32.12**	**372**	**376**	**34.1**	**PCB 153**
$^{13}C_{12}$-PCB 202	**36.07**	**440**	**442**	**112**	
PCB 202	36.08	428	430	112.4	
PCB 201	36.48	428	430	112.4	PCB 204
PCB 204	36.58	428	430	112.4	
PCB 197	36.86	428	430	112.4	PCB 204
PCB 200	37.61	428	430	112.4	PCB 204
PCB 198	38.35	428	430	112.4	PCB 196/203
PCB 199	38.45	428	430	112.4	PCB 196/203
PCB 196/203	38.6	428	430	112.4	
PCB 195	39.37	428	430	112.4	PCB 194
PCB 194	39.9	428	430	112.4	
PCB 205	40.03	428	430	112.4	
$^{13}C_{12}$-PCB 206	**40.82**	**474**	**476**	**128**	
PCB 208	39.35	462	464	128.2	
PCB 207	39.56	462	464	128.2	PCB 208
PCB 206	40.82	462	464	128.2	
$^{13}C_{12}$-PCB 209	**41.51**	**512**	**510**	**117**	
PCB 209	41.51	500	498	117	

[1] FS = Field Standard

SGS AXYS Analytical Services Ltd.

Typical retention Times, Surrogates Used and RRF Determination for E2 Pesticides by GC/ECD

Compound Name	Typical Retention time (min)		Surrogate	RRF Determined From
	DB-5 column	DB-17 column		
delta-HCH	11.1	10.2	d_4-alpha-Endosulfan	delta-HCH
cis-Heptachlor Epoxide	15.6	13.1	d_4-alpha-Endosulfan	cis-Heptachlor Epoxide
alpha-Endosulfan	17.4	15.0	d_4-alpha-Endosulfan	alpha-Endosulfan
Dieldrin	18.9	17.0	d_4-beta-Endosulfan	Dieldrin
Endrin	20.2	19.5	d_4-beta-Endosulfan	Endrin
beta-Endosulfan	20.8	21.7	d_4-beta-Endosulfan	beta-Endosulfan
Endrin Aldehyde	22.1	24.4	d_4-beta-Endosulfan	Endrin Aldehyde
Endosulfan Sulfate	23.9	25.9	d_4-beta-Endosulfan	Endosulfan Sulfate
Endrin Ketone	27.5	32.5	d_4-beta-Endosulfan	Endrin Ketone
Methoxychlor	29.8	33.3	d_4-beta-Endosulfan	Methoxychlor
Labeled Surrogates			**Recovery Calculated Against**	
d_4-alpha-Endosulfan	17.3	14.9	$^{13}C_{12}$-PCB 153	
d_4-beta-Endosulfan	20.7	21.5	$^{13}C_{12}$-PCB 153	
Recovery Standard				
$^{13}C_{12}$-PCB 153	22.7	20.2		

Surrogates Used and RRF Determination for Technical Toxaphene by GC/LRMS

Compound Name	Typical Retention Time (min)	Quantification Ion (mz)	Confirmation Ion (mz)	RRF Determined from
Toxaphene Peak T1	30.2	159	161	Toxaphene Peak T1
Toxaphene Peak T2	31.3	159	161	Toxaphene Peak T2
Toxaphene Peak T4	34.1	159	161	Toxaphene Peak T4
Toxaphene Peak T5	35.1	159	161	Toxaphene Peak T5
Toxaphene Peak T6	35.5	159	161	Toxaphene Peak T6
Surrogate Standard				
^{13}C-PCB 159	34.4	372	374	^{13}C-PCB 159
Recovery Standard				
^{13}C-PCB 138	33.5	372	374	

SGS AXYS Analytical Services Ltd.

Analyte Ions Monitored, Surrogates Used, and RRF Determination for Toxaphene Congeners/Parlars by GC/LRMS (ECNI)

(No entry in the “RRF Used" field designates an RRF derived from that same compound.)

IUPAC	Parlar Suite (Lab Code)	Quantified against labeled standard	Typical Retention Time	RT Win. (sec)	mass1 (Qt)	mass2 (Cf)	%Cf/Qt ratio	Ion Ratio Limit (+/- %)
2-exo,3-endo,6-exo,8,9,10-HexaCB	Hex sed (2)	$^{13}C_{12}$-PCB-180	18.13	10	307	309	161	20
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	Hept sed (3)	$^{13}C_{12}$-PCB-180	19.13	10	341	343	192	20
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	(Peak 5)	$^{13}C_{12}$-PCB-180	21.31	10	341	343	192	20
2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	Parlar 32 (6)	$^{13}C_{12}$-PCB-180	21.79	10	341	343	192	20
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	(Peak 7)	$^{13}C_{12}$-PCB-180	22.03	10	341	343	192	20
2-endo,3-exo,6-exo,8,9,10,10-HeptaCB	(Peak 9)	$^{13}C_{12}$-PCB-180	23.54	10	341	343	192	20
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	Parlar 26 (4)	$^{13}C_{12}$-PCB-180	20.48	10	375	377	222	20
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	Parlar 39 (10)	$^{13}C_{12}$-PCB-180	23.76	10	375	377	222	20
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	Par 40/41 (11,12)	$^{13}C_{12}$-PCB-180	24.14	10	375	377	222	20
2,2,5-endo,6-exo,8,8,9,10-OctaCB	Parlar 42a (13)	$^{13}C_{12}$-PCB-180	24.39	10	375	377	222	20
2-exo,5,5,8,9,9,10,10-OctaCB	Parlar 44 (14)	$^{13}C_{12}$-PCB-180	24.75	10	375	377	222	20
2,2,5-endo,6-exo,8,9,10,10-OctaCB	(Peak 15)	$^{13}C_{12}$-PCB-180	26.08	10	375	377	222	20
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	(Peak 18)	$^{13}C_{12}$-PCB-180	26.99	10	375	377	222	20
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	Parlar 50 (16)	$^{13}C_{12}$-PCB-180	26.24	10	411	413	112	20
2,2,3-exo,5,5,9,9,10,10-NonaCB	(Peak 17)	$^{13}C_{12}$-PCB-180	26.84	10	411	413	112	20
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	Parlar 56 (19/20)	$^{13}C_{12}$-PCB-180	27.90	10	411	413	112	20
2,2,3-exo,5,5,8,9,10,10-NonaCB	Parlar 58 (21)	$^{13}C_{12}$-PCB-180	28.44	10	411	413	112	20
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	Parlar 59	$^{13}C_{12}$-PCB-180	28.72	10	411	413	112	20
2,2,5,5,8,9,9,10,10-NonaCB	Parlar 62 (22)	$^{13}C_{12}$-PCB-180	29.50	10	73	71	157	20
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	Parlar 63	$^{13}C_{12}$-PCB-180	29.93	10	411	413	112	20
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	Parlar 69	$^{13}C_{12}$-PCB-180	32.53	10	445	447	128	20
Labeled Compounds								
$^{13}C_{12}$-PCB-180		13C-PCB 138	28.28	20	406	408	91.9	20
$^{13}C_{12}$-PCB-138			24.89	20	372	370	78.7	20

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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QC Acceptance Criteria for Analysis of E2 Pesticides by GC/ECD

Analyte	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS						Procedural Blank Level ng	Acceptable Matrix Spike in matrices except tissue % Recovery [1]	Acceptable Matrix Spike in tissue % Recovery [1]
	Solids ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng			
Delta-HCH	0.1	1	0.1	0.1	1	1	<1	60-130	60-130
cis-Heptachlor Epoxide	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
alpha-Endosulfan	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Dieldrin	0.1	1	0.1	0.1	1	1	<1	60-130	65-130
Endrin	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
Endosulfan sulfate	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Endrin ketone	0.1	1	0.1	0.1	1	1	<1	60-130	65-130
beta-Endosulfan	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Endrin Aldehyde	0.1	1	0.1	0.1	1	1	<1	60-130	Note [2]
Methoxychlor	0.5	5	0.5	0.5	5	5	<5	60-130	60-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 column			
Final Vol, µL	100	100	100	100	100	100			

[1] Recoveries quoted are guidelines only and vary according to matrix. Consult detailed method performance data available with method documentation for specific criteria.

[2] Endrin aldehyde in tissue samples is reported as an estimated "Information Value" – recovery limits do not apply

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
d_4-alpha-Endosulfan	40-130
d_4-beta-Endosulfan	40-130

QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Instrument Sensitivity	S/N ratio ≥3:1 for 4 pg methoxychlor.
Instrument Linearity	Linearity is demonstrated by at least a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% (delta-HCH may be 25%)
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e.,the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF: Continuing Cal Ver	RRFs from opening/closing calibration standards must be within ±25% of the mean RRFs from initial calibration.
Chromatogram Quality (GC Resolution)	1. Valley height between d_4-alpha-Endosulfan and alpha-Endosulfan and between d_4-beta-Endosulfan and beta-Endosulfan must be ≤50% for equal concentrations. 2. Endrin (or $^{13}C_{12}$-endrin) breakdown must be ≤20%.
Analyte /Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.

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QC Acceptance Criteria for Analysis of E1 Pesticides by GC/MS

Analyte	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS (SDL) *						Procedural Blank Level ng	Acceptable Matrix Spike in all matrices except tissue % Recovery [1]	Acceptable Matrix Spike in tissue % Recovery [1]
	Solid ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng			
Dichlorobenzenes	1	10	1.0	1.0	10	10	<10	Note 1	Note 1
Trichlorobenzenes	1	10	1.0	1.0	10	10	<10	Note 1	Note 1
Tetrachlorobenzenes	0.5	5	0.5	0.5	5	5	<5	Note 1	Note 1
Pentachlorobenzene	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Hexachlorobenzene	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
alpha-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
beta-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
gamma-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
Heptachlor	0.5	5	0.5	0.5	5	5	<5	70-130	70-130
Aldrin	0.5	5	0.5	0.5	5	5	<5	70-130	70-130
Oxychlordane	0.5	5	0.5	0.5	5	5	<5	60-130	70-130
Octachlorostyrene	0.5	5	0.5	0.5	5	5	<5	60-130	70-130
trans-Chlordane	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Chlordane	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDE	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDE	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
trans-Nonachlor	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Nonachlor	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDD	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDD	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDT	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDT	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Mirex	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Technical Toxaphene (determined as present by the detection of a component)	15	150	15	15	150	150	<150	60-130	60-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 column			
Final volume, µL	100	100	100	100	100	100			

* SDLs listed are estimates based on typical sample matrix type and may be higher depending on particular sample characteristics

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SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES	
$^{13}C_6$-1,4-Dichlorobenzene	Note 1	
$^{13}C_6$-1,2,3-Trichlorobenzene	Note 1	
$^{13}C_6$-1,2,3,4-Tetrachlorobenzene	Note 1	
$^{13}C_6$-Pentachlorobenzene	30-130	
$^{13}C_6$-Hexachlorobenzene	30-130	
$^{13}C_6$-beta-HCH	30-130	
$^{13}C_6$-gamma-HCH	40-130	
$^{13}C_{10}$-Heptachlor	30-130	
$^{13}C_{10}$-Aldrin	30-130	
$^{13}C_{10}$-trans-Chlordane	30-130	
$^{13}C_{10}$-trans-Nonachlor	30-130	
$^{13}C_{12}$-p,p'-DDE	40-130	
$^{13}C_{12}$-p,p'-DDT	40-130	
$^{13}C_{12}$-PCB 159	40-130	(only when technical toxaphene is analyzed)

QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Procedural Blank	See above table or <10% of analyte value
Instrument Sensitivity	S/N ≥3:1 for 10 pg HCB, for 10 pg p,p'-DDT and for 20 pg oxychlordane. S/N ≥2:1 for 2.5 ng of Technical Toxaphene with a minimum of 4 peaks detected
Instrument Linearity	For a minimum 5-point calibration, a relative standard deviation of the RRFs ≤20% for all compounds, except for $^{13}C_{12}$-pp'-DDT where RSD of RRF ≤25%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e., the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF: Continuing CAL Ver	RRFs for all compounds from opening/closing calibration standards must be within ±20% of the mean RRFs from the initial calibration.
Chromatogram Quality **Max Peak Width:** **Resolution:**	1. Peak width at half height for p,p'-DDT is 5 sec. 2.Valley height between p,p'-DDD and o,p-DDT must be less than 10% the height of the peaks 3. PCB 209 peak must be symmetrical with negligible tailing, ≤20 sec. 4. p,p'-DDT breakdown must be ≤15%.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window for target compounds	RRT must be within ±3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labeled surrogate) Authentic compound must elute after its labeled analogue

Note 1: Recovery of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These compounds may be offered as "compounds of opportunity", reportable only when recoveries are adequate for quantification. Formal recovery acceptance limits have not been established.

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QC Acceptance Criteria for Analysis of PCB Congeners by GC/MS

Congener (IUPAC)	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS [1]						Procedural Blank Level ng	Acceptable Matrix Spike in matrices except tissue % Recovery	Acceptable Matrix Spike in tissue % Recovery
	Solids ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng/col			
Monochlorinated (1-3)	0.5	5	0.5	0.5	5	5	<1	60-130	70-130
Dichlorinated (4-15)	0.5	5	0.5	0.5	5	5	<1	60-130	70-130
Trichlorinated (16-39)	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
Tetrachlorinated (40-81)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Pentachlorinated (82-127)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Hexachlorinated (128-169)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Heptachlorinated (170-193)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Octachlorinated (194-205)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Nonachlorinated (206-208)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Decachlorinated (209)	0.5	5	0.5	0.5	5	5	<1	70-130	70-130
Aroclor Equivalents [2]	1	10	1	1	10	10	<1		
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 col			
Final volume, µL	100	100	100	100	100	100			

[1] Sample specific detection limits may vary depending on sample characteristics
[2] Aroclor formulations are not included in the batch QC OPR sample. Limit for biennial (every two years) matrix spikes with Aroclor formulations is 50-150% recovery.

SURROGATE STANDARD	% RECOVERY RANGES (All Matrices)
$^{13}C_{12}$-PCB 3	15-130
$^{13}C_{12}$-PCB 8	20-130
$^{13}C_{12}$-PCB 28	40-130
$^{13}C_{12}$-PCB 101	40-130
$^{13}C_{12}$-PCB 118	40-130
$^{13}C_{12}$-PCB 180	40-130
$^{13}C_{12}$-PCB 202	40-130
$^{13}C_{12}$-PCB 206	40-130
$^{13}C_{12}$-PCB 209	40-130

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QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean (applicable to concentrations >10 times the DL).
Procedural Blank	See above or <10% of analyte value.
Matrix Spike Recovery	See above; PCB 19 must be greater than 55%; PCB 104 must be greater than 60%.
Instrument Sensitivity	S/N ratio ≥3:1 for 10 pg PCB 118.
Instrument Linearity	Linearity is determined by at least a 5-point calibration with a relative standard deviation of the RRFs ≤20%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e., the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF Continuing CAL VER	RRFs from opening/closing calibration standards must be within ±20% of the mean RRFs from the initial calibration for all compounds.
Chromatogram Quality **Max. Peak Width:** **Resolution:**	1. PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds. 2. Valley height must be ≤80% of smallest peak height of PCB 28/31 pair.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window for target compounds	RRT must be within ±3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labeled surrogate). Authentic compound must elute after its labeled analogue.

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QC Acceptance Criteria for Analysis of Toxaphene congeners/Parlars by GC/HRMS (ECNI)

- Concentration of target analytes in the procedural blanks must be less than 10 ng/sample.
- The percent recovery of the surrogate standards must be between 40 - 130%.
- The percent recovery of toxaphene congeners/Parlars in a spiked reference sample must be between 50 - 150%.
- The difference between target concentrations for MS/MSD samples (where performed) should be less than 40%.

QC Acceptance Criteria for Optional Analysis of Coplanar PCBs by GC/HRMS

Analyte:	ACCEPTABLE DETECTION LIMITS					Procedural Blank Level pg	Acceptable Matrix Spike % Recovery	
	Solid pg/g	Aqueous pg/L	Tissue pg/g	Pulp pg/g	XAD Column pg/col		GC/LRMS	GC/HRMS
3,3',4,4'-TCB (#77)	0.2	2	0.2	0.2	2	<3	70-130	70-130
3,3'4,4',5-PCB (#126)	0.2	2	0.2	0.2	2	<2	70-130	70-130
3,3'4,4'5,5'-HCB (#169)	0.2	2	0.2	0.2	2	<3	70-130	70-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 col			
Final Vol, µL	20	20	20	20	20			

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
^{13}C-3,3',4,4'-TCB (#77)	40-130
^{13}C-3,3',4,4',5-PCB (#126)	40-130
^{13}C-3,3',4,4',5,5'-HCB (#169)	40-130

GC Parameter	Specification
ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
PROCEDURAL BLANK:	All analytes must be <3 pg or ≤1% of sample's analyte values.
INSTRUMENT SENSITIVITY:	S/N ratios should be ≥3:1 for 0.05 pg of 3,3',4,4',5,5'-HxCB injected.
INSTRUMENT LINEARITY:	Linearity determined by at least a 5-point calibration over the range of 10 pg/µL to 1000 pg/µL if all coplanar PCBs with an RSD of the RRFs are within ±20%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to ±20% over a 12-hour period.
RRF: Cal Ver	RRFs from opening/closing calibration standards must be within ±25% of the mean RRFs from the initial calibration.
CHROMATOGRAM QUALITY:	All chromatograms visually inspected but the 3,3',4,4',5,-PCB peak is examined specifically.
Max Peak Width:	15 sec for PCB 169
Resolution:	Visually acceptable
ANALYTE/SURROGATE RATIOS:	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.

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APPENDIX: SUMMARY OF KEY ATTRIBUTES OF METHODS MLA-007, EPA 8270C/D, EPA 8081A/D, EPA 625 AND EPA 608

Analysis by GC/LRMS, Key Attributes of SGS AXYS MLA-007, EPA 8270C/D and EPA 625				
	MLA-007	**EPA 8270C**	**EPA 8270D**	**EPA 625**
MS acquisition mode	SIM	Full Scan or optional SIM [1]	Full Scan or optional SIM [1]	Full Scan or alternate SIM [1]
Qualitative Identification Criteria	Retention time & ratio of 2 ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions
MS Ion Ratio Criteria	Within 20 % of theoretical	Within 30 % of reference spectrum	Within 30 % of reference spectrum	Within 20 % of reference spectrum
MS Tuning Type and Check Frequency	PTFBA, daily	DFTTP [1], 12 hrs	DFTTP [1], 12 hrs	DFTTP [1], 12 hrs
Quantification References	Isotopically Labeled Standards added prior to extraction	Internal Standards added just before instrumental analysis	Internal Standards added just before instrumental analysis	Internal Standards added just before instrumental analysis
Recovery correction of results	YES	NO	NO	NO
Calibration, minimum # levels	CCV Procedure: 5 levels OPTIONAL Single Point BRACKETING: 1 level	5	5	3
Initial Calibration Limit (% rsd)	20 % (DDT is 25%)	15 %	20 %	35 %
Calibration Verification Frequency	Initially and every 12 hrs	Initially and every 12 hrs	Initially and every 12 hrs	Daily
Calibration Verification Relative Response Limit (% diff.)	CCV Procedure: < 20 % of I-CAL OPTIONAL BRACKETING Procedure based on runs before and after samples: <20%	< 20 % of I-CAL	< 20 % of I-CAL	< 20 % of I-CAL
Calibration Verification IS area (% of I-CAL midpoint)	50-200 %	50-200 %	50-200 %	n.a.
Calibration verification IS RT (diff. from I-CAL midpoint)	n.a.	30 sec.	30 sec.	n.a.
Extraction	DCM, L/L (aqueous), DCM, sox. (solids)	Options specified externally	Options specified externally	DCM, L/L (aqueous), pH >11 or pH other [3]

Notes: [1] SIM acquisition is permitted option in EPA8270 for high sensitivity applications
[2] Based on availability, use of less ions is permitted where necessary
[3] Alternate procedures are permitted under Federal Register, Vol. 77 Issue 97 (May 18, 2012) Part 136.6

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Analysis by GC/ECD, Key Attributes of SGS AXYS MLA-007, EPA 8081A/B and EPA 608

	MLA-007	EPA 8081A	EPA 8081B	EPA 608
Sample Preservation and Storage	0 – 4ºC, dark No preservation required	≤6ºC $Na_2S_2O_3$ if chlorinated	≤6ºC $Na_2S_2O_3$ if chlorinated	4ºC Adjust pH 5-9 if not extracted by 72 hrs
Sample/Extract Hold Time	7 days / not defined	7 / 40 days	7 / 40 days	7 / 40 days
GC Columns	Capillary	Capillary	Capillary	Packed columns, OPTIONAL capillary columns permitted
Detection	ECD	ECD	ECD	ECD
Qualitative Identification Criteria	Relative retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute Retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute Retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute Retention times. Confirmation by dual column GC/ECD or by GC/MS
Quantification Technique and References	Combination of isotope dilution and internal standard added prior to extraction	External standard or optional internal standard prior to instrument	External standard or optional internal standard prior to instrument	External standard or optional internal standard prior to instrument
Recovery correction of results	YES	NO	NO	NO
Calibration, minimum # levels	CCV Procedure: 5 levels OPTIONAL Single Point BRACKETING: 1 level	5	5	3
Initial Calibration Limit (% rsd)	20 %	20 %	20 %	10 % otherwise use regression
Calibration Verification Frequency	Initially, every 12 hrs, every 20 samples before and after samples	Initially, every 12 hrs, every 20 samples before and after samples	Initially, every 12 hrs, every 20 samples, before and after samples	Daily
Calibration Verification Relative Response Limit (% diff.)	< 20 % of I-CAL (d-HCH is 25%)	< 15 % of I-CAL	< 20 % of I-CAL	< 15 % of I-CAL
Extraction	DCM, L/L stirring DCM, Soxhlet	Options specified externally include DCM L/L sep. funnel and DCM Soxhlet	Options specified externally include DCM L/L sep. funnel and DCM Soxhlet	DCM L/L sep. funnel
Clean-up	Florisil	Not specified	Not specified	Florisil sulfur removal
Spiked Sample Requirement	Optional by contract	Not specified	Not specified	10% at 1-5 times sample conc.
QC spike frequency	1 per batch or 5%	Not specified	Not specified	When spiked sample test fails
IPR acceptance limit range	N/A	Not specified	Not specified	Varying
OPR acceptance limit range	60 – 130 %, or narrower	Not specified	Not specified	EPA 608 limits are wider than MLA-007

SGS AXYS ANALYTICAL SERVICES LTD.

SGS AXYS METHOD MLA-007: ANALYTICAL METHOD FOR THE DETERMINATION OF

PCB AROCLORS, TOTAL PCBs and PCB CONGENERS CHLORINATED PESTICIDES, CHLOROBENZENES TECHNICAL TOXAPHENE TOXAPHENE CONGENERS/PARLARS

SCOPE

This document describes the analytical procedures for the quantitative determination of any of the following analytical compound groups or combinations thereof:

- Individual PCB congeners
- PCBs as Aroclor equivalents, determined by weighted summing of individual PCB congeners
- Total PCBs by congener group, determined by summing of individual PCB congeners
- Total PCB, determined by summing of individual PCB congeners
- Chlorinated pesticides, including Technical Toxaphene and Chlorobenzenes
- Toxaphene as individual congeners/Parlars

The method covers the following matrices: Solids (sediment/soil/sludge/ash/pulp), particulate filters, tissue (including blood and milk), aqueous samples, XAD-2 columns (resin and filters), ambient air samples (PUF and filter), and solvent extracts.

Samples are spiked with isotopically labeled surrogate standards or internal standards prior to analysis. Samples are extracted by solid-liquid extraction, liquid-liquid extraction or Soxhlet extraction, fractionated by column chromatography, and analyzed by gas chromatography with quadrupole (low-resolution) mass spectrometric detection (GC/MS) and electron capture detection (GC/ECD).

This method may be used for analysis of samples where USEPA Methods 608, 625, 1625B, 8081A, 8081B, 8270C, 8270D, or Method SM18 6630B have been requested **provided that the modifications described in this document are permitted by contract**. Key attributes of methods MLA-007, EPA 8270C/D, EPA 625, EPA 608 and EPA 8081A/B are summarized in Appendix D.

Approved 16-May-2017: John Cosgrove, Vice President and Senior Technical Director
Shea Hewage, Director of Operations
Dale Hoover, QA Manager

REFERENCES

1. USEPA Method 608, Organochlorine Pesticides and PCB's.

2. USEPA Method 625, Acids and Base/Neutrals.

3. USEPA Method 1625B, Semivolatile Organic Compounds by Isotope Dilution GC/MS.

4. USEPA Method 8081A, 8081B, Organochlorine Pesticides by Gas Chromatography.

5. USEPA Method 8270C, 8270D, Semivolatile Organic Compounds by GC/MS.

6. SM18, Part 6630B, Liquid-Liquid Extraction Gas Chromatographic Method Smith, L.M. "Carbon Dispersion Glass Fibers as An Adsorbent for Contaminant Enrichment and Fractionation", Anal. Chem. (1981), **53**, p. 2154-56.

7. Stalling, D.L., Smith, L.M., Petty, J.D. "Approaches to Comprehensive Analyses of Persistent Halogenated Environmental Contaminants", Measurement of Organic Pollutants in Water and Wastewater", ASTM STP 686 C.E. Van Hall, Ed.; American Society for Testing Materials, 1979, p 302-323.

CHANGES FROM PREVIOUS REVISIONS OR VERSIONS

The table below lists the details of the changes from Revision 13 Version 08 of this document (May 2017).

Page	Change Details
General	Clarified that the heptachlor epoxide isomer analyzed by MLA-007 is cis-heptachlor epoxide (a.k.a. heptachlor epoxide, isomer B), CAS no. 1024-57-3. Removed all references to obsolete extraction standard TCMX (tetrachloro-m-xylene).
14-17	Section A: Added CAS numbers for most analytes.
15	Section A.3: Removed delta-HCH from the E1 compound list.
18, 74	Sections B and 8.13: Added information describing current practice: Interference affecting PCB 11,
39	Section 4: Replaced reference to obsolete SQA-014 with reference to SQA-001.
45	Section 5: Removed reference to obsolete form FWO-312; replaced it by references to FWO-356 and FWO-357.
62	Section 6: Clarified the final evaporation volumes of the solvent exchange done prior to cleanup.
64	Section 6.3.2: Moved the copper treatment to be performed just before the alumina columning.
71	Section 8.1: Clarified that the authentic compound must elute after its labeled analogue.
72	Section 8.4: Changed the formulas for calculation of Aroclor 1221 and Aroclor 1232 equivalent concentrations to avoid an interference that occurs with PCB 1.
91	Appendix B, section B4, Analysis: Corrected the column id and added reference to MLA-010.

The table below lists the details of the changes from Revision 13 Version 07 of this document.

Page	Change Details
18	Table 1: Added biosolids as a new matrix. Added footnote 5 pertaining to biosolids.
23	Section 3.1: Clarified a few items (spatulas, water bath temperature used with rotary evaporator, magnetic stirring bars).
23	Section 3.2: Corrected document error for the Soxhlet cleaning procedure.
29-31	Table 4: Added columns for the E1 sensitivity standard and the extra high level p,p'-DDE CAL.
32	Table 6: Added column for the E2 sensitivity standard.
38, 40, 41	Tables 7, 8 and 9: Clarified analysis duplicate and bracketing cal criteria by also providing these in terms of RPD and RSD, as applicable.
43	Section 5: Added references to a few worksheets.
65-67	Sections 7.1 and 7.2: Made separate headings for breakdown. Corrected that the injection volume is 1 µL or 2 µL for E1, and 1 µL for E2. Section 7.2: Restated the endrin breakdown check in terms of ^{13}C-labeled endrin instead of native endrin, to reflect current use of new breakdown standard prepared with labeled endrin. Updated the breakdown equation to reflect the change to use labeled endrin.

The table below lists the details of the changes from Revision 13 Version 06 of this document.

Page	Change Details
23	Section 3.2: Clarified cleaning procedure for Soxhlet apparatus including thimble.
29-32	Tables 4-6: Added “CS” based calibration solution names.
36	Section 4: Added SAR solution description.
46	Deleted obsolete section 5.1.4 “Accelerated Solvent Extraction (ASE) for Solids”
63-64	Added section 6.3.4 “Acid Wash in a Centrifuge Tube”. Internal supporting data in AXYS Product Development and Improvement Docs: AcidWashandRecovery.xls.

The table below lists the details of the changes from Revision 13 Version 05 of this document.

Page	Change Details
1, 94	Scope and Appendix D: Added EPA 608 to Scope and to table “Analysis by GC/ECD, Key Attributes of AXYS MLA-007, EPA 8081A/B and EPA 608”

The table below lists the details of the changes from Revision 13 Version 04 of this document.

Page	Change Details
24	Section 3.3.4: Updated the Toxaphene standard solution spiking volume to current practice which is 250 µL.
28	Corrected document error: Calibration solution levels for the coelution PCB 34/23 was doubled to account for that both congeners PCB 34 and PCB 23 are included.
32-34	Sections 3.4 and 3.5: Added shelf life information where applicable.
33	Section 3.5: Changed Silica to current supplier and type
34, 62	Sections 3.6.1 and 6.2.1: Rephrased the deactivation grade description of Florisil to allow for necessary cutpoint adjustments.
39-40	Table 8: Technical Toxaphene: Corrected the estimated SDLs to be 150 ng/sample; corrected the blank limit to be 150 ng/sample.
41	Table 9: Deleted the OPR recovery specifications for Aroclors from the routine batch QC. Revised the biennial (every 2 years) matrix spike limit for Aroclors to be 50-150% recovery.
49	Section 5.3.3: Corrected document error – the 10 cm filter paper must be solvent rinsed, not baked.
63	Section 6.3: Deleted redundant comment that additional cleanup is performed on request by an IA Chemist.
70	Section 8.4: Changed the congener to Aroclor 1221 conversion factor from 1.9 to 1.4 to reflect actual practice and adjusted the congener to Aroclor 1260 conversion factor from 7.1 to 6.0 based on historical and on-going method performance data.
87	Appendix B: Added reference to MLA-010 for cleanup column packing preparation.
94-95	Appendix D: Added comparative method extraction details

The table below lists the details of the changes from Revision 13 Version 03 of this document.

Page	Change Details
15	Section B “Interferences”: Added information about occasional heptachlor interference.
17, 18	Tables 1 and 2: Changed “frozen” sample storage conditions temperature to be -20°C. Changed hold time for waters (pesticides) to 7 days.
21	Section 3.1: Specified filter paper make and type.
23, 43, 45, 60, 64, 66	Sections 3.3.3, 5.1, 5.1.4, 6.2, 7.1 and 7.2: Added identification of a method default procedure versus an optional procedure.
32	Section 3.4: Specified glass fibre filter according to current practice. Glass Fibre Filter Bed - Added reference to SLA-108.
37, 38, 40	Tables 7, 8 and 9: Added spike recovery limits specific to tissue matrix.
38	Table 8: Increased the method acceptance blank limit for technical Toxaphene to be 100 ng (was 10 ng).
41, 50, 52	Sections 5, 5.4 and 5.5: Added reference to new SOP SLA-124 “Liquid-Liquid Extraction Supplemental Techniques”.
47-49	Section 5.3: Clarified that current practice for aqueous samples is that suspended solids are separated from aqueous phase by centrifugation, not by filtration. Added reference to SLA-124 Liquid-Liquid Supplemental Techniques. Aligned % suspended solids limits for aqueous samples to follow EPA 1668C (to be ≤1% and >1%, instead of <1% and ≥1%). Added reference to new SOP SLA-124 “Liquid-Liquid Extraction Supplemental Techniques”.
50	Section 5.4: Updated lipid determination description to current practice.
52	Section 5.5: Changed lipid determination instructions to refer to SLA-020 and deleted references to obsolete SLA-030 SOP
53	Section 5.6.2: Clarified current practice for drying of XAD-2 resin.
59	Section 5.9: Replaced procedure instructions with a reference to SLA-123 “Splitting of Sample Extracts”.
86	Section B.2.1: Updated Carbon/Celite procedures to current practice.
various	Renumbered some sections in accordance with updated method format standard.

The table below lists the details of the changes from Revision 13 Version 02 of this document.

Page	Change Details
68	Section 9.4 “Quantification of delta-HCH”: Clarified the identity of the recovery standards in those cases when delta-HCH is quantified from both fractions E1 and E2.
76	Table 11: Added typical retention times for E2 compounds on DB-5 and DB-17 columns.

The table below lists the details of the changes from Revision 13 Version 01 of this document.

Page	Change Details
11	Clarified that dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes are non-routine target compounds and must be specifically requested by clients if required.
34	Added details of recommended matrices for QC sample preparation
67	Deleted reference to internal standard quantification of selected E2 compounds
68, 76	Changed the quantification procedure to yield recovery corrected concentration results for all target compounds captured in the E2 fraction. The protocol for d-HCH was adjusted to require quantification from the E2 fraction only, the quantification reference for d-HCH and Heptachlor Epoxide was changed from ^{13}C-PCB 153 recovery standard to d4-alpha-Endosulfan surrogate standard, and the quantification reference for Dieldrin, Endrin, Endrin Aldehyde, Endrin Ketone and Methoxychlor was changed from ^{13}C-PCB-153 recovery standard to d4-beta-Endosulfan surrogate standard.

The table below lists the details of the changes from revision 12 of this document.

Page	Change Details
1, 2	Updated EPA method reference codes to include EPA 8270D and EPA 8081B
9, 34, 35	Added note that the recoveries of di- through tetrachlorobenzenes may be low due to volatilization during workup and that these compounds may be offered only as “compounds of opportunity”. Recovery acceptance limits for these compounds deleted.
35	Hexachlorobutadiene recovery specification deleted.
33	Table 7. I-CAL limit for d-HCH changed to 25 %RSD from 20 %RSD
35, 36	Tables 8 and 9: Clarified that the ± 3 sec. RT windows for E1 Pests and PCBs only apply to target compounds.
37	Added recovery specification for biennial (every 2 years) Aroclor formulation matrix spikes
47-48	Corrected blood/serum/plasma extraction protocol and lipid determination to current practice: ≥10 g sample size and 1:1:1:3 extraction solvent ratio.
55	Section 6.1.1: Clarified that gel permeation cleanup is mandatory for all tissue matrices.
56	Changed Florisil elution solvent mix to 10:90 DCM:Hexane.
56, 57, 61	Sections 6.2.1, 6.3.1 and 7.2: Increased the final extract volume of fraction E2 from tissue and solid samples to be 200 µL (instead of 100 µL).
59, 85	Added Appendix C - Instrumental mass calibration and resolution protocol
86	Added Appendix D - Summary of Key Attributes of MLA-007, EPA 8270C/D, EPA 8081A/B and EPA 625

The table below lists the details of the changes from revision 11 of this document.

Page	Change Details
10, 33	Changed endrin aldehyde in tissue matrix to be “Information Value” only.
13	Corrected document number for SOP “Sample Disposal” to be SAD-014.
24	Table 3, footnote 1: Corrected the aqueous authentic spiking solution to have 2 times higher concentration than the spiking solution for the other matrices. Footnote 2: Deleted.

Page	Change Details
29	Clarified approval for use of anhydrous sodium sulfate.

The table below lists the details of the changes from revision 10 of this document.

Page	Change Details
1	Edited "Scope" for clarity.
9-10	Deleted obsolete 'Analysis Codes' table and all associated text references.
11	Section 1.1: Added definitions for Technical Toxaphene and for Toxaphene congeners/ Parlars.
15	Clarified footnote 4.
19, 20, 34, 73	Section 3.3.1: Changed to use $^{13}C_{12}$-PCB-159 as a surrogate standard for Technical Toxaphene instead of $^{13}C_{12}$-PCB-180. Added conc. and spiking volume for this surrogate standard solution.
20, 22	Section 3.3.4 and Table 3: Corrected Toxaphene conc. in the separate Toxaphene authentic standard solution to be 10000 ng/mL. Specified typical spike volume to be 1000 µL.
21	Endrin breakdown solution: Added $^{13}C_{12}$-endrin (80 ng/mL) as an option. Corrected the endrin concentration to be 120 ng/mL.
22-24	Table 3: Corrected authentic levels of endrin aldehyde, most of the Chlorobenzenes and several PCBs. Corrected surrogate levels to be at target value 1000 ng/mL and also to be at the same level as the calibration standards in Table 4. Added recovery standard $^{13}C_{12}$-PCB-153. Added surrogate std $^{13}C_{12}$-PCB-159 for technical Toxaphene.
28	Added Table 5: Nominal concentrations of calibration standards for Technical Toxaphene. Table 6: Corrected endrin aldehyde to be at the same levels as endrin.
29	Section 3.4: Changed powdered anhydrous sodium sulfate to be J.T. Baker 12-60 mesh.
34-35	Table 8: Deleted delta-HCH E1 individual specification (delta-HCH is recovered predominately in the E2 fraction and is specified accordingly) Clarified that the Toxaphene entry in this table is Technical Toxaphene and not individual Toxaphene congeners/Parlars. Updated typical SDL for Technical Toxaphene to be 10 ng/sample. Added surrogate recovery specification for $^{13}C_{12}$-PCB-159 (to be 40-130%). Deleted OPR specification for GC/HRMS. Added clarifying footnote about SDL.
36	Table 9: Deleted OPR specification for GC/HRMS.
38	Updated list of applicable worksheets
43-45	Section 5.3: Corrected criterion for performing a separate analysis of the solids phase of an aqueous sample to be "≥1% suspended solids" instead of "visible particles". Replaced filtering procedures for aqueous samples with centrifugation procedures. Sections 5.3.2 and 5.3.3.1: Added a liquid-liquid magnetic stirring extraction option, validated in July 2009 by MLA-007 R10, as the default extraction option for aqueous samples.
47	Section 5.5: Clarified blood extraction procedures. Added separate instructions for lipid determination in 1) serum & plasma and 2) whole blood. Added instruction always to perform a Biobead cleanup on the E2 fraction of blood/serum extracts.
60	Section 7, table with typical GC/MS operating conditions: Deleted reference to obsolete Finnigan instrument. Corrected source temperature, added quadrupole temperature.
63	Section 8: Added that ion ratios for technical Toxaphene compounds are determined

Page	Change Details
	empirically.
73	Table 12: Corrected that the RRF for the surrogate standard is determined from the surrogate standard (and not from the target compounds). Added typical retention times.
74 ff	Appendix A: Clarified that the methodology is GC/LRMS.
75	Appendix A, Section A.1, Sensitivity: Changed the S/N ratio specification for Parlar 62 to be 5:1 (instead of 10:1).
76	Appendix A, Section A.1, Linearity: Changed the linearity range to be 25-5000 ng/mL.

The table below lists the details of the changes from revision 9 of this document.

Page	Change Details
40	Added reference to Accelerated Solvent (ASE) extraction for extraction of solids
75	Reinstated (from MLA-007 Rev 08) and updated analysis of Toxaphene congeners/Parlars by GC/MS-ECNI.

The table below lists the details of the changes from revision 8 of this document.

Page	Change Details
	General Changes Added references to SOPs for handling hazardous samples. Deleted analysis of Toxaphene by ECNI. Deleted photomirex, oxadiazon, and dacthal as targets. Added information about analysis total toxaphene concurrently with pesticides by EI GC/MS. Included the optional use of a layer of Na_2SO_4 instead of sand in thimble. Revised handling of extract after each matrix specific extraction to include a solvent exchange.
16-25	Section 3.3 Deleted reference to Technical Specialist for preparation on standards Revised Table 3 to include amount of standard added to sample. Corrected some concentrations in Table 4, specifically PCB 187/182 and TCMX. Added information about toxaphene calibration std and authentic std.
26	Section 3.4 Updated list of reagents. Revised baking temp from 300°C to a minimum of 300°C.
29	Section 4.0 Added requirement to add field standard to Blank, deleted this requirement for SPM. Deleted reference to specific clean matrices, information is in SLA-016.
31-32	Table 7. Revised linearity specification for labeled DDT. Added specs for hexachlorobutadiene and octachlorostyrene.
36	Section 5.1 Added note about drying wet sediments. Added volume of DCM for large Soxhlet.
40	Section 5.3 Revised volume of solvent to extract aqueous samples to 100 mL/litre of sample. Revised volume DCM to extract filtrate by overnight stirring to 100mL. Deleted toluene keeper and concentration step from treatment of filtrate extract (Option 1). Deleted option of air drying filter with particulate. Added determining amount of particulate by SS.
44	Section 5.5 Revised volume of hexane for sample >10 g to be a minimum of 50 mL. Deleted Biobead cleanup of extract.

Page	Change Details
46	Section 5.6 Completely rewritten to describe revised procedures, including sample handling procedures, XAD-2 drying procedures, water removal procedures. Deleted requirement to record volume of water collected in Dean-Stark.
52	Section 5.9 Revised rinses of extract container to 3 hexane rinses.
	Section 7.1 Added information about calibration for toxaphene analysis. Revised GC/MS program.
62	Section 8.0 Added retention time specifications for GC/ECD.
64	Section 9.5 Added this section to describe quantification of toxaphene.
65	Revised quantification of delta-HCH.
66	Table 9. Revised retention times as GC program has changed.
	Corrections made 14-Dec-2006 Pg 19: Revised amt. spiked into SPM for some chlorobenzenes. Pg 31: Revised blank level for trichlorobenzene. Page 32: Deleted PCB 38 as surrogate.

TABLE OF CONTENTS

LIST OF APPENDICES

LIST OF TABLES

INTRODUCTION

A. ANALYTES OF INTEREST

This document describes the determination of the following compounds:

A.1 PCBs as Aroclor Equivalents [1] PCBs as Congener Groups [2]

Compound	CAS No.
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242/1016	53469-21-9/12674-11-2
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Total monochloro-PCBs	27323-18-8
Total dichloro-PCBs	25512-42-9
Total trichloro-PCBs	25323-68-6
Total tetrachloro-PCBs	26914-33-0
Total pentachloro-PCBs	25429-29-2
Total hexachloro-PCBs	26601-64-9
Total heptachloro-PCBs	28655-71-2
Total octachloro-PCBs	55722-26-4
Total nonachloro-PCBs	53742-07-7
Total decachloro-PCBs	2051-24-3
Total PCBs	1336-36-3

[1] Aroclor equivalents are calculated by summing the concentrations of a suite of characteristic marker PCB congeners and multiplying by a congener to Aroclor conversion factor.

[2] Total PCBs are calculated as the sum of the individual PCB congener.

A.2 Chorobenzenes

Compound	CAS No.
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
1,3,5-Trichlorobenzene	108-70-3
1,2,4-Trichlorobenzene	120-82-1
1,2,3-Trichlorobenzene	87-61-6
1,2,3,5/1,2,4,5-Tetrachlorobenzene	634-90-2/95-94-3
1,2,3,4-Tetrachlorobenzene	634-66-2
Pentachlorobenzene	608-93-5
Hexachlorobenzene	118-74-1

Dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes are non-routine compounds and must be specifically requested by clients if required. The recoveries of these may be low due to loss through volatilization during the analytical work-up and they can be reported only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.

A.3 Pesticides

Compound	CAS No.
E1 Pesticides by GC/LRMS	
Hexachlorobenzene	118-74-1
alpha-HCH	319-84-6
beta-HCH	319-85-7
gamma-HCH	58-89-9
Heptachlor	76-44-8
Aldrin	309-00-2
Oxychlordane	27304-13-8
trans-Chlordane	5103-74-2
cis-Chlordane	5103-71-9
o,p'-DDE	3424-82-6
p,p'-DDE	72-55-9
trans-Nonachlor	39765-80-5
cis-Nonachlor	5103-73-1
o,p'-DDD	53-19-0
p,p'-DDD	72-54-8
o,p'-DDT	789-02-6
p,p'-DDT	50-29-3
Mirex	2385-85-5
Hexachlorobutadiene (upon request)	87-68-3
Octachlorostyrene (upon request)	29082-74-4
Technical Toxaphene (upon request)	8001-35-2
E2 Pesticides by GC/ECD	
cis-Heptachlor epoxide	1024-57-3
alpha-Endosulfan	959-98-8
delta-HCH	319-86-8
Dieldrin	60-57-1
Endrin	72-20-8
Methoxychlor	72-43-5
Endosulfan sulfate	1031-07-8
Endrin ketone	53494-70-5
beta-Endosulfan	33213-65-9
Endrin aldehyde [1]	7421-93-4

[1] Recovery of endrin aldehyde in tissue matrix may be very low and the accuracy of results are not defined. Endrin aldehyde results for tissue samples are reported as "Information Values" only and represent estimated concentrations.

A.4 PCB Congeners by GC/MS

This method is suitable for the analysis of all 209 PCB congeners. It is possible that loss of chlorine from a more highly chlorinated congener may increase the response and produce a false concentration for a less-chlorinated congener eluting at the same retention time. This effect may be significant for certain "toxic" congeners assigned TEF values by the World Health Organization; any concentrations reported for PCBs 77, 81, 123, and 126 should be interpreted as maximum values due to the potential for interference. There may be instances where response for other congeners is elevated; however, the effect in relation to the overall PCB content will be negligible.

A.5 Analysis of Toxaphene

A.5.1 Technical Toxaphene - (sum of major peaks)

Technical Toxaphene is an insecticide produced by chlorinating camphene mixtures. The product is estimated to contain between 600 to 900 chlorinated bornanes, or chlorinated camphene compounds. The Technical Toxaphene product sold by Hercules and others is used as the analytical reference standard for measurement of the total amount of Technical Toxaphene in environmental samples. The analysis is performed by GC/LRMS (EI), involves comparison of 5-7 major peaks in the Technical Toxaphene calibration standard to those same peaks detectable in samples, and results in a single value. No attempt is made to identify the individual components of the Toxaphene mixture. EPA 8081 is based on the measurement of Technical Toxaphene.

A.5.2 Toxaphene Congeners/Parlars (selected chlorobornanes, Parlar II Suite)

Toxaphene Congeners/Parlars consists of 600 to 900 individual chlorinated bornane or camphene components which can be referred to as congeners (the term 'Parlar' refers to a naming convention for a subset of congeners identified and isolated by the researcher Dr. Harun Parlar). The congeners may be part of the original Technical Toxaphene product or may result from degradation or dechlorination through a variety of processes. Toxaphene congener/Parlars analysis quantifies a number of specific congeners present in the greatest concentration in samples. Measurement of Toxaphene congeners/Parlars is typically accomplished through GC/LRMS (ECNI), such as EPA 8276 or GC/HRMS (ECNI) methods. The individual components determined are:

Compound		CAS No.
2-exo,3-endo,6-exo,8,9,10-HexaCB	Hex sed (2)	57981-29-0
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	Hept sed (3)	208049-58-5
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	(Peak 5)	163390-24-7
2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	Parlar 32 (6)	254969-81-8 51775-36-1
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	(Peak 7)	206360-10-3 254969-83-0
2-exo,5-exo,6-endo,8,9,10,10-HeptaCB (aka 2-endo,3-exo,6-exo,8,9,10,10-HeptaCB)	(Peak 9)	163390-25-8
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	Parlar 26 (4)	142534-71-2
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	Parlar 39 (10)	64618-67-3
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	Par 40/41 (11,12)	166021-27-8 165820-16-6
2,2,5-endo,6-exo,8,8,9,10-OctaCB	Parlar 42a (13)	58002-18-9
2-exo,5,5,8,9,9,10,10-OctaCB	Parlar 44 (14)	165820-17-7
2,2,5-endo,6-exo,8,9,10,10-OctaCB	(Peak 15)	64618-69-5
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	(Peak 18)	254969-88-5
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	Parlar 50 (16)	66860-80-8
2,2,3-exo,5,5,9,9,10,10-NonaCB	(Peak 17)	
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	Parlar 56 (19/20)	64618-70-8 253340-63-5 64618-71-9
2,2,3-exo,5,5,8,9,10,10-NonaCB	Parlar 58 (21)	165820-20-2
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	Parlar 59	155750-49-5
2,2,5,5,8,9,9,10,10-NonaCB	Parlar 62 (22)	154159-06-5
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	Parlar 63	151214-79-8
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	Parlar 69	151183-19-6

A.6 Optional Analysis of Coplanar PCBs

The following PCBs may be determined as an optional analysis, described in Appendix B at the end of this document.

PCB 81
PCB 77
PCB 126
PCB 169

B. CONTAMINATION OR INTERFERENCES

Potential interferences can come from almost all aspects of the analysis procedure. Care should be taken to ensure that all glassware is cleaned according to the specifications listed in Section 3.2.

PCB congeners and pesticides may be destroyed in the presence of acid and caution must be exercised to limit the exposure the sample and extract to acid if pesticide analysis is required.

An occasional interference has been observed as a peak eluting at approximately the same retention time as heptachlor. This interfering peak does not meet the ion ratio requirements for heptachlor. The exact source is unknown, tentative identification of the interference indicates that it is part of a suite of diterpene compounds of plant origin, similar in structure to kaurene. The interference results in an increase of the detection limit of heptachlor, and can be resolved by analyzing the extract using high resolution mass spectrometry.

The ^{13}C-gamma-HCH surrogate standard can produce a closely eluting, non-ratioing response interference affecting PCB 11. Care should be taken to differentiate PCB 11 from the interference.

Records of background levels within reference materials should be maintained in order to correct QC samples analyzed within the same batch.

Interferences may be resolved using the greater sensitivity and specificity of GC/HRMS (gas chromatography with high resolution mass spectrometry). GC/HRMS may be performed in particular situations after approval by the Project Manager.

C. SAFETY

Refer to SAF-001 *Safety Manual* for safety procedures. Refer to SLA-079 *Agricultural Hazard protocols for Soils* and SLA-082 *Handling of Human Biohazardous Samples* for procedures for handling hazardous samples.

D. POLLUTION PREVENTION AND WASTE MANAGEMENT

SGS AXYS Analytical Services Ltd. complies with all federal, provincial and municipal regulations governing waste management, including land disposal restrictions and sewage discharge regulations. SGS AXYS' waste disposal procedures have been developed to comply with all pollution prevention regulations.

All standards are prepared in volumes consistent with volumes required by the method to minimize the disposal of standards.

Refer to SAF-001 *Safety Manual* and to SAD-014 *Sample Disposal* for procedures for disposing of laboratory wastes.

E. DEFINITIONS

Refer to QDO-001 *SGS AXYS Analytical Services QA/QC Policies and Procedures Manual* for definitions of terms used in this document.

F. METHOD PERFORMANCE

Method performance is validated through analysis of matrix-specific reference samples, including spikes (SPM's) and certified reference materials. Ongoing method performance is monitored through QC samples analyzed alongside samples. The parameters monitored include percent recovery of labeled surrogates, blank concentrations, native compound recoveries and percent difference between duplicate tests.

This method has been validated to demonstrate that it is fit for the intended use.

ANALYSIS PROCEDURES

1. SAMPLE PRESERVATION AND STORAGE

Sample receipt and storage information are summarized in Table 1. Where possible samples are stored frozen to minimize any degradation.

Where adherence to USEPA Method holding times is required the storage times summarized in Table 2 are applicable.

Table 1: PCB/Pesticide Analysis Sample Storage Requirements

Matrix	Sample Size (per analysis)	Sample Container [1]	Condition Upon Receipt	Storage Conditions [2]	Sample Hold Time Guideline [3]	Extract Holding Time	Preservation
Solids (Sediment/Soil/ Sludge)	10-15 g wet	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Biosolids [5]	Up to 2 g dry, but not more than 15 g wet	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Ash	5 g dry	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Particulate Filter Paper	whole filter	Glass or foil wrapped	< 4°C	-20°C, dark	1 year	not defined	none required
Pulp (dry)	20 g dry	Glass or foil wrapped	ambient	ambient	30 days [4]	not defined	none required
Pulp (wet)	N/A	Glass	0 – 4°C	4°C, dark	30 days [4]	not defined	none required
Tissue	5-10 g wet	Glass or foil wrapped	< 4°C	-20°C, dark	1 year	not defined	none required
Blood/Serum	5-20 g	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Milk	50 g	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Ambient Air Sample	PUF & Filter	Glass	< 4°C	-20°C, dark	1 year	not defined	none required
Aqueous Samples (Water/Effluent/Wet Sludge)	1 litre	Brown glass	0 – 4°C	4°C, dark	7 days [4]	not defined	none required
XAD-2 Columns	one column resin particulate filter(s) wound glass filter	As is Glass Foil wrapped Foil wrapped	0 – 4°C 0 – 4°C 0 – 4°C 0 – 4°C	4°C, dark 4°C, dark -20°C, dark -20°C, dark	30 days [4] 30 days [4] 1 year 1 year	not defined not defined not defined not defined	none required none required none required none required
Solvent Extract	one	Glass	< 4°C	-20°C, dark	1 year	not defined	none required

[1] All glass containers should be organically clean; i.e. purchased certified clean, baked or solvent-rinsed. All containers must be tightly sealed with screw cap lids (PTFE or foil-lined) to prevent loss of volatiles or contamination from volatiles. If samples are received in clear glass containers, they must be protected from the light.

[2] Storage temperatures quoted are nominal temperatures. Samples stored at a nominal temperature of 4°C are permitted a variance of ±2°C and samples stored at -20°C are permitted a variance of ±4°C

[3] Hold times quoted are recommended guidelines; there is no evidence to indicate that properly stored samples are not stable for longer periods of time. Client negotiated requests for specific holding times or other method-specific holding times are adhered to.

[4] 1 year if PCBs only are analyzed (hold time based on EPA Method 1668A recommendation).

[5] Significant judgment based on prior experience with particular samples and/or sample appearance may apply to sample size selection for biosolids due to the potential to overwhelm routine method clean-up capabilities.

Table 2: EPA Method Sample Storage Requirements

Analysis	Matrix	Sample Size	Container	Condition Upon Receipt	Storage Conditions[2]	Sample Holding Times[3]	Extract Holding Times	pH Adjustment	Residual Cl- Check
EPA 608 Chlorinated Pesticides	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Adjust to pH 5 – 9 if >72h to extr	Add $Na_2S_2O_3$ if +ve
EPA 625 Chlorinated Pesticides	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$ if +ve
EPA 1625C PAHs	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$
Chlorinated Hydrocarbons	Solid	2 – 30 g	Glass	4°C, dark	-20°C[4], dark	(14 days)	40 days	Not required	Not required
EPA 8081B Chlorinated Pesticides	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$
	Solid	2 – 30 g	Glass	4°C	-20°C[4], dark	(14 days)	40 days	Not required	Not required
EPA 8082A PCB's	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$
	Solid	2 – 30 g	Glass	0 - 4°C, dark	-20°C[4], dark	(14 days)	40 days	Not required	Not required
EPA 8270D PAHs, PCBs	Aqueous	1 L	Amber glass	0 - 4°C, dark	0 - 4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$
Pesticides Chlorinated Hydrocarbons	Solid	2 – 30 g	Glass	0 - 4°C, dark	-20°C[4], dark	(14 days)	40 days	Not required	Not required

[1] All glass containers should be organically clean; i.e. purchased certified clean, baked or solvent-rinsed. All containers must be tightly sealed with screw cap lids (PTFE or foil-lined) to prevent loss of volatiles or contamination from volatiles. If samples are received in clear glass containers, they must be protected from the light.

[2] Storage temperatures quoted are nominal temperatures. Samples stored at a nominal temperature of 4°C are permitted a variance of ±2°C. Samples stored frozen are stored a temperatures colder than -10ºC but no warmer.

[3] Storage times in brackets are times from sample collection. Storage times without brackets are times from Validated Time of Sample Receipt (VTSR) of sample by SGS AXYS Analytical Services.

[4] This sample storage temperature is lower than required in the specific EPA methods and is a more stringent requirement by SGS AXYS Analytical Services.

2. SAMPLE PRETREATMENT AND PREPARATION

Refer to the following standard operating procedures:

Document ID	Title
SLA-011	Compositing Samples
SLA-012	Dissection of Samples
SLA-013	Procedures for Homogenization of Solids and Tissues
SLA-014	Thawing Homogenized Samples Prior to Analysis
SLA-015	Moisture Determination
SLA-043	Removing Sampling Media from Field Sampling Equipment
SLA-084	Preparation of Aqueous Samples for Extraction
SLA-092	Determination of Suspended Solids Content in Aqueous Samples

Aqueous Samples

Refer to the standard operating procedures SLA-084 “Preparation of Aqueous Samples for Extraction”.

Whole Blood/Blood Serum/Blood Plasma

Allow blood sample to thaw prior to subsampling for analysis following procedures in standard operating procedure SLA-014. Homogenize by shaking.

Milk

Allow milk to thaw prior to homogenization and subsampling for analysis following procedures in standard operating procedure SLA-014. Homogenize the sample by shaking well.

Particulate Filter Papers (Wet)

Allow particulate filter papers to partially thaw. Fold the filter paper to fit into the soxhlet thimble for extraction.

Ambient Air (PUF and Filter)

If received in the sampling apparatus, use solvent rinsed forceps to withdraw the PUF plug from the sampler. The associated filter is typically received in a petri dish or wrapped in foil. Use solvent rinsed forceps to handle the filter.

Pulp and Sludge

Homogenize dry pulp and sludge samples according to the procedures described in SLA-013. Determine moisture content of dry pulp and sludge samples following the procedure

described in SLA-015. Consult with Lab Supervisor for treatment of wet pulp or sludge samples.

Sediment/Soils/Ash/Fly ash

Thaw the sample prior to homogenization and subsampling procedures following the procedures described in SLA-014. Homogenize the entire sample before subsampling for analysis as described in SLA-013.

Determine moisture content on an accurately weighed subsample (1–2 g, depending upon sample available) as described in SLA-015.

Tissue

Thaw the sample prior to homogenization and subsampling following the procedures described in SLA-014. If the tissue sample is received as a homogenate, stir the homogenate well with a spatula prior to subsampling. Otherwise, homogenize tissue samples prior to analysis following the procedures described in SLA-013.

If required, determine moisture content of an accurately weighed subsample of (1 g) as described in SLA-015.

XAD-2 Columns and Filters

Refer to the following standard operating procedure:

Document ID	Title
SLA-043	Removing Sampling Media from Field Sampling Equipment

for details of removing the resin from the column. Filters are partially thawed prior to extraction.

3. MATERIALS AND REAGENTS

Refer to the following standard operating procedures:

Document ID	Title
SLA-001	Cleaning Laboratory Items
SLA-002	Glassware and Laboratory Equipment Proofs
SLA-009	Preparation of Standards
SLA-018	Solvent Rinsing of Glassware for Organic Analysis
SLA-019	Solvent Proofs
SLA-022	Use of Drying Ovens and Muffle Furnace
SLA-023	Use of Balances
SLA-036	Cleaning of GC/MS and GC/ECD Microvials
SLA-041	Reagent Preparation
SLA-044	Activation of Copper Foil, Turnings and Powder
SLA-093	Baking of Anhydrous Sodium Sulfate
SQA-009	Storage and Control of Standards
SQA-003	Standard Solution Validation

3.1 Equipment List

Apparatus and materials used for the preparation of samples for analysis are listed below. Any brand names used are for example only, and can be substituted with other equipment with equivalent or better specifications.

Extraction

Separatory funnels (125 mL, 250 mL, 500 mL, 1000 mL)
Erlenmeyer flasks (250 mL, 500 mL, 1000 mL)
Round bottom flasks (100 mL, 125 mL, 250 mL, 500 mL, 1000 mL))
Beakers
Graduated Cylinders
Class A volumetric flasks, pipettes
Disposable pipettes
Disposable centrifuge tubes (15 mL)
Hamilton Syringes (5 µL, 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, 1000 µL)
Autosampler vials (amber glass, 800 µL)
Chromatography columns (1 cm x 12 cm, 3 cm x 50 cm, 1 cm x 25 cm)
Silanized glass wool
Filter paper – Ahlstrom, glass fibre filter, 161 grade, 1.1 µm, 42.5 mm (or equivalent)
Filter paper – Pall, glass fiber, A/E grade, 1.0 µm, 102 mm diameter (or equivalent)
PTFE tape

Aluminum Foil
Spatula – stainless steel
Disposable spoons
Rotary evaporator, with water bath at ≤30 °C
Magnetic stirring plate, with pre-cleaned PTFE coated magnetic stirring bars
Soxhlet apparatus w/heating mantle
Soxhlet/Dean Stark apparatus
Water bath capable of maintaining up to 50°C
Balance – Top loading and analytical (2-, 3-, & 4-place)
Glassware ovens
Drying ovens
Muffle furnace

Nitrogen source with manifold apparatus

Instruments

Agilent 6890N Gas Chromatograph or equivalent;
Agilent 5973N mass spectrometer or equivalent;
J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness);
J&W DB-17MS capillary column (30 m, 0.25 mm i.d., 0.25 µm film thickness) (optional)

3.2 Glassware

All glassware used in the preparation of reagents and in the analytical procedure must be organically clean. Glassware must be washed and baked using standard operating procedures. Baked glassware must be solvent rinsed with dichloromethane prior to use. Soxhlet glassware is rinsed with dichloromethane.

If baked glassware is not available, glassware must be washed and solvent rinsed following the procedures described in SLA-018, *Solvent Rinsing of Glassware*. If the glassware is to be used immediately, a final rinse is done using the solvent appropriate to the extraction procedure; otherwise the glassware is allowed to air dry, and capped with aluminum foil.

3.3 Preparation of Standard Solutions - GC/MS (E1) and GC/ECD (E2) Analysis

The analysis of less polar chlorinated pesticides, PCBs and chlorobenzenes by low resolution GC/MS and of the more polar pesticides by GC/ECD requires the use of the surrogate, recovery, and calibration standards described below. Details of the preparation, composition and validation of concentrations of these standards are described on Standard Data Sheets.

3.3.1 Surrogate Standard

Samples are spiked with a suite of isotopically labeled surrogate standards prior to extraction procedures. The surrogate standard solution is prepared to have the nominal concentrations of surrogates presented in Table 3. An aliquot of surrogate standard solution is added to each sample prior to extraction. Typically, an aliquot of 40 µL, equivalent to

approximately 40 ng of each standard, is added to the sample. The suite of surrogates added depends upon the analytes being analyzed (Table 3). The amount of surrogate added is dependent upon sample size, final extract volume, and concentration of analytes in the sample, and may be adjusted accordingly.

For the analysis of technical Toxaphene a separate surrogate standard solution containing 1000 ng/mL of $^{13}C_{12}$-PCB-159 is prepared. Typically an aliquot of 40 µL is added to samples that will be analyzed for Technical Toxaphene.

3.3.2 Recovery Standard

Samples extracts are spiked with isotopically labeled recovery standards prior to instrumental analysis. The recovery standard solutions are prepared to have the nominal concentrations of recovery standards presented in Table 3. Recovery standard solutions are prepared for both the GC/MS analysis and the GC/ECD analysis. An aliquot of recovery standard is added to the final extract just prior to instrumental analysis. The amount of recovery standard added is designed to match the amount of surrogate in the final extract.

3.3.3 Calibration Solutions

A series of five calibration solutions containing native analytes, labeled surrogates standards, and labeled recovery standards is used to establish linearity of the analytical instrument (default calibration procedure). The calibration solutions are prepared to have the nominal concentrations presented in Table 4 for PCB congeners and less-polar pesticides, Table 5 for Technical Toxaphene and Table 6 for the more polar pesticides. Calibration solutions are prepared for both the GC/MS analysis and the GC/ECD analysis. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level calibration solution is analyzed at least every 12 hours.

For the analysis of Technical Toxaphene, a separate series of calibration solutions containing Toxaphene (Absolute Standards Inc., catalogue no. 17218), labeled surrogate standard ($^{13}C_{12}$-PCB-159), and labeled recovery standard is used to establish linearity of the analytical instrument. A mid-level calibration solution is analyzed at least every 12 hours.

A calibration standard is prepared containing p,p'-DDE at a concentration of 10,000 ng/mL and surrogate and recovery standards at the same concentrations as the initial calibration standards. The standard is analyzed during the initial calibration, and used to demonstrate linearity in the response of p,p'-DDE up to five times the concentration of the highest calibration standard.

3.3.4 Authentic Spiking Standard

The authentic spiking solution is prepared to have the nominal concentrations presented in Table 3. An aliquot of authentic spiking solution is added to an unspiked reference material (USM) to prepare a spiked reference sample as a QC sample. Typically a 1000 µL aliquot (20

µL of a more concentrated solution for aqueous samples), equivalent to 40 ng of each target analyte, is added to the matrix.

An aliquot (typically 250 µL) of an authentic spiking solution containing Toxaphene (10000 ng/mL) is also spiked into the reference sample when this analyte is analyzed.

3.3.5 Retention Time Markers

A separate calibration standard containing all 209 PCB congeners and selected pesticides as well as isotopically labeled PCB's and pesticides is used as retention time (RT) markers is analyzed daily.

3.3.6 Solvent Blank

During the analysis of a batch of samples, a solvent blank is analyzed after the calibration solution to monitor carryover from the previous injection.

E1 Analysis: For the GC/MS analysis of pesticides, the solvent blank is prepared from toluene and contains $^{13}C_{12}$-DDT to monitor the breakdown of DDT in the injector.

E2 Analysis: A solution containing endrin (120 ng/mL) or $^{13}C_{12}$-endrin (80 ng/mL) is prepared in hexane. This solution is analyzed by GC/ECD at least once every 12 hours and is used to monitor the extent of endrin breakdown.

Table 3: Analysis of Chlorobenzenes, Pesticides and PCBs
Nominal Concentrations of Standard Solutions (ng/mL)

Compound Name	Nominal Conc[1]. in Authentic Std Solution (ng/mL)	Amt Added to each SPM Sample (ng)
1,3-dichlorobenzene	40	40
1,4-dichlorobenzene	40	40
1,2-dichlorobenzene	40	40
1,3,5-trichlorobenzene	40	40
1,2,4-trichlorobenzene	40	40
1,2,3-trichlorobenzene	40	40
1,2,3,5- /1,2,4,5-tetrachlorobenzene	80	80
1,2,3,4-tetrachlorobenzene	40	40
Pentachlorobenzene	40	40
Hexachlorobenzene	40	40
alpha-HCH	80	80
beta-HCH	80	80
gamma-HCH	80	80
delta-HCH	80	80
Heptachlor	40	40
Aldrin	80	80
trans-Chlordane	80	80
cis-Chlordane	80	80
trans-Nonachlor	80	80
cis-Nonachlor	80	80
o,p'-DDE	40	40
p,p'-DDE	40	40
o,p'-DDT	40	40
p,p'-DDT	40	40
Octachlorostyrene	40	40
Oxychlordane	80	80
o,p'-DDD	40	40
p,p'-DDD	40	40
Mirex	40	40
Dieldrin	40	40
alpha-Endosulfan	40	40
beta-Endosulfan	40	40
Endosulfan Sulfate	40	40
Endrin	40	40
Methoxychlor	40	40
Endrin Aldehyde	40	40
Endrin Ketone	40	40
cis-Heptachlor epoxide	40	40
Toxaphene (separate solution)	10000	10000

Table 3 (cont'd)

Compound Name	Nominal Conc.[1] in Authentic Std Solution (ng/mL)	Amt Added to Each SPM Sample (ng)	Compound Name	Nominal Conc.[1] in Authentic Std Solution (ng/mL)	Amt Added to Each SPM Sample (ng)
PCB 1	40	40	PCB 114	40	40
PCB 3	40	40	PCB 118	40	40
PCB 4	40	40	PCB 123	40	40
PCB 8	40	40	PCB 126	40	40
PCB 15	40	40	PCB 138	40	40
PCB 18	40	40	PCB 149	40	40
PCB 19	40	40	PCB 151	40	40
PCB 28	40	40	PCB 153	40	40
PCB 31	40	40	PCB 155	40	40
PCB 34/23	80	80	PCB 156	40	40
PCB 37	40	40	PCB 157	40	40
PCB 40	40	40	PCB 167	40	40
PCB 44	40	40	PCB 169	40	40
PCB 49	40	40	PCB 170	40	40
PCB 52	40	40	PCB 180	40	40
PCB 54	40	40	PCB 183	40	40
PCB 56	40	40	PCB 187/182	80	80
PCB 66	40	40	PCB 188	40	40
PCB 77	40	40	PCB 189	40	40
PCB 81	40	40	PCB 194	40	40
PCB 87	40	40	PCB 196	40	40
PCB 95	40	40	PCB 202	40	40
PCB 99	40	40	PCB 205	40	40
PCB 101	40	40	PCB 208	40	40
PCB 104	40	40	PCB 206	40	40
PCB 110	40	40	PCB 209	40	40
PCB 105	40	40			

Table 3 (cont'd)

Labeled Surrogates	**Nominal Conc.[1] in Surrogate Std Solution (ng/mL)**	**Amt Added to Each Sample (ng)**
$^{13}C_6$-1,4-dichlorobenzene [2]	1000	40
$^{13}C_6$-1,2,3-trichlorobenzene [2]	1000	40
$^{13}C_6$-1,2,3,4-tetrachlorobenzene [2]	1000	40
$^{13}C_6$-Pentachlorobenzene [2]	1000	40
$^{13}C_6$-Hexachlorobenzene [2]	1000	40
$^{13}C_6$-beta-HCH [2]	1000	40
$^{13}C_6$-gamma-HCH [2]	1000	40
$^{13}C_{10}$-Heptachlor [2]	1000	40
$^{13}C_{12}$-Aldrin [2]	1000	40
$^{13}C_{10}$-trans-Chlordane [2]	1000	40
$^{13}C_{10}$-trans-Nonachlor [2]	1000	40
$^{13}C_{12}$-p,p'-DDE [2]	1000	40
$^{13}C_{12}$-p,p'-DDT [2]	1000	40
d_4-alpha-Endosulfan [2]	1000	40
d_4-beta-Endosulfan [2]	1000	40
$^{13}C_{12}$-PCB 3 [3]	1000	40
$^{13}C_{12}$-PCB 8 [3]	1000	40
$^{13}C_{12}$-PCB 28 [3]	1000	40
$^{13}C_{12}$-PCB 101 [3]	1000	40
$^{13}C_{12}$-PCB 118 [3]	1000	40
$^{13}C_{12}$ PCB 180 [3]	1000	40
$^{13}C_{12}$-PCB 202 [3]	1000	40
$^{13}C_{12}$-PCB 206 [3]	1000	40
$^{13}C_{12}$-PCB 209 [3]	1000	40
$^{13}C_{12}$-PCB 159 [3] (separate solution)	1000	40
Recovery Standards	**Nominal Conc. in Recovery Std Solution (ng/mL)**	
$^{13}C_{12}$-PCB 52	1000	40
$^{13}C_{12}$-PCB 138	1000	40
$^{13}C_{12}$-PCB 153	1000	40
Extraction Standard	**Nominal Conc. in Ext'n Std. Sol'n (ng/mL)**	**Amt Added to each sample (ng)**
PCB 204	900	Contract dependent

[1] Concentrations are two times higher in the authentic spiking solution used for aqueous samples.
[2] Surrogates spiked for analysis of chlorinated pesticides.
[3] Surrogates spiked for analysis of PCBs, Aroclors, and Technical Toxaphene.

Table 4: Analysis of E1 Chlorobenzenes, Pesticides and PCBs
Nominal Concentrations of Calibration Standard Solutions (ng/mL)

Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
1,3-dichlorobenzene	10	20	40	160	400	2000	
1,4-dichlorobenzene	10	20	40	160	400	2000	
1,2-dichlorobenzene	10	20	40	160	400	2000	
1,3,5-trichlorobenzene	10	20	40	160	400	2000	
1,2,4-trichlorobenzene	10	20	40	160	400	2000	
1,2,3-trichlorobenzene	10	20	40	160	400	2000	
1,2,3,5- /1,2,4,5-tetrachlorobenzene	20	40	80	320	800	4000	
1,2,3,4-tetrachlorobenzene	10	20	40	160	400	2000	
Pentachlorobenzene	10	20	40	160	400	2000	
Hexachlorobenzene	10	20	40	160	400	2000	
alpha-HCH	20	40	80	320	800	4000	
beta-HCH	20	40	80	320	800	4000	
gamma-HCH	20	40	80	320	800	4000	
delta-HCH	20	40	80	320	800	4000	
Heptachlor	10	20	40	160	400	2000	
Aldrin	20	40	80	320	800	4000	
trans-Chlordane	20	40	80	320	800	4000	
cis-Chlordane	20	40	80	320	800	4000	
trans-Nonachlor	20	40	80	320	800	4000	
cis-Nonachlor	20	40	80	320	800	4000	
o,p'-DDE	10	20	40	160	400	2000	
p,p'-DDE	10	20	40	160	400	2000	10000
o,p'-DDT	10	20	40	160	400	2000	
p,p'-DDT	10	20	40	160	400	2000	
Octachlorostyrene	10	20	40	160	400	2000	
Oxychlordane	20	40	80	320	800	4000	
op'-DDD	10	20	40	160	400	2000	
pp'-DDD	10	20	40	160	400	2000	
Mirex	10	20	40	160	400	2000	
PCB 1	10	20	40	160	400	2000	
PCB 3	10	20	40	160	400	2000	
PCB 4	10	20	40	160	400	2000	
PCB 8	10	20	40	160	400	2000	
PCB 15	10	20	40	160	400	2000	
PCB 18	10	20	40	160	400	2000	
PCB 19	10	20	40	160	400	2000	
PCB 34/23	10	40	80	320	800	4000	
PCB 28	10	20	40	160	400	2000	

Table 4 (cont'd)

Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
PCB 31	10	20	40	160	400	2000	
PCB 34	10	20	40	160	400	2000	
PCB 37	10	20	40	160	400	2000	
PCB 40	10	20	40	160	400	2000	
PCB 44	10	20	40	160	400	2000	
PCB 49	10	20	40	160	400	2000	
PCB 52	10	20	40	160	400	2000	
PCB 54	10	20	40	160	400	2000	
PCB 56	10	20	40	160	400	2000	
PCB 66	10	20	40	160	400	2000	
PCB 77	10	20	40	160	400	2000	
PCB 81	10	20	40	160	400	2000	
PCB 87	10	20	40	160	400	2000	
PCB 95	10	20	40	160	400	2000	
PCB 99	10	20	40	160	400	2000	
PCB 101	10	20	40	160	400	2000	
PCB 104	10	20	40	160	400	2000	
PCB 105	10	20	40	160	400	2000	
PCB 110	10	20	40	160	400	2000	
PCB 114	10	20	40	160	400	2000	
PCB 118	10	20	40	160	400	2000	
PCB 123	10	20	40	160	400	2000	
PCB 126	10	20	40	160	400	2000	
PCB 138	10	20	40	160	400	2000	
PCB 149	10	20	40	160	400	2000	
PCB 151	10	20	40	160	400	2000	
PCB 153	10	20	40	160	400	2000	
PCB 155	10	20	40	160	400	2000	
PCB 156	10	20	40	160	400	2000	
PCB 157	10	20	40	160	400	2000	
PCB 167	10	20	40	160	400	2000	
PCB 169	10	20	40	160	400	2000	
PCB 170	10	20	40	160	400	2000	
PCB 180	10	20	40	160	400	2000	
PCB 183	10	20	40	160	400	2000	
PCB 187/182	20	40	80	320	800	4000	
PCB 188	10	20	40	160	400	2000	
PCB 189	10	20	40	160	400	2000	
PCB 194	10	20	40	160	400	2000	
PCB 196	10	20	40	160	400	2000	

Table 4 (cont'd)

Compound Name	Calibration Standards						
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (Mid-Level) (ng/mL)	Level F CS-5 (ng/mL)	Extra high CAL (ng/mL)
PCB 202	10	20	40	160	400	2000	
PCB 204	10	20	40	160	400	2000	
PCB 205	10	20	40	160	400	2000	
PCB 206	10	20	40	160	400	2000	
PCB 208	10	20	40	160	400	2000	
PCB 209	10	20	40	160	400	2000	
Labeled Surrogates							
$^{13}C_6$-1,4-dichlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-1,2,3-trichlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-1,2,3,4-tetrachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-Pentachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-Hexachlorobenzene	400	400	400	400	400	400	400
$^{13}C_6$-beta-HCH	400	400	400	400	400	400	400
$^{13}C_6$-gamma-HCH	400	400	400	400	400	400	400
$^{13}C_{10}$-Heptachlor	400	400	400	400	400	400	400
$^{13}C_{12}$-Aldrin	400	400	400	400	400	400	400
$^{13}C_{10}$-trans-Chlordane	400	400	400	400	400	400	400
$^{13}C_{10}$-trans-Nonachlor	400	400	400	400	400	400	400
$^{13}C_{12}$-p,p'-DDE	400	400	400	400	400	400	400
$^{13}C_{12}$-p,p'-DDT	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 3	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 8	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 28	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 101	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 118	400	400	400	400	400	400	400
$^{13}C_{12}$ PCB 180	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 202	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 206	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 209	400	400	400	400	400	400	400
Recovery Standards							
$^{13}C_{12}$-PCB 52	400	400	400	400	400	400	400
$^{13}C_{12}$-PCB 138	400	400	400	400	400	400	400

Note: Level A is a sensitivity standard only.

Table 5: Analysis of Technical Toxaphene
Nominal Concentrations of Calibration Solutions (ng/mL)

Compound Name	Calibration Standards					
	Level A CS-0 (ng/mL)	Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (ng/mL)	Level E CS-4 (ng/mL)	Level F CS-5 (ng/mL)
Technical Toxaphene	2500	5000	25000	100000	250000	800000
Surrogate Standard						
$^{13}C_{12}$-PCB 159	400	400	400	400	400	400
Recovery Standard						
$^{13}C_{12}$-PCB 138	400	400	400	400	400	400

Notes: Level A is a sensitivity standard only.
Level D CAL VER or bracketing CAL level.

Table 6: GC/ECD Analysis of E2 Pesticides
Nominal Concentrations of Calibration Solutions (ng/mL)

Compound Name	Level A CS-0 (ng/mL)	Calibration Standards				
		Level B CS-1 (ng/mL)	Level C CS-2 (ng/mL)	Level D CS-3 (Mid-Level) (ng/mL)	Level E CS-4 (ng/mL)	Level G CS-5 (ng/mL)
Dieldrin	4	8	40	80	160	800
alpha-Endosulfan	4	8	40	80	160	800
beta-Endosulfan	4	8	40	80	160	800
Endosulfan Sulfate	4	8	40	80	160	800
Endrin	4	8	40	80	160	800
Methoxychlor	4	8	40	80	160	800
Endrin Aldehyde	4	8	40	80	160	800
Endrin Ketone	4	8	40	80	160	800
cis-Heptachlor epoxide	4	8	40	80	160	800
delta-HCH	5	10	50	100	200	500
Surrogates						
d_4-alpha-Endosulfan	75	75	75	75	75	75
d_4-beta-Endosulfan	75	75	75	75	75	75
Recovery Standard						
$^{13}C_{12}$-PCB 153	75	75	75	75	75	75

Notes: Level A is a sensitivity standard only.

3.4 Preparation of Reagents

Activated Copper is freshly prepared according to standard operating procedure SLA-044.

Ammonium Sulfate ($(NH_4)_2SO_4$, saturated) is prepared by dissolving ammonium sulfate (700 g) in ultra pure water (1 L). The solution is extracted by shaking twice with dichloromethane (2 x 100 mL) and once with hexane (100 mL). Shelf life 3 months, after expiry re-extract and re-use.

Anhydrous Sodium Sulfate (Na_2SO_4, granular 12–60 mesh, J.T. Baker, or demonstrated equivalent approved for use of by Operations Management and QA) is cleaned by baking at a minimum of 300 °C as described in SLA-093. Shelf life 2 months, after expiry rebake and re-use.

Anti-Bumping Granules (EM Science) are baked for 8 hours at a minimum of 300 °C prior to use and stored in a clean baked jar.

Glass Fibre Filters (Ahlstrom, 161 grade, 1.1 µm, 42.5 mm diameter, or equivalent) are cleaned by baking overnight at a minimum of 300 °C.

Glass Fibre Filter Bed – refer to SLA-108 “Preparation of Filter Bedding” for instructions about preparation, cleaning and storage.

Hydrochloric Acid (conc., Seastar Chemicals, quartz distilled) is used as received. Indefinite shelf life.

Hydrochloric Acid (HCl, 1M) is prepared by adding HCl (100 mL) to ultra pure water (1 L). Shelf life 2 years, discard when expired.

Reagent Sand (Aldrich Chemicals, white quartz, -50 +70 mesh) is proofed by lot prior to use and maybe cleaned by soxhlet extraction with dichloromethane for 16 hours as necessary. The clean stand is stored in a clean jar.

Silanized glass wool is stored in a clean glass jar and solvent rinsed with toluene (2 times) and hexane (2 times) prior to use.

Sodium Hydroxide (NaOH, 1 M) is prepared by dissolving sodium hydroxide pellets (AR grade, 40 g) in ultra pure water (1 L). The solution is extracted by shaking twice with dichloromethane (100 mL) and once with hexane (100 mL). The solution is stored in an amber jar with PTFE lined lid. Typically 3 L is prepared. Shelf life 3 months, after expiry re-extract and re-use.

Sodium Hydroxide (NaOH, 6 N) is prepared by dissolving sodium hydroxide pellets (AR grade, 240 g) in ultra pure water (1 L). The solution is extracted by shaking twice with dichloromethane (2 x100 mL) and once with hexane (100 mL). The solution is stored in an amber jar with PTFE lined lid. Shelf life 3 months, discard after expiry.

Solvents: All solvents used are high purity, distilled in glass solvents, either HPLC grade or pesticide residue grade. Each lot number of solvent must be checked for impurities by performing a solvent proof, prior to use. All solvent mixtures used in the analyses are made by mixing the appropriate quantities on a volume basis. Shelf life 2 years for solvent mixtures, discard after expiry.

Sulfuric Acid (H_2SO_4, conc., Seastar Chemicals, quartz distilled) is used as received. Indefinite shelf life.

Ultra Pure Water (Seastar Chemicals or equivalent, contaminant free) is used as received. Indefinite shelf life.

3.5 Preparation of Chromatography Materials

Refer to the following Standard Operating Procedures:

Document ID	Title
SCH-001	Deactivation/Activation Procedures
SCH-002	Column Packing Procedures
SCH-003	Column Cutpoint Procedures
SCH-004	Layered Silica Gel Chromatography Procedures
SCH-005	Preparation of Carbon/Celite Column
SCH-006	Preparation and Maintenance of Biobead Columns

The degrees of deactivation for the column materials below are nominal degrees. The exact degree of deactivation for each material is determined as required.

Alumina 1% Deactivated (Fisher, Basic Brockman Activity 1, 60–325 mesh) is baked for a minimum of 8 hours at 450°C and deactivated with ultra pure water (typically 1% by weight) as described in SCH-001. Deactivated alumina is stored under nitrogen in a stoppered flask and allowed to equilibrate for 24 hours. Cutpoints are determined prior to use (SCH-003). Shelf life 6 months, after expiry redetermine cutpoints.

22% H_2SO_4 Acidic Silica is prepared by adding H_2SO_4 (conc., equivalent to 22% of final weight of acidified silica) to baked silica and agitating until homogenous, as described in SCH-004. The mixture is stored in a stoppered round bottom flask. Shelf life 2 months, discard after expiry.

44% H_2SO_4 Acidic Silica is prepared by adding H_2SO_4 (conc., equivalent to 44% of final weight of acidified silica) to baked silica and agitating until homogenous, as described in SCH-004. The mixture is stored in a stoppered round bottom flask. Shelf life 2 months, discard after expiry.

28% NaOH Basic Silica is prepared by adding NaOH solution (equivalent to 28% of final weight of basic silica) to baked silica and agitating until homogenous, as described in SCH-004. The silica is stored in a stoppered round bottom flask. Shelf life 2 months, discard after expiry.

Biobeads (SX-3, Bio-Rad, 200 - 400 mesh) are soaked in 1:1 dichloromethane:hexane for 24 hours prior to column preparation as described in SCH-006.

4.5% Carbon/Celite Mixture is prepared by mixing activated carbon AX-21 (4.5 g, 10µm – 100µm, Anderson Dev. Corp.) with Celite 545 (95.5 g) and shaking until the mixture is uniform as

described in SCH-005. The mixture is stored in a stoppered glass reagent bottle. The mixture must be well agitated before use. Cutpoints are determined as described in SCH-003. Shelf life 6 months, after expiry redetermine cutpoints.

Florisil 2.0% Deactivated (Supelco or US Silica, Pesticide grade, 60/100 mesh) is activated by heating at 450 °C for a minimum of 8 hours and deactivated with ultra pure water as described in SCH-001. Florisil is stored under nitrogen in a stoppered flask and allowed to equilibrate for 24 hours. The cutpoints are determined prior to use. (SCH-003). Shelf life 3 months, after expiry redetermine cutpoints.

Silica Gel (SiliCycle G60, 60–200 µm, 60 Å, or equivalent) is heated for 8 hours at 450 °C and stored, under nitrogen, in a stoppered reagent bottle following the procedures described in SCH-001. Shelf life 3 months, after expiry rebake and re-use.

3.6 Preparation of Chromatography Columns

3.6.1 Florisil Column

Fill a glass column (1 cm x 25 cm with 100 mL reservoir) with hexane and dry pack the Florisil (8 g, 2.0–2.1% deactivated) following the procedures described in SCH-002. Cap the Florisil with a 10 mm layer of anhydrous sodium sulfate.

3.6.2 BioBead Column

Slurry-packed Biobeads with 1:1 dichloromethane:hexane into a glass column (3 cm x 50 cm) equipped with a sintered glass frit.

Biobead columns are re-used after cleaning procedures have been carried out. Refer to SCH-006 for the preparation, use, and cleaning of Biobead columns.

3.6.3 Layered Acid/Base Silica Column

Note: This column cannot be used if the analysis of pesticides is required.

Prepare a layered silica chromatography column (10 mm O.D. x 250 mm long) by sequentially dry packing the following layers in hexane following the procedures described in SCH-002:

neutral silica (0.5 g), basic silica (28% NaOH, 2 g), neutral silica (0.5 g), acidic silica (44% H_2SO_4, 4 g), acidic silica (22% H_2SO_4, 2 g), and neutral silica (1 g)

3.6.4 Alumina Column

Note: This column cannot be used if the analysis of pesticides is required.

Pack alumina (baked, 1% deactivated 6 g) into a glass chromatographic column (1 cm x 15 cm with 50 mL reservoir) filled with hexane following the procedures described in SCH-002 Cap the alumina with a 10 mm layer of anhydrous granular sodium sulfate.

4. QUALITY ASSURANCE/QUALITY CONTROL

Refer to the following Standard Operating Procedure:

Document ID	Title
SLA-016	Preparation of QA/QC Samples

All samples are analyzed in batches. The composition of a batch is detailed on a Batch List, including the method number, quality control samples and standards to use.

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.

- Blanks - One procedural blank is analyzed with each batch. A procedural blank is prepared by spiking an aliquot of the surrogate standard solution into a clean matrix. If required, an aliquot of field standard is also added. The procedural blank is extracted and analyzed using the same procedures as the test samples in the analysis batch. The following materials or equivalent, prepared as per Section 3.4, are recommended for blank preparation: Aqueous - 1L purified water, Seastar Chemicals; solids - 10 g reagent sand, Aldrich Chemicals; tissue - 0.3 g canola oil (Rimini).

- Initial Precision and Accuracy (IPR) is demonstrated when commencing the method or when significant changes have been made to the method. The IPR is carried out by the analysis of four spiked reference samples (SPM; prepared as described below).

- Spiked Reference Samples - A spiked reference matrix (SPM) sample is analyzed with each batch. The reference sample to be analyzed is assigned to the analyst when the batch is assigned. The SPM sample is prepared by accurately weighing an in-house reference matrix (known to contain low background levels of target analytes) into a beaker and spiking with an aliquot of authentic spiking standard solution. The matrix is spiked with an aliquot of surrogate standard solution and extracted, following procedures in Section 5 of this method. The following materials or equivalent, prepared as per Section 3.4, are recommended for SPM preparation: Aqueous - 1L purified water, Seastar Chemicals; solids - 10 g reagent sand, Aldrich Chemicals; tissue - 0.3 g canola oil, Rimini, or 10 g clean fish tissue.

- Reference Samples – Certified reference materials (CRM) are commercially available and used to validate and periodically check the methods. The type of reference material to be analyzed is assigned to the analyst when the batch is assigned.

- Duplicates – A duplicate sample is analyzed with analysis batches containing 7-20 test samples or as required by contract, provided sufficient sample is available. For some matrices (XAD-2 columns, filters) only field duplicates (if available) can be analyzed.

- Surrogate/Authentic/Recovery (SAR) solution - An optional diagnostic standard that may be prepared and included with pesticide batches.

- For multi-component analysis such as Aroclors and Toxaphene where incompatibilities may preclude spiking each batch reference sample, a reference sample must be analyzed every two years.

Batch composition can be altered to suit batch or quality control requirements specified under contract.

All elements of the QA/QC program at SGS AXYS Analytical Services are documented in the most recent version of QDO-001 *QA/AC Policies and Procedures Manual* and in SQA-001 *Data Validation Procedures*.

The quality control limits for duplicate samples, procedural blanks, reference samples, surrogate recoveries, and detection limits are specified in the following tables:

Table 7	QC Acceptance Criteria for E2 Pesticides by GC/ECD
Table 8	QC Acceptance Criteria for E1 Pesticides by GC/MS
Table 9	QC Acceptance Criteria for PCB Congeners by GC/MS

Table 7: QC Acceptance Criteria for Analysis of E2 Pesticides by GC/ECD

Analyte	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS						Procedural Blank Level ng	Acceptable Matrix Spike in matrices except tissue % Recovery [1]	Acceptable Matrix Spike in tissue % Recovery [1]
	Solids ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng			
Delta-HCH	0.1	1	0.1	0.1	1	1	<1	60-130	60-130
cis-Heptachlor epoxide	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
alpha-Endosulfan	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Dieldrin	0.1	1	0.1	0.1	1	1	<1	60-130	65-130
Endrin	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
Endosulfan sulfate	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Endrin ketone	0.1	1	0.1	0.1	1	1	<1	60-130	65-130
beta-Endosulfan	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Endrin Aldehyde	0.1	1	0.1	0.1	1	1	<1	60-130	Note [2]
Methoxychlor	0.5	5	0.5	0.5	5	5	<5	60-130	60-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 column			
Final Vol, µL	100	100	100	100	100	100			

[1] Recoveries quoted are guidelines only and vary according to matrix. Consult detailed method performance data available with method documentation for specific criteria.

[2] Endrin aldehyde in tissue samples is reported as an estimated "Information Value" – recovery limits do not apply

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
d_4-alpha-Endosulfan	40-130
d_4-beta-Endosulfan	40-130

QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean, (applicable to concentrations >10 times the DL)
Instrument Sensitivity	S/N ratio ≥3:1 for 4 pg methoxychlor.
Instrument Linearity	Linearity is demonstrated by at least a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% (delta-HCH may be 25%)
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e.,the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF: Continuing Cal Ver	RRFs from opening/closing calibration standards must be within ±25% of the mean RRFs from initial calibration.
Chromatogram Quality (GC Resolution)	1. Valley height between d_4-alpha-Endosulfan and alpha-Endosulfan and between d_4-beta-Endosulfan and beta-Endosulfan must be ≤50% for equal concentrations. 2. Endrin (or $^{13}C_{12}$-endrin) breakdown must be ≤20%.
Analyte /Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.

Table 8: QC Acceptance Criteria for Analysis of E1 Pesticides by GC/MS

Analyte	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS (SDL) *						Procedural Blank Level ng	Acceptable Matrix Spike in all matrices except tissue % Recovery [1]	Acceptable Matrix Spike in tissue % Recovery [1]
	Solid ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng			
Dichlorobenzenes	1	10	1.0	1.0	10	10	<10	Note 1	Note 1
Trichlorobenzenes	1	10	1.0	1.0	10	10	<10	Note 1	Note 1
Tetrachlorobenzenes	0.5	5	0.5	0.5	5	5	<5	Note 1	Note 1
Pentachlorobenzene	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Hexachlorobenzene	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
alpha-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
beta-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
gamma-HCH	0.2	2	0.2	0.2	2	2	<2	70-130	70-130
Heptachlor	0.5	5	0.5	0.5	5	5	<5	70-130	70-130
Aldrin	0.5	5	0.5	0.5	5	5	<5	70-130	70-130
Oxychlordane	0.5	5	0.5	0.5	5	5	<5	60-130	70-130
Octachlorostyrene	0.5	5	0.5	0.5	5	5	<5	60-130	70-130
trans-Chlordane	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Chlordane	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDE	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDE	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
trans-Nonachlor	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Nonachlor	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDD	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDD	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDT	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDT	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Mirex	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Technical Toxaphene (determined as present by the detection of a component)	15	150	15	15	150	150	<150	60-130	60-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 column			
Final volume, µL	100	100	100	100	100	100			

* SDLs listed are estimates based on typical sample matrix type and may be higher depending on particular sample characteristics

Table 8 cont'd…

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**	
$^{13}C_6$-1,4-Dichlorobenzene	Note 1	
$^{13}C_6$-1,2,3-Trichlorobenzene	Note 1	
$^{13}C_6$-1,2,3,4-Tetrachlorobenzene	Note 1	
$^{13}C_6$-Pentachlorobenzene	30-130	
$^{13}C_6$-Hexachlorobenzene	30-130	
$^{13}C_6$-beta-HCH	30-130	
$^{13}C_6$-gamma-HCH	40-130	
$^{13}C_{10}$-Heptachlor	30-130	
$^{13}C_{10}$-Aldrin	30-130	
$^{13}C_{10}$-trans-Chlordane	30-130	
$^{13}C_{10}$-trans-Nonachlor	30-130	
$^{13}C_{12}$-p,p'-DDE	40-130	
$^{13}C_{12}$-p,p'-DDT	40-130	
$^{13}C_{12}$-PCB 159	40-130	(only when technical toxaphene is analyzed)

QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Procedural Blank	See above table or <10% of analyte value
Instrument Sensitivity	S/N ≥3:1 for 10 pg HCB, for 10 pg p,p'-DDT and for 20 pg oxychlordane. S/N ≥2:1 for 2.5 ng of Technical Toxaphene with a minimum of 4 peaks detected.
Instrument Linearity	For a minimum 5-point calibration, a relative standard deviation of the RRFs ≤20% for all compounds, except for $^{13}C_{12}$-pp'-DDT where RSD of RRF ≤25%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e., the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF: Continuing CAL Ver	RRFs for all compounds from opening/closing calibration standards must be within ±20% of the mean RRFs from the initial calibration.
Chromatogram Quality **Max Peak Width:** **Resolution:**	1. Peak width at half height for p,p'-DDT is 5 sec. 2.Valley height between p,p'-DDD and o,p-DDT must be less than 10% the height of the peaks 3. PCB 209 peak must be symmetrical with negligible tailing, ≤20 sec. 4. p,p'-DDT breakdown must be ≤15%.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window for target compounds	RRT must be within ±3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labeled surrogate) Authentic compound must elute after its labeled analogue

Note 1: Recovery of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These compounds may be offered as "compounds of opportunity", reportable only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.

Table 9: QC Acceptance Criteria for Analysis of PCB Congeners by GC/MS

Congener (IUPAC)	TYPICAL SAMPLE SPECIFIC DETECTION LIMITS [1]						Procedural Blank Level ng	Acceptable Matrix Spike in matrices except tissue % Recovery	Acceptable Matrix Spike in tissue % Recovery
	Solids ng/g	Aqueous ng/L	Tissue ng/g	Pulp ng/g	Ambient Air ng	XAD-2 column ng/col			
Monochlorinated (1-3)	0.5	5	0.5	0.5	5	5	<1	60-130	70-130
Dichlorinated (4-15)	0.5	5	0.5	0.5	5	5	<1	60-130	70-130
Trichlorinated (16-39)	0.1	1	0.1	0.1	1	1	<1	60-130	70-130
Tetrachlorinated (40-81)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Pentachlorinated (82-127)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Hexachlorinated (128-169)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Heptachlorinated (170-193)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Octachlorinated (194-205)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Nonachlorinated (206-208)	0.1	1	0.1	0.1	1	1	<1	70-130	70-130
Decachlorinated (209)	0.5	5	0.5	0.5	5	5	<1	70-130	70-130
Aroclor Equivalents [2]	1	10	1	1	10	10	<1		
Typical Sample Size:	10 g	1 L	10 g	10 g	1 PUF/filter	1 col			
Final volume, µL	100	100	100	100	100	100			

[1] Sample specific detection limits may vary depending on sample characteristics
[2] Aroclor formulations are not included in the batch QC OPR sample. Limit for biennial (every two years) matrix spikes with Aroclor formulations is 50-150% recovery.

SURROGATE STANDARD	% RECOVERY RANGES (All Matrices)
$^{13}C_{12}$-PCB 3	15-130
$^{13}C_{12}$-PCB 8	20-130
$^{13}C_{12}$-PCB 28	40-130
$^{13}C_{12}$-PCB 101	40-130
$^{13}C_{12}$-PCB 118	40-130
$^{13}C_{12}$-PCB 180	40-130
$^{13}C_{12}$-PCB 202	40-130
$^{13}C_{12}$-PCB 206	40-130
$^{13}C_{12}$-PCB 209	40-130

QC Parameter	Specification
Analysis Duplicate	The relative difference must be ≤40%, i.e., the duplicates must agree to within ±20% of the mean (applicable to concentrations >10 times the DL).
Procedural Blank	See above or <10% of analyte value.
Matrix Spike Recovery	See above; PCB 19 must be greater than 55%; PCB 104 must be greater than 60%.
Instrument Sensitivity	S/N ratio ≥3:1 for 10 pg PCB 118.
Instrument Linearity	Linearity is determined by at least a 5-point calibration with a relative standard deviation of the RRFs ≤20%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to within ±20% over a 12-hour period, i.e., the relative difference must be ≤40%, which is equivalent to 28.3% RSD.
RRF Continuing CAL VER	RRFs from opening/closing calibration standards must be within ±20% of the mean RRFs from the initial calibration for all compounds.
Chromatogram Quality **Max. Peak Width:** **Resolution:**	1. PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds. 2. Valley height must be ≤80% of smallest peak height of PCB 28/31 pair.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window for target compounds	RRT must be within ±3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labeled surrogate). Authentic compound must elute after its labeled analogue.

5. EXTRACTION PROCEDURES

Samples are analyzed in batches. The samples to be analyzed, the batch QC samples, the name and volumes of the surrogate and recovery standards required, and any additional information concerning the analysis are documented on a Batch List given to an analyst. Refer to SOP SLA-033 for details of assigning analysis batches.

Each analyst performs the extraction procedure according to the following written extraction procedures and completes an analysis worksheet (see following table) for each sample during the sample extraction. The analyst is referred to the following Standard Operating Procedures for details of routine laboratory techniques.

Document ID	Title
SLA-004	Sample Control Procedures
SLA-005	Use and Maintenance of Rotary Evaporator Equipment
SLA-006	Nitrogen Blowdown Concentration Technique
SLA-007	Kuderna-Danish Concentration Technique
SLA-008	Preparing Extracts for Instrumental Analysis
SLA-017	Spiking Procedure
SLA-020	Gravimetric Lipid Determination by Weight of Extract
SLA-023	Use of Balances
SLA-024	Sealing Ampoules
SLA-027	Completing a Worksheet
SLA-033	Procedures for Making an Analysis Batch
SLA-045	Removal of Sulfur From Extracts Using Activated Copper
SLA-067	Use of Nitrogen Cylinders
SLA-072	Computer Preparation of Labels
SLA-078	Spike Witness Program
SLA-081	Labeling Protocols and Sample Transfer Procedures
SLA-082	Handling of Human Biohazards
SLA-084	Preparation of Aqueous Samples for Extraction
SLA-085	Subsampling Procedures for Solids and Tissues
SLA-087	Transferring an Ampouled Standard to a Reacti-Vial
SLA-092	Determination of Suspended Solids Content in Aqueous Samples
SLA-108	Preparation of Filter Bedding (“Popcorn”)
SLA-124	Liquid-Liquid Extraction Supplemental Techniques

The following worksheets are available to document the analysis:

FWO-305	Sample Labeling Information
FWO-306	Sample Weight and Moisture
FWO-307	Aqueous Samples
FWO-308	Lipid Sheet
FWO-309	Standards Spiking into C-Tube
FWO-310	Sample Spiking Sheet
FWO-311	Extract Splitting
FWO-314	Notes
FWO-317	Extraction – Blood, Milk, Nonylphenols
FWO-319	Extraction – Ash, Acid extractable Pesticides
FWO-331	Dean-Stark Extraction Worksheet
FWO-356	Extract Cleanup - Biobead/Cu
FWO-357	Extract Cleanup - Columning

5.1 Soxhlet Extraction of Solids

The following Soxhlet extraction procedures are suitable for sediment, soil, sludge, ash, pulp, and particulate filters (dry).

5.1.1 Sludge, Ash, Soil and Sediment Soxhlet Extraction **(Default Procedure)**

This extraction procedure is applicable to the matrices and sample sizes listed below.

Matrix	Typical Sample Size
Sludge	10 g (Dry)
Ash	5 g
Sediment/Soil	5–10 g (Wet)

NOTE: Some soil or sediment samples may contain pests or other pathogens that could be harmful to agriculture, and therefore be classified as an Agricultural Hazard. Refer to the Batch List to determine if any samples are considered Agricultural Hazards. Samples considered Agricultural Hazards must be handled following the safety procedures described in SLA-079, *Agricultural Hazard Protocols for Soils*.

For extraction of fly ash samples, refer to Section 5.1.2.

Accurately weigh a homogenized sample to at least two decimals into a beaker containing anhydrous granular sodium sulfate (75 -100 g). Stir the mixture well with a spatula, and allow the mixture to dry to a free-flowing powder for a minimum of 30 minutes.

Note: For very wet sediments, it may be preferable to weight the sample into a beaker and add the anhydrous sodium sulfate in portions, stirring well after each addition.

Place a layer of silica or anhydrous sodium sulfate (2 g for small thimbles, 5 g for large thimbles, neutral, baked) into a clean Soxhlet thimble. Quantitatively transfer the mixture into the thimble. Add an aliquot(s) of the appropriate surrogate standard solutions(s) to the mixture, and allow to equilibrate for at least 30 minutes. Add dichloromethane (300 mL for small thimbles, 600 mL for large thimbles) and 4–5 anti-bumping granules to the Soxhlet's round bottom flask. Place the thimble into the Soxhlet body and extract the mixture for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of at least 4 cycles per hour.

Allow the mixture to cool. Concentrate the extract by rotary evaporation (water bath <30 °C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane. Add activated copper to the extract to remove sulfur.

The extract is now ready for chromatographic cleanup procedures described in Section 6.0. If only coplanar PCBs are required, the extract can proceed directly to the coplanar PCB congener chromatographic cleanup step (Appendix B).

5.1.2 Fly Ash Samples

Matrix	Sample Size
Fly Ash	5 g

NOTE: Omit the acidification step if pesticide analysis is required.

Accurately weigh a homogenized subsample of fly ash to at least two decimals into a beaker. Add hydrochloric acid (50 mL, 1 M HCl). Digest for 30 minutes in an ultrasonic bath. Using pH paper, check that the pH is <2. If not, add hydrochloric acid (HCl, 1M) drop wise until the pH is <2, and repeat the sonication step.

Pour the mixture through a Millipore filtration apparatus containing a baked glass fibre filter paper. Wash the filter with beaker rinses of ultra pure water (2 x 100 mL). Check that the pH of the final volume of filtrate passing through the filter is neutral (based on pH of the ultra pure water).

Filtrate:

Adjust the pH of the filtrate to 6.5–7.5 with sodium hydroxide (6N, NaOH). Transfer the filtrate with dichloromethane rinses to a separatory funnel (500 mL) and extract by shaking with dichloromethane (2 x 100 mL). Combine the filtrate extract with the toluene filter extract (from below).

Filter:

Note: The occasional formation of an interference during the extraction procedure and the high boiling point of the toluene may limit the quantification of PCBs 1 through 15 in the filter matrix.

Remove the filter and place in clean Petri dish in a fumehood and allow to air dry overnight OR place in a beaker and mix with granular anhydrous sodium sulfate. Place a layer of silica or anhydrous sodium sulfate (2 g for small thimbles, 5 g for large thimbles, neutral, baked) into a clean Soxhlet thimble. Transfer the filter or filter and sodium sulfate into the thimble. Add an aliquot of the surrogate standard(s) into the thimble, and allow to equilibrate for at least 30 minutes. Add toluene and 4–5 anti-bumping granules to the Soxhlet's round bottom flask. Place the thimble into the Soxhlet body and extract the mixture for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour.

Allow the extract to cool. Combine the extract with the dichloromethane extract from above and dry over granular anhydrous sodium sulfate. Concentrate the extract by rotary evaporation (water bath <30 °C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 50 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane. Add activated copper to the extract to remove sulfur.

The extract is now ready for chromatographic cleanup procedures described in Section 6.0. If only coplanar PCBs are required, the extract can proceed directly to the coplanar PCB congener chromatographic cleanup step (Appendix B).

5.1.3 Pulp Soxhlet Extraction

This extraction procedure is applicable to the matrix and sample size listed below.

Matrix	Typical Sample Size
Pulp	20 g (Dry)

Accurately weigh a homogenized pulp sample to at least two decimals. Place a layer of silica or anhydrous sodium sulfate (2 g for small thimbles, 5 g for large thimbles, neutral, baked) into a clean Soxhlet thimble. Transfer the pulp sample into the thimble. Add an aliquot(s) of the appropriate surrogate standard solution(s) to the thimble, and allow to equilibrate for at least 30 minutes. Add dichloromethane (300 mL) and 4–5 anti-bumping granules to the Soxhlet's round bottom flask. Place the thimble into the Soxhlet body and extract the mixture for a minimum of 16 hours. Adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour.

Allow the mixture to cool. Concentrate the extract by rotary evaporation (water bath <30°C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane. Add activated copper to the extract to remove sulfur.

The extract is now ready for chromatographic cleanup procedures described in Section 6.0. If only coplanar PCBs are required, the extract can proceed directly to the coplanar PCB congener chromatographic cleanup step (Appendix B).

5.2 Tissue Extraction

This extraction procedure is applicable to the matrix and sample size listed below.

Matrix	Sample Size
Tissue	5–10 g (wet)

5.2.1 Sample Preparation

Accurately weigh a homogenized tissue sample to at least two decimals into a beaker containing anhydrous granular sodium sulfate (75 - 100 g). Stir the mixture well with a spatula, and allow the mixture to dry to a free-flowing powder for a minimum of 30 minutes.

5.2.2 Extraction

Place a layer of silica or anhydrous sodium sulfate (2 g for small thimbles, 5 g for large thimbles, neutral, baked) into a clean Soxhlet thimble. Quantitatively transfer the mixture into the thimble. Add an aliquot(s) of the appropriate surrogate standard solutions(s) to the sample, and allow to equilibrate for at least 30 minutes. Add dichloromethane (300 mL) and 4 – 5 anti-bumping granules to the Soxhlet's round bottom flask. Place the thimble into the Soxhlet body and extract the mixture for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the mixture to cool.

If a lipid analysis is required, concentrate the extract to 5 mL by rotary evaporation (<30 °C) and carry out the lipid determination according to standard operating procedures (refer to SLA-020). Following lipid analysis, reduce the volume of final extract to 1 mL by rotary evaporation (<30 °C). Add hexane (1 mL) to the extract for gel permeation column cleanup (Section 5.2.3).

If a lipid analysis is not required, concentrate the extract to 1 mL by rotary evaporation (<30 °C). Add hexane (1 mL) to the extract for gel permeation column cleanup (Section 5.2.3).

5.2.3 Gel Permeation Cleanup

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Collect the first fraction and discard. Collect the second fraction in a round bottom flask and concentrate by rotary evaporation (water bath <30 °C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane.

The extract is ready for the column cleanup procedure (Section 6.0). If only coplanar PCBs are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B).

5.3 Aqueous Sample Extraction

This extraction procedure is suitable for aqueous samples including drinking water, non-potable water, effluents and aqueous sludge.

Matrix	Sample Size
Aqueous	1–4 L

The extraction procedure for aqueous samples depends on the percentage of suspended solid in the sample. The typical sample size is 1 L.

5.3.1 Sample Preparation

Aqueous samples are homogenized and subsampled by the analyst prior to extraction. An aliquot of surrogate standard solution is added to an accurately weighed sample prior to extraction. The analyst must refer to SLA-084 *"Preparation of Aqueous Samples for Extraction"* for complete details of sample homogenization, subsampling, rinsing, surrogate spiking and centrifugation procedures.

If the amount of particulate is required, a determination of suspended solids (SS) must be carried out prior to analysis. This is done according to procedures in SLA-092.

Requirements for Procedures to Separate Solids and Aqueous Phases of the Sample

The aqueous sample extraction procedure depends on the percentage of suspended solids in the sample. Estimate the percent suspended solids by visual inspection. If in doubt of a reasonably accurate visual solid percent estimation, determine the percent suspended solids on a subsample of the sample according to SLA-092 "Determination of Suspended Solids (SS) in a Sample":

- Samples ≤1% suspended solids (SS) are not centrifuged (or filtered) prior to extraction (Section 5.3.2, *Extraction of Samples with ≤1% Suspended Solids*).
- Samples with >1% suspended solids (SS) are centrifuged prior to extraction (Section 5.3.3, *Extraction of Samples with >1% Suspended Solids*). If a particulate weight is required, refer to the Project Notes for details on determining the weight.

NOTE: Samples with ≤1% SS may also be subject to centrifugation prior to extraction as described in SLA-084 if indicated in the Project Notes or Batch List. Any samples that have been centrifuged must be extracted following the procedures described in Section 5.3.3.

5.3.2 Extraction of Samples with ≤1% Suspended Solids

*5.3.2.1 Extraction by Magnetic Stirring **(Default Procedure**)*

After homogenization, subsampling and surrogate spiking, add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar to the sample in an Erlenmeyer flask and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer. Dry the extract over anhydrous sodium sulfate for at least 30 minutes. Transfer the extract into a round-bottom flask with dichloromethane rinses. Concentrate the extract by rotary

evaporation (water bath <30 °C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract under a gentle stream of nitrogen to 300 µL. Make the volume to 1 mL with hexane. The extract is ready for the column cleanup procedure (Section 6.0). If only coplanar PCBs are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B)

5.3.2.2 Extraction by Shaking in a Separatory Funnel ***(Optional Procedure)***

After homogenization and subsampling, extract the sample by adding dichloromethane (100 mL/litre of sample) to the separatory funnel containing the sample and shaking vigorously for two minutes. Collect the dichloromethane layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers and dry over anhydrous sodium sulfate for at least 30 minutes. Transfer the extract into a round-bottom flask with dichloromethane rinses. Concentrate the extract by rotary evaporation (water bath <30°C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane. The extract is ready for the column cleanup procedure (Section 6.0). If only coplanar PCBs are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B).

5.3.3 Extraction of Samples with >1% Suspended Solids

The analyst must refer to SLA-084 *"Preparation of Aqueous Samples for Extraction"* for complete details of sample homogenization, subsampling, rinsing and surrogate spiking procedures.

After homogenization, subsampling and surrogate spiking, centrifuge the entire sample or subsample as described below. **NOTE:** The following centrifugation procedure cannot be used if particulate weight is required.

Transfer a portion of the sample to a clean 500 mL glass jar with a clean solvent rinsed 10 cm filter paper (Pall, A/E grade, 1.0 µm, 102 mm diameter) in the bottom of the jar. Spin the sample in the centrifuge at 1500 rpm until the solid has settled. Decant the supernatant into an Erlenmeyer flask (if proceeding according to Section 5.3.3.1 a) or a separatory funnel (if proceeding according to Section 5.3.3.1 b). Repeat the procedure until the entire sample has been centrifuged, leaving the filter paper in place throughout the process. Once the entire sample has been processed, dry the particulate and filter in the jar by mixing with anhydrous sodium sulfate. Extract the solids as described for particulate in Section 5.3.3.2.

Rinse the jar with ultra pure water and dichloromethane. Add the rinses to the separatory funnel/Erlenmeyer flask containing the supernatant. Extract the supernatant as described in Section 5.3.3.1.

5.3.3.1 Supernatant

a. Transfer the supernatant and rinses to a clean flask. Add dichloromethane (300 mL) and a pre-cleaned PTFE magnetic stir bar to the flask, and extract by stirring the solution (with vortex) for a minimum of 30 minutes. Quantitatively transfer the solution to a separatory funnel and draw off the dichloromethane layer. Discard the aqueous layer.

Or optional procedure

b. Extract the supernatant by adding dichloromethane (100 mL/litre of filtrate) to the separatory funnel containing the filtrate and shaking vigorously for two minutes. Collect the dichloromethane (lower) layer in an Erlenmeyer flask. Repeat the extraction twice more. Combine the dichloromethane layers. Discard the aqueous layer.

At this point, the extract may be treated in one of two ways, depending upon time constraints.

Option 1: Dry the dichloromethane extract over anhydrous sodium sulfate for a minimum of 30 minutes. Quantitatively transfer the extract with dichloromethane rinses to a round bottom flask and use this flask for the Soxhlet extraction of the particulate as described below.

Option 2: Dry the dichloromethane extract over anhydrous sodium sulfate for a minimum of 30 minutes. Quantitatively transfer the extract with dichloromethane rinses to a round bottom flask. With dichloromethane rinses, quantitatively add the concentrated particulate extract from the Soxhlet procedure below to the filtrate extract. Concentrate the combined extract to 1–2 mL by rotary evaporation.

5.3.3.2 Particulate

Place a layer of silica or anhydrous sodium sulfate (2 g for small thimbles, 5 g for large thimbles, neutral, baked) into a clean Soxhlet thimble. Transfer the dried filter/filter bed/particulate with solvent rinses (use extraction solvent) into the thimble. Add the rinses to the Soxhlet body. Place the Soxhlet thimble in the Soxhlet apparatus.

Option 1: Extract using use the round bottom flask containing the supernatant extract. Add anti-bumping granules (4–5 granules).

Option 2: Add dichloromethane (300 mL) to the Soxhlet's round bottom flask. Add anti-bumping granules (4–5 granules).

Heat the sample under reflux for a minimum of 16 hours; adjust as necessary to achieve a reflux rate of at least 4 cycles per hour.

Allow the solution to cool. Concentrate the extract by rotary evaporation (water bath <30 °C) to 1–2 mL (the extract is ready to be combined with the supernatant extract if option 2 was chosen. Combine the extracts and concentrate to 1–2 mL by rotary evaporation). Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL (50 µL if toluene present) under a gentle stream of nitrogen. Make the volume to 1 mL with hexane.

The extract is ready for the column cleanup procedure (Section 6.0). If only coplanar PCBs are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B).

Should problems be encountered for liquid-liquid extraction of complex samples consult SLA-124 *Liquid-Liquid Extraction Supplemental Techniques*.

5.4 Milk Extraction – Solvent Extraction Procedure

This extraction procedure is applicable to the matrix and sample size listed below

Matrix	Sample Size
Milk	40–50 g

Accurately weigh a subsample of milk (to at least two decimals) into a 500 mL separatory funnel. Add an aliquot(s) of surrogate standard(s) which have been dissolved in ~1 mL acetone, and allow to equilibrate for at least 30 minutes. Add the sample to 200 mL of 2:1 acetone:hexane. Shake the mixture for 2 minutes and allow the layers to separate.

Transfer the aqueous (bottom) layer to an Erlenmeyer flask and the hexane (top) layer to a separate Erlenmeyer. Return the aqueous layer to the separatory funnel and repeat the extraction with hexane (200 mL). Combine the hexane extracts. Discard the aqueous layer.

Return the hexane extracts to the separatory funnel and wash by shaking with ultra pure water (2 x 50 mL).

Drain the hexane extract into an Erlenmeyer flask and dry over anhydrous granular sodium sulfate for a minimum of 30 minutes.

a) **If a lipid determination is required**, concentrate the extract to 5 mL by rotary evaporation (<30 °C) and carry out the lipid determination according to SLA-020 “Gravimetric Lipid Determination by Weight of Extract”. Following lipid analysis, reduce the volume of the final extract to 1 mL by rotary evaporation (<30 °C). Add hexane (1 mL) to the extract for gel permeation column cleanup (Section 6.1.1).

b) **If a lipid analysis is not required**, transfer the extract to a 500 mL round bottom flask.

Concentrate the extract to ~1 mL by rotary evaporation. Add 1 mL dichloromethane.

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Collect the first fraction and discard. Collect the second fraction in a round bottom flask.

Concentrate the extract by rotary evaporation (water bath <30 °C) to 1–2 mL. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream. Make the volume to 1 mL with hexane.

The extract is ready for chromatographic cleanup procedure (Section 6.0). If only coplanar PCBs are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B).

Should problems be encountered for liquid-liquid extraction of complex samples consult SGS AXYS SOP SLA-124 *Liquid-Liquid Extraction Supplemental Techniques*.

5.5 Blood Extraction Procedure

This extraction procedure is applicable to the matrices and sample size listed below.

Matrix	Sample Size
Whole Blood Serum Plasma	10–20 g

Refer to the SGS AXYS document SLA-082, *Handling of Human Biohazards*, for details on the safety precautions required when handling blood samples. Use caution when using bleach to decontaminate equipment used in the analysis of human biohazards and **avoid contact between acid and bleach.** Note that ammonium sulfate, used in the extraction below, is an acidic solution.

Extraction Procedures

1. Accurately weigh to a minimum of two decimals, a sample into a round bottom flask (250 mL). Add an aliquot of surrogate standard solution, cover the flask with clean aluminum foil and allow the solution to equilibrate for at least 30 minutes.
2. Add ethanol, hexane and saturated ammonium sulfate to the sample and extract by shaking on the shaker table for 30 minutes. Refer to the Batch List for the volume of reagents to use.

   NOTE: The proportion of reagents used is critical to complete extraction of analytes. The minimum sample size required is 10 g and the volumes of reagents are scaled to sample size in a ratio of sample:ethanol:saturated ammonium sulfate:hexane of 1:1:1:3. An alternate procedure for sample sizes less than 10 g, using 10 mL ethanol, 50 mL hexane and 10 mL saturated ammonium may be applied on a custom basis to meet client or historical project requirements - this option requires written pre-approval of the Project Manager including confirmation that the client has been made aware of potential solvent-system effects on gravimetric determination of lipids in blood matrices.
3. Decant the hexane layer into a separatory funnel (500 mL).
4. Add additional hexane (100 mL) to the aqueous layer in the round bottom flask and repeat the extraction step. Add the hexane layer to the hexane in the separatory funnel. Discard the aqueous phase.
5. Wash the hexane extracts by shaking with ultra pure water (2 x 50 mL) to remove residual ethanol. Discard the aqueous layer.
6. Transfer the hexane extract to an Erlenmeyer flask and dry over anhydrous sodium sulfate for a minimum of 30 minutes.
7. Refer to the Batch List to determine whether a lipid determination is required. If so, carry out the following procedures:
   Lipid Determination: Proceed with the lipid analysis following the procedures described in SLA-020 "*Gravimetric Lipid Determination by Weight of Extract*".

8. Quantitatively transfer the extract with hexane rinses to a clean round bottom flask.
9. The extract is ready for cleanup procedures (Section 6.0). NOTE: The E2 pesticide fraction (Section 6.2.1) must undergo additional cleanup on a Biobead column (Section 6.3.1). If only coplanar PCBs are required, the extract can proceed directly to coplanar PCB chromatographic cleanup step (Appendix B).

Should problems be encountered for liquid-liquid extraction of complex samples consult SGS AXYS SOP SLA-124 *Liquid-Liquid Extraction Supplemental Techniques*.

5.6 XAD-2 Columns (Resin and Filter) Extraction

This procedure is applicable to the analysis of an XAD-2 column. The analysis of an XAD-2 column usually consists of two analyses, as the filter and XAD-2 resin are extracted and analyzed separately. Upon client request, the filter and resin extracts may be combined to form a single extract.

The occasional formation of an interference during the extraction procedure and the high boiling point of the toluene may limit the quantification of PCBs 1 through 15 in the filter matrix.

5.6.1 Sample Handling Procedures

Refer to document SLA-043 for details of sample handling procedures. Remove the water from the resin as described in Section 5.6.2. If the sample consists of a large amount of XAD-2 resin or multiple filters, it may be necessary to use more than one Soxhlet apparatus to extract the sample. Distribute the dried XAD-2 resin (Section 5.6.2) or filters evenly amongst the required number of Soxhlets. Spike the sample in multiple Soxhlets with surrogate standard as follows:

1. Dilute the aliquot of surrogate standard in a centrifuge tube with acetone (5 mL). Ensure the solution is homogeneous by using a disposable pipette to withdraw and expel the solution several times.
2. Use a disposable pipette to distribute the surrogate solution evenly amongst the Soxhlets.
3. Rinse the centrifuge tube with acetone (5 mL). Mix the rinsate in the centrifuge tube by vortex mixing. Distribute the rinsate evenly amongst the Soxhlets. Repeat the rinse step once more with another 5 mL acetone.
4. Once the extraction is complete, combine the extracts.

5.6.2 XAD-2 Resin – Drying Procedure

1. Place a pre-cleaned Soxhlet thimble into a beaker.
2. Quantitatively transfer the XAD-2 resin to the pre-cleaned thimble with ultra pure water and allow to drain.
3. Rinse the resin with ultra pure water and allow to drain. Discard the water.

5.6.3 XAD-2 Resin Extraction

Add the appropriate volume of solvents to make up either 300 mL or 600 mL of dichloromethane (as required for the size of Soxhlet body) to the round bottom flask of the Soxhlet apparatus. Add 4–5 anti-bumping granules to the flask. If the extract is being analyzed for multiple compound types, use 80:20 toluene:acetone as the extraction solvent.

Add a small bed of solvent-rinsed glass wool to the bottom of the Soxhlet body to prevent clogging of the siphon tube during the reflux procedure. Spike an aliquot of the

surrogate solution (diluted in 1 mL acetone) onto the XAD-2 resin in the thimble and allow to equilibrate for at least 30 minutes. If multiple analyses are required, it may be acceptable to spike some of the surrogates after the extraction and extract splitting procedures (Section 5.9) to avoid surrogate solution incompatibilities. Refer to the Batch List for spiking instructions.

Soxhlet extract the sample for 16 hours using dichloromethane (or 80:20 toluene:acetone if multiple analyses are required) as the solvent; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Allow the solution to cool. Remove water from the extract (Section 5.6.3.1).

EXTREMELY IMPORTANT – DO NOT CONCENTRATE THESE SAMPLES BY ROTARY EVAPORATION WITHOUT A WATER REMOVAL STEP.

5.6.3.1 Removal of Water from XAD-2 Resin Extracts

The water removal step depends on the amount of visible water.

1. If less than 3 mL of water is visible, transfer the entire extract to an Erlenmeyer flask.

2. If greater than 3 mL of water is visible, transfer the extract to a separatory funnel and drain the water from the sample extract. Discard the water. Quantitatively transfer the extract to an Erlenmeyer flask.

3. Dry the extract over anhydrous granular sodium sulfate. Return the extract to the round bottom flask with complete dichloromethane rinses. Concentrate the extract by rotary evaporation to 1–2 mL for extract splitting. If a 1000 mL round bottom flask was used for the Soxhlet extraction, transfer the extract with dichloromethane rinses to a 500 mL round bottom flask during the evaporation process. The extract is ready to be gravimetrically split according to procedures in Section 5.9 of this method. Refer to the Batch List for spiking and extract splitting instructions.

5.6.4 Filter Soxhlet Extraction

This extraction procedure is applicable to wet glass fibre filters or filter cartridges (wound glass filters).

Place the filters or filter cartridge directly into a Soxhlet body. Use a large Soxhlet body to hold the filter(s); thimbles are not required. Use a 1000 mL round bottom flask for the solvent.

Spike an aliquot of the surrogate solution onto the filter(s) in the Soxhlet. If multiple analyses are required, it may be acceptable to spike some of the surrogates after the extraction and extract splitting procedures (Section 5.9) to avoid surrogate solution incompatibilities. Refer to the Batch List for spiking instructions.

Assemble the Soxhlet apparatus with a Dean Stark adapter. Soxhlet extract the filter(s) or filter cartridge until all the water is removed from the sample (i.e., no water collecting in the

sidearm) with 700 mL of toluene; adjust as necessary to achieve a reflux rate of a minimum of 4 cycles per hour. Check the volume of water in the sidearm periodically to ensure that it does not fill completely. Drain the water from the sidearm as necessary during the extraction process and top up with toluene as necessary.

When the extraction has finished, drain the water from the receiver. Monitor the amount of water collected, typically 50–150 mL should be present. Contact the Lab Supervisor if the amount of water is anomalous. Remove the Dean Stark adapter and continue with the Soxhlet extraction for another 8–12 hours. Allow the solution to cool. Concentrate by rotary evaporation to 1–2 mL. The extract is ready to be gravimetrically split according to procedures in Section 5.9. Refer to the Batch List for spiking and extract splitting instructions.

5.7 Ambient Air (PUF and Filter) Extraction and Particulate Filter Extraction Procedure

Matrix	Sample Size
Air Sample	PUF + Filter(s)
Particulate Filter Paper	1 Filter (dry)

This method is applicable to the analysis of ambient air samples consisting of a PUF and associated filter(s). The PUF and filter(s) are extracted together as one sample. The method is also applicable to particulate filter papers received dry.

5.7.1 Sample Preparation

Particulate Filter Papers (dry)

Handle the particulate filter paper(s) with clean solvent-rinsed forceps. Fold the filter paper(s) to fit into a Soxhlet thimble for extraction. Using clean forceps place the filter(s) into a Soxhlet thimble that has a layer of neutral silica or granular anhydrous sodium sulfate (baked, 2 g for small thimbles, 5 g for large thimbles) in the bottom. Be sure that the level of the filter in the thimble is not higher than the height of the siphon arm.

PUF/Filter

Using clean solvent-rinsed forceps, transfer the PUF and filter(s) to a Soxhlet thimble that has a layer of neutral silica or granular anhydrous sodium sulfate (baked, 2 g for small thimbles, 5 g for large thimbles) in the bottom. Be sure that the level of the filter in the thimble is not higher than the height of the siphon arm.

5.7.2 Soxhlet Extraction Procedure

Place the Soxhlet thimble containing the PUF and filter(s) in a Soxhlet apparatus. If a single analysis is required, spike the surrogate solution(s) onto the sample prior to starting the extraction. If multiple analyses are required, refer to the batch List for surrogate spiking instructions. Heat the sample under reflux for a minimum of 16 hours using dichloromethane (or 80:20 toluene:acetone if multiple analyses) as the solvent; adjust as necessary to achieve a reflux rate of 4 cycles per hour.

Allow the extract to cool and concentrate by rotary evaporation to 1 mL. Gravimetrically split the extract (Section 5.9) into two equal portions, one for the PCB/pesticide analysis and one as back-up. If multiple analyses are required, refer to the Batch List for extract splitting instructions.

5.8 Samples Submitted as Extracts

This extraction procedure is applicable to the matrix listed below.

Matrix	Sample Size
Solvent Extract	One Extract

5.8.1 Sample Preparation

Determine the weight of the extract as received as follows:

- Weigh the extract to at least two decimals (three decimals if extract is below 1 g) in the original container (usually jar or ampoule).
- Transfer the extract to a centrifuge tube (if received in an ampoule) or to an Erlenmeyer flask with solvent rinses using the same solvent as in the sample.
- Re-weigh the original container once it has air-dried.
- Record all weights on the worksheet.
- Calculate the weight of the extract.

Determine from the Batch List if the sample requires a single analysis or multiple analyses. Check the Batch List for detailed splitting and surrogate spiking instructions.

PCB/Pesticide Analysis Only

If only PCB/pesticide analysis is required, add aliquots of surrogate standard solutions to the extract and allow to equilibrate for 30 minutes. Dry the extract according to procedures in Section 5.8.2.

Multiple Analyses

If multiple analyses are required, the surrogate standards may be spiked before or after extract drying and splitting procedures. Refer to the Batch List for detailed splitting and surrogate spiking instructions. Dry the extract according to procedures in Section 5.8.2.

5.8.2 Extract Drying Procedures

Inspect the extract for the presence of water.

1. If less than 3 mL of water is visible, transfer the entire extract to an Erlenmeyer flask with solvent rinses (the same solvent as the sample). Proceed to Step 3.
2. If greater than 3 mL of water is visible, transfer the extract to a separatory funnel with solvent rinses (same solvent as sample) and separate water from the solvent. Save the solvent layer in a clean Erlenmeyer flask. Add solvent (25–50 mL) to the aqueous layer in the separatory funnel and shake for 2 minutes. Allow the mixture to separate, and draw off the organic layer. Repeat the extraction procedure, and discard the aqueous layer. Combine solvent extracts with the original solvent extract in the Erlenmeyer. Proceed to Step 3.
3. Dry the extract over anhydrous granular sodium sulfate. Quantitatively transfer the extract to a round bottom flask with solvent rinses. Concentrate the extract by rotary evaporation to 1–2 mL for extract splitting. Refer to the Batch List for extract splitting instructions. The extract is ready to be gravimetrically split according to procedures in Section 5.9.

5.9 Gravimetric Splitting Procedures

Gravimetrically split the extract, as described in SLA-123 “Splitting of Sample Extracts”, into two portions; one for the PCB/pesticide analysis and the other as backup. If multiple analyses are required, the extract may be split into many portions. Refer to the Batch List for extract splitting instructions.

6. COLUMN CLEANUP PROCEDURES

If the sample is not a hexane extract, a solvent exchange to hexane must be done before proceeding to column cleanup procedures. To exchange solvent, transfer the extract to a centrifuge tube with hexane and evaporate to 300 µL (50 µL if toluene or solvents other than DCM or alkanes are present) under a stream of nitrogen. Add hexane (1 mL).

6.1 Optional Cleanup Procedures

Extracts may be subject to the following additional cleanup procedures as necessary (e.g., the extract appears discoloured) prior to the Florisil column cleanup (Section 6.2). If pesticide analysis is required, only the gel permeation column (Section 6.1.1) can be used.

6.1.1 Gel Permeation Cleanup (**mandatory** for all tissue matrices)

Load the extract onto a Biobead SX-3 gel permeation column with 1:1 dichloromethane:hexane and elute with 1:1 dichloromethane:hexane at 5 mL/min. Refer to the most recent Biobead cutpoint determination for the elution volume. Collect the first fraction and discard. Collect the second fraction and evaporate to a small volume by rotary evaporation. Transfer to extract to a centrifuge tube with hexane rinses. Concentrate the extract under a gentle stream of nitrogen to 1–2 mL if only PCB and E1 pesticides are required, or to 300 µL if both E1 and E2 fractions are required. The extract is ready for the Florisil column cleanup procedure (Section 6.2).

6.1.2 Layered Acid/Base Silica Column Cleanup

Note: The following cleanup procedure cannot be used if pesticide analysis is required. If pesticide analysis is required, proceed to Section 6.2.

Load the extract onto a layered acid/base silica column (as described in Section 3.6.3) with rinses (2 x 1 mL hexane). Elute the column with hexane (100 mL) and collect all the eluate. Concentrate the extract to a small volume by rotary evaporation (water bath <30 °C). Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 300 µL under a gentle stream of nitrogen. Make the volume to 1 mL with hexane. The extract is ready for the column cleanup procedure (Section 6.2).

6.2 Florisil Column Cleanup (Default Procedure)

Sample extracts are cleaned up on a Florisil column for which column cutpoints are regularly determined. The following fractions are eluted from the Florisil column:

- E1 contains non-polar and moderately polar chlorinated pesticides, PCB congeners including coplanar PCBs (NOS), chlorobenzenes, and Toxaphene.
- E2 contains the more polar chlorinated pesticides.

The extract must be hexane, if not, a solvent exchange to hexane must be done before proceeding to the Florisil column cleanup procedures. To exchange solvent, transfer the extract to a centrifuge tube with hexane and evaporate to 300 µL (50 µL if toluene is present) under a stream of nitrogen. Make the volume to 1 mL with hexane.

Some extracts require additional cleanup procedures to remove interferences. These procedures are described in Section 6.3.

The additional cleanup procedures for the analysis of coplanar PCBs by HRGC/HRMS are described in Appendix B.

6.2.1 Florisil Column Cleanup of E1 and E2

Quantitatively transfer the extract to a Florisil column (8 grams, 2.0–2.1% deactivated) prepared as described in Section 3.6.1. Rinse the container (2 x 1 mL) using a portion of the elution solvent volume for the E1 fraction, and add the rinses to the column. Elute the column with 10:90 dichloromethane:hexane (E1) and collect the eluate in a round bottom flask. Elute the column with dichloromethane (E2) and collect the eluate in a round bottom flask. Use the elution volumes determined from the Florisil cutpoint determination of the particular batch of Florisil. Typical cutpoint are as follows:

E1	10:90 dichloromethane:hexane	45 mL
E2	dichloromethane	50 mL

E1 Fraction:

Concentrate the E1 fraction to a small volume (~1 mL) by rotary evaporation. Add activated copper to remove sulfur. Transfer the extract to a centrifuge tube with solvent rinses. The first rinse must be with toluene; all subsequent rinses are with hexane. Concentrate the extract to ~350 µL under a stream of nitrogen.

E2 Fraction:

Add 1 mL toluene to the extract as a “keeper”. Concentrate the extract to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to ~350 µL under a stream of nitrogen.

Both Fractions (separately):

Transfer the extract to an autosampler vial. Rinse the centrifuge tube with hexane, vortex, and add the rinse to the autosampler vial. Concentrate the extract to no less than 200 µL under a gentle stream of nitrogen. Repeat the rinse and concentration sequence once more, taking care not to reduce the volume in the autosampler vial to less than 200 µL until after the final rinse. Concentrate the extract to ~60 µL (~160 µL for fraction E2 tissue and solid sample extracts). Add an aliquot of the appropriate recovery standard to each extract. Adjust the volume with toluene to 100 µL, except for fraction E2 tissue and solid sample extracts where the final extract volume should be adjusted to 200 µL. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 7.0).

E1 is analyzed by GC/MS for some or all of the following: Aroclors; less-polar chlorinated pesticides; Toxaphene, PCB congeners; and chlorobenzenes (Section 7.1).

E2 is analyzed by GC/ECD for the more polar pesticides (Section 7.2).

If coplanar PCBs are required, proceed to the separation and cleanup of coplanar PCBs (Appendix B).

6.3 Additional Cleanup Procedures

Any of the following cleanup procedures may be used in addition to the routine cleanup procedures. Depending on the nature of the matrix or extract, an analyst may perform additional cleanup procedures prior to instrumental analysis.

6.3.1 Biobead Column

Uncap the autosampler vial. Transfer the extract to a centrifuge tube with enough 1:1 dichloromethane:hexane to make the volume to 2 mL. Load the extract onto a Biobead column prepared as described in Section 3.6.2 and elute with 1:1 dichloromethane:hexane. Refer to the most recent Biobead cutpoint for the volume of solvent to collect.

Collect the first fraction and discard. Collect the second fraction into a round bottom flask. Add 1 mL toluene as a "keeper" and concentrate to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to ~350 µL under a stream of nitrogen.

Transfer the extract to an autosampler vial. Rinse the centrifuge tube with hexane, vortex, and add the rinse to the autosampler vial. Concentrate the extract to no less than 200 µL under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more, taking care not to reduce the volume in the autosampler vial to less than 200 µL until after the final rinse. Concentrate the extract to 100 µL, except for fraction E2 tissue and solid sample extracts where the final extract volume should be 200 µL. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 7.0).

6.3.2 Alumina Column

This cleanup procedure cannot be used if pesticide analysis of the extract is required.

Uncap the autosampler vial. Transfer the extract to a centrifuge tube with hexane rinses. Concentrate the extract to 50 µL under a gentle stream of nitrogen, then add enough hexane to make the volume to 1 mL. If required, add activated copper to the extract to remove sulfur. Load the extract in hexane onto an alumina column (6 g, 1% deactivated) prepared as described in Section 3.6.4. Elute the column with hexane (15 mL, discard) followed by 1:1 dichloromethane:hexane (35 mL, retain).

Add 1 mL of toluene as a "keeper", and concentrate the eluate to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to ~350 µL under a stream of nitrogen.

Transfer the extract to an autosampler vial. Rinse the centrifuge tube with hexane, vortex, and add the rinse to the autosampler vial. Concentrate the extract to no less than 200 µL under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more, taking care not to reduce the volume in the autosampler vial to less than 200 µL until after the final rinse. Concentrate the extract to 100 µL. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 7.0).

6.3.3 Acid Wash in a Separatory Funnel

CAUTION: The following cleanup procedure must not be used for Florisil E2 fractions. It can be used for Florisil E1 fraction PCBs and pesticides.

- **Ensure that the extract contains no residual water.**
- **Limit the contact time between the extract and H_2SO_4 to no more than 15 minutes per wash.**

Transfer the Florisil E1 fraction with hexane rinses to a 125 mL separatory funnel. Make the volume up to 50 mL with hexane. Add concentrated sulfuric acid (30 mL) to the extract and agitate the mixture gently (10–15 inversions) to avoid emulsions. Allow the layers to separate. Drain the lower (acidic) layer and discard. If the aqueous layer is strongly coloured repeat the procedure up to 3 more washes with sulfuric acid. The total contact time with acid must not be more than 15 minutes.

Wash the extract by shaking with ultra purewater. Drain the aqueous layer and discard. Transfer the organic layer to a clean 250 mL Erlenmeyer flask and dry over anhydrous sodium sulfate (5–10 g, 10–15 minutes). Transfer the extract to a round bottom flask and add 1 mL toluene as a “keeper”. Concentrate the extract to 1 mL by rotary evaporation.

Prepare a glass wool column by placing a small amount of silanized glass wool into the end of a clean Pasteur pipette and rinsing the column with toluene followed by hexane. Elute the extract through the glass wool column and collect in a centrifuge tube. Concentrate the extract to 350 µL under a stream of nitrogen. Concentrate the extract after adding each rinse, taking care not to reduce the volume in the centrifuge tube to less than 200 µL to avoid the risk of losing the lighter (more volatile) compounds.

Transfer the extract to an autosampler vial. Rinse the centrifuge tube with hexane, vortex, and add the rinse to the autosampler vial. Concentrate the extract to 350 µL under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more, taking care not to reduce the volume in the autosampler vial to less than 200 µL until after the final rinse. Concentrate the extract to 100 µL. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 7.0).

6.3.4 Acid Wash in a Centrifuge Tube

This optional cleanup procedure may be applied to Florisil E1 fractions in response to, or in anticipation of, matrix interferences generally seen in solid matrices. The procedure must not be used for Florisil E2 fractions. No negative impact has been observed for PCBs in the E1 fraction.

Transfer the Florisil E1 fraction with hexane rinses to a 15 mL centrifuge tube. Reduce the volume to ~1-2 mL in hexane. Add concentrated sulfuric acid (~1 mL) to the extract and vortex the mixture thoroughly. Let stand (no more than 15 minutes). Draw off the lower (acidic) layer and discard. **Note:** If pesticide analysis is required, do not repeat the procedure with additional washes with sulfuric acid. If the aqueous layer is strongly coloured and only PCB analysis is required, repeat the procedure with up to 3 more washes with sulfuric acid.

Wash the extract by adding ~1 mL ultrapure water and vortex mixing. Draw off the lower (aqueous) layer and discard. Repeat the wash with a second ~1 mL portion of ultra pure water.

Prepare a glass wool drying column by placing a small amount of silanized glass wool into the end of a clean Pasteur pipette and adding a few cm of anhydrous sodium sulfate. Elute the extract with hexane rinses through the drying column and collect in a centrifuge tube.

Perform any further required clean-up (if required) and concentrate and microvial the extract as normal.

7. INSTRUMENTAL ANALYSIS

7.1 Quadrupole GC/MS Analysis of E1 for PCBs (Aroclors and Congeners), Technical Toxaphene, Pesticides and Chlorobenzenes

Mass Calibration and Resolution

Tuning and mass calibration is performed as described in SIN-029 every 3 days or as required to meet daily tuning check requirements. The PFTBA (FC043) procedure is used. Tuning is checked daily by verifying mass calibration, mass resolution and peak relative response as described in Appendix C.

Sensitivity

The sensitivity of the GC/MS is checked on every instrument log and after any instrument maintenance. Sensitivity specifications are summarized in Table 8 for E1 fraction pesticides and Table 9 for PCB congeners.

GC Resolution

The GC resolution is checked daily. QC specifications are summarized in Table 8 for E1 fraction pesticides and Table 9 for PCB congeners.

DDT Breakdown

The percent breakdown of DDT is checked before every batch of samples or at least every 12 hours. The breakdown is monitored by checking the breakdown of $^{13}C_{12}$-labeled DDT in the solvent blank. The percent breakdown is calculated as follows:

$$\%\ \text{Breakdown} = 100 \times \frac{\left(\text{sum of degradation peak area } (^{13}C_{12}\text{-DDD} + {}^{13}C_{12}\text{-DDE})\right)}{\left(\text{sum of all peak areas } (^{13}C_{12}\text{-DDT} + {}^{13}C_{12}\text{-DDE} + {}^{13}C_{12}\text{-DDD})\right)}$$

If any of the QC criteria are not met, the GC column and/or injector must be retuned or serviced before continuing with the analysis.

Calibration

Initial calibration (the method default procedure) is performed using a series of five solutions that encompass the working concentration range of the instrument. The initial calibration solutions contain a suite of labeled surrogates, recovery standards and target compounds. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution. The frequency of initial calibrations is specified in SIN-028. Depending on the request of the client, either continuing calibration protocols or bracketing calibration protocols are followed. Continuing calibration protocols use the mean RRFs determined from the initial calibration to calculate the analyte concentrations. The optional bracketing calibration procedure uses mean RRFs from the analysis of the mid-level calibration solutions analyzed before and after the batch of samples to calculate analyte concentrations.

Analysis

Analysis of the E1 fraction is done on a low-resolution mass spectrometer (LRMS) equipped with a gas chromatograph (GC), and an auto-sampler, operating on manufacturer's software. A J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness) is coupled directly to the MS source. The MS is operated at a unit mass resolution in the electron ionization (EI) mode using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard. A splitless/split injection sequence is used. The ions acquired are listed in Table 10.

A typical extract volume is 100 µL; 1.0 µL or 2.0 µL is injected. If necessary, extracts are diluted with solvent to bring all target responses within the calibration range.

Typical operating conditions for the GC/MS are presented below.

GC Temperature Program		**General GC Conditions**	
Temp (°C)	50	Injector Temp (°C)	280
Hold time (min)	0.95	Injector	Splitless, 2 min
Rate (C° min^{-1})	13.3	Carrier Gas	Helium
Temp (°C)	150	Maximum Temp (°C)	325
Hold time (min)	0		
Rate (C° min^{-1})	3.47	**MS Conditions**	
Temp (°C)	250	Source Temp (°C)	286
Hold time (min)	0	Quadrupole Temp (°C)	150
Rate (C° min^{-1})	57.81	Electron Energy (eV)	70
Temp (°C)	320	Mass Resolution	Unit
Hold time (min)	9.51	Detector Voltage (V)	Variable
Total Run time (min)	48.01		

The IA Chemist is referred to Instrument Method MIN-007 (E1 PCB) for further details of the instrumental analysis. When Technical Toxaphene is required, the above GC program is used, and the masses for Toxaphene are added to the acquisition program (Method CLGR0621.M).

Analysis Sequence

The sequence in which a batch of samples is analyzed, after successful calibration, is as follows:

Calibration Solution
Toxaphene Calibration solution (where required)
Reference Sample
Toluene Blank (containing $^{13}C_{12}$-p,p'-DDT)
Procedural Blank
Samples
Calibration Solution
Toxaphene Calibration solution (where required)

7.2 GC/ECD Analysis of E2 for Pesticides

Sensitivity

The sensitivity of the GC/ECD is checked on every instrument log and after any instrument maintenance. Sensitivity specifications are summarized in Table 7 for E2 fraction pesticides.

GC Resolution

The GC resolution is checked daily. QC specifications are summarized in Table 7 for E2 fraction pesticides.

Endrin Breakdown

The breakdown of endrin is checked before every bracket of samples or at least every 12 hours. The percent breakdown is determined from the analysis of the solution that contains $^{13}C_{12}$-endrin (but not its breakdown products) and is calculated as follows:

$$\%\,\text{Breakdown} = \frac{100(\ \text{Sum of degradation peak areas}(\ ^{13}\text{C - endrin ketone} + {}^{13}\text{C - endrin aldehyde})\ )}{(\ \text{Sum of all peak areas}(\ ^{13}\text{C - endrin} + {}^{13}\text{C - endrin ketone} + {}^{13}\text{C - endrin aldehyde})\ )}$$

If any of the QC criteria are not met, the GC column and/or injector must be serviced before continuing with the analysis.

Calibration

Two calibration protocols are described below, the 'initial' multi-level calibration procedure is the method default procedure, the 'bracketing' calibration procedure is an optional procedure.

Initial calibration is performed using a series of five solutions that encompass the working concentration range of the instrument. The initial calibration solutions contain a suite of labeled surrogates, recovery standards and target compounds. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution. The frequency of initial calibrations is specified in SIN-028. Depending on the request of the client, either continuing calibration protocols or bracketing calibration protocols are followed. Continuing calibration protocols use the mean RRFs determined from the initial calibration to calculate the analyte concentrations.

Optional 'bracketing' calibration procedures use mean RRFs from the analysis of the mid-level calibration solutions analyzed before and after the batch of samples to calculate analyte concentrations.

Analysis

Analysis of the E2 fraction is done on a ^{63}Ni electron capture detector (ECD) equipped with a gas chromatograph (GC), an automatic injector, and running manufacturer's software. A J&W DB-5 capillary column (60 m, 0.25 mm i.d., 0.10 µm film thickness) is coupled directly to the ECD source. Where required, confirmation is performed by simultaneous analysis on a J&W DB-17MS capillary column (30 m, 0.25 mm i.d., 0.25 µm film thickness) coupled to a second ECD source.

A typical extract volume is 100 µL, except for tissue and solid sample E2 extracts where the final extract volume is 200 µL; 1.0 µL is injected. If necessary, extracts are diluted with solvent to bring all target responses within the calibration range.

Typical operating conditions for the GC/ECD are presented below.

GC Temperature Program		**General GC Conditions**	
Temp (°C)	85	Injector Temp (°C)	140
Hold time (min)	1.0	Injector	Splitless, 2 min
Rate 9 (°C min^{-1})	25	Carrier Gas	Helium
Temp (°C)	190	Maximum Temp (°C)	325
Hold time (min)	0		
Rate (°C min^{-1})	1.3		
Temp (°C)	230		
Hold time (min)	1.33		
Rate (°C min^{-1})	25		
Temp (°C)	315		
Hold time (min)	1.3		

The IA Chemist is referred to Instrument Method MIN-007 (E2) for further details of the instrumental analysis.

Analysis Sequence

The typical sequence in which a batch of samples is analyzed, after successful calibration, is as follows:

Endrin Breakdown Solution[1]
Calibration Solution
Reference Sample
Solvent Blank
Procedural Blank
Samples
Calibration Solution

[1] The endrin breakdown solution can also be injected after the initial calibration solution.

8. QUALITATIVE AND QUANTITATIVE DETERMINATION

8.1 GC/LRMS Peak Identification

A chromatographic peak is identified as a target compound if the following criteria are met for the quantification and confirmation ions:

1. Peak responses must be at least three times the background noise level;
2. The relative retention time must be within the window predicted from the initial calibration runs and the surrogate standard retention times. Additionally the authentic compound must elute after its labeled analogue;
3. Peak maxima for quantification and confirmation ions must coincide within two seconds;
4. The relative ion abundance ratios must be within 20% of the theoretical except for oxy-chlordane and technical Toxaphene components, for which the ion ratios are determined empirically;
5. Any response at the masses for PCB congeners containing one or two more chlorine atoms than the target PCB congener that occurs at the same retention time as the target PCB congener should be evaluated for possible effect. A fragment ion from a more highly chlorinated congener may enhance the response of the target congener. Where the target response may be significantly enhanced, the concentration is reported as a maximum possible value.

8.2 GC/ECD Peak Identification

A chromatographic peak is identified as a target compound as long as the following criteria are met for the quantification peak:

1. Peak responses must be at least three times the background noise level;
2. The peak must elute within the retention time window predicted from the calibration runs.
   - **Bracketing Cal:** The retention time shift from the opening calibration to the closing calibration must be ≤3 sec. The retention time shift of any sample within the bracket must be ≤3 sec of the opening calibration.
   - **Continuing Cal:** The retention time shifts of opening and closing CAL/VERs must be ≤15 sec on the initial calibration.
3. For dual column analysis the calculated concentrations from the two columns must agree within 40% RPD.

8.3 Concentrations

Target concentrations are determined with respect to a labeled surrogate as shown in Table 10 for E1 fraction pesticides and PCB congeners and Table 11 for E2 fraction pesticides. Mean relative response factors (RRF), determined from either a multi-level initial calibration series or a mid-level calibration standard run at the beginning and end of the samples, are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\underset{\text{(ng/g or ng/L)}}{\text{Concentration of Target}} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{concentration of Qt Std}}{\text{concentration of Target}}\right)$

and the Qt Std is either the surrogate or the internal standard

Concentration results for target compounds are recovery corrected by the method of quantification. Surrogate recoveries are determined similarly against the recovery (internal) standard and are used as general indicators of overall analytical quality.

8.4 Calculation of Aroclor Equivalent Concentrations

Aroclor equivalent concentrations may be calculated by converting the summed concentrations of a suite of characteristic PCB congeners to concentrations using empirical factors determined from the analysis of Aroclor mixtures[1].

Aroclor 1016[2] = the sum of PCB 8/18/31/28 concentrations multiplied by 3.0;
Aroclor 1221 = the sum of PCB 3/4/8 concentrations multiplied by 3.0;
Aroclor 1232 = the sum of PCB 8/18/28/31 concentrations multiplied by 5.0;
Aroclor 1242[2] = the sum of PCB 8/18/31/28 concentrations multiplied by 3.8;
Aroclor 1248 = the sum of PCB 66/44/49 concentrations multiplied by 5.5;
Aroclor 1254 = the sum of PCB 87/97/99 concentrations multiplied by 10;
Aroclor 1260 = the sum of PCB 183/180/170 concentrations multiplied by 6.0;

1. The congeners listed may have additional co-eluting congeners but these are insignificant with respect to the sums
2. Aroclors 1016 and 1242 may be reported as combined 1016/1242 using the 1242 factor if allowed by contract

Environmental samples with no clearly identified Aroclor signature are quantified as 1242/1254/1260 mixtures. Results may be reported as Aroclor 1248 instead of Aroclor 1242 and 1254 where the congener pattern clearly indicates this formulation. Other Aroclor formulations may be reported by calibration against the specific Aroclor solutions.

Where identification of specific Aroclors is required, sample extracts are reinjected alongside calibration (single point) standards of the Aroclor most closely matching the sample pattern and the Aroclor equivalents are quantified using the summed area responses of 3–10 congeners for each Aroclor identified.

8.5 Quantification of p,p'-DDE

An additional high level calibration standard containing p,p'-DDE (see Section 3.3.3) is analyzed during the initial calibration of the instrument to extend the calibration range for that compound. If the concentration of the high level p,p'-DDE standard is within 20% of the estimated concentration extrapolated from the initial calibration range, then the area response of p,p'-DDE is considered to be linear up to the concentration of the high level p,p'-DDE standard. Extracts with p,p'-DDE concentrations greater than the initial calibration range can be quantified without dilution. If the concentration of the high level p,p'-DDE standard does not meet the criteria, extracts with p,p'-DDE concentrations greater than the initial calibration range must be diluted prior to quantification.

8.6 Quantification of delta-HCH

The target analyte delta-HCH elutes primarily in the E2 fraction and is quantified from the E2 fraction analysis data using the recovery corrected procedure described in the method.

Upon specific demonstration of significant d-HCH content in the E1 fraction it is permissible, with prior project management approval, to remediate low E2 fraction recovery by quantifying d-HCH using a summation of the E1 and E2 fraction data as an alternative to re-analysis. For this procedure the concentrations are determined in each of the E1 and E2 fractions by quantification against the corresponding recovery standards (^{13}C-PCB-52 in the E1 fraction and ^{13}C-PCB-153 in the E2 fraction) and the summed concentrations are then recovery corrected with respect to d4-alpha-Endosulfan surrogate. The QC samples are processed similarly for accuracy demonstration.

8.7 Quantification of Technical Toxaphene

Technical Toxaphene is determined by summing the responses of five peaks and quantifying by internal standard quantification procedures. The calculations use the mean RRF determined from the calibration run(s). Table 12 presents typical retention times, the masses monitored and the quantification references.

8.8 Quantification of E2 Fraction Pesticides using Dual Column GC/ECD

Where analysis of E2 fraction pesticides is confirmed using the DB-17MS column, the data is reported as follows:

- If the analyte is not detected in either column, the concentration is reported as "ND", with the detection limit taken from the primary column;
- If the analyte is detected on only one of the columns, the concentration is reported as "ND", with the detection limit taken from the column with the non-detect;
- If the analyte is detected on both columns:
    - If %RPD$\leq$40%, the concentration and detection limit are reported from the primary column;

- If the %RPD>40%, the results are reported from the column with the lower concentration. The concentration result is flagged as a maximum possible value to indicate that it has not been confirmed as present.

8.9 Quantification of Technical Chlordane

Technical chlordane can be quantified by summing the concentrations of Oxychlordane, cis- and trans-Chlordane, and cis- and trans-Nonachlor, and multiplying by 2.5.

8.10 Optional High Concentration Sample Procedure

For samples with target concentrations that would exceed the working range of the method an extraction standard (PCB 204) is added to the sample prior to analysis instead of the labeled surrogates. The labeled surrogates are spiked to a split portion of the extract immediately after extraction and the analysis is carried through. The recoveries of the extraction standards are monitored to ensure quantitative recovery from the extraction step. Final sample concentrations are adjusted to reflect the extract split factor.

8.11 Optional Low Concentration Sample Procedure

The spiking level of labeled surrogate and recovery standards may be reduced by up to a factor of 10, and the extract volume reduced accordingly to a minimum volume of 20 µL.

8.12 Option for Non-Recovery Corrected Data

Where non-recovery corrected data is required to align with particular method procedures (i.e., EPA Method 608, 625, etc.) final concentrations may be calibrated with respect to the internal (recovery) standards added prior to instrument analysis. Where this option is employed the results must be flagged as non-recovery corrected.

8.13 PCB 11 Interference

The pesticide surrogate ^{13}C-gamma-HCH elutes shortly before native PCB 11 and produces peaks in the quantification and confirmation channels of PCB 11 (ions 222 and 224) that do not meet the ion ratio requirements for the native PCB. The pesticide interference sometimes elutes within the PCB 11 retention time window, i.e., ±3 sec of the retention time of PCB 11 in the calibration standard, adjusted to the retention time reference ^{13}C-PCB 8. The interference peak and PCB 11 are nevertheless typically well resolved in the windowing standard (see section 3.3.5 in MLA-007, or 'Other Specifications' in MIN-007) run towards the beginning of each analysis sequence. Provided adequate resolution between the interference peak and PCB 11 is demonstrated in the windowing standard, care should be taken not to select the interference peak as PCB 11.

9. REPORTING CRITERIA AND PRACTICES

Sample specific detection limits (SDL) are determined from the analysis data by converting the minimum detectable area to a concentration following the same procedures used to convert target peak responses to concentrations. The estimated minimum detectable area is determined as three times the height of the noise in the m/z channel of interest, converted to an area using the area height ratio of the corresponding labeled surrogate peak.

Table 10: Analyte Ions Monitored, Surrogates Used, and RRF Determination for E1 Pesticides and PCB Congeners by LR GC/MS

(Target compounds are listed below the labeled standard used for quantification. No entry in the "RRF Used" field designates an RRF derived from that same compound.)

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
^{13}C-PCB 52 -REC[1]	**23.27**	**302**	**304**	**130**	
$^{13}C_6$-1,4-DiCB	**7.57**	**154**	**152**	**156**	
1,3-DiCB	7.52	146	148	64	
1,4-DiCB	7.57	146	148	64	
1,2-DiCB	7.77	146	148	64	
$^{13}C_6$-1,2,3-TriCB	**9.63**	**190**	**188**	**312**	
1,3,5-TriCB	8.71	180	182	95.9	
1,2,4-TriCB	9.2	180	182	95.9	
1,2,3-TriCB	9.63	180	182	95.9	
$^{13}C_6$-1,2,3,4-TetCB	**11.77**	**224**	**222**	**208**	
1,2,3,5-/1,2,4,5-TetCB	11.09	216	214	78.2	
1,2,3,4-TetCB	11.77	216	214	78.2	
Hexachlorobutadiene	9.65	225	260	38.4	1,2,3,4-TetCB
$^{13}C_6$-PentaCB	**14.07**	**256**	**260**	**20.4**	
PentaCB	14.07	250	252	64	
$^{13}C_6$-HexaCB	**17.87**	**292**	**294**	**42.6**	
HexaCB	17.87	284	286	80	
$^{13}C_6$-beta HCH	**18.7**	**225**	**227**	**48**	
beta-HCH	18.7	219	217	78.2	
$^{13}C_6$-gamma HCH	**18.94**	**225**	**227**	**48**	
alpha-HCH	17.52	219	217	78.2	
gamma-HCH	18.94	219	217	78.2	
delta-HCH	20.01	219	217	78.2	
$^{13}C_{10}$-Heptachlor	**22.25**	**277**	**279**	**80**	
Heptachlor	22.28	272	270	50	
$^{13}C_{12}$-Aldrin	**23.92**	**270**	**272**	**64**	
Aldrin	23.94	263	261	62.5	
$^{13}C_{10}$-trans-Chlordane	**27.19**	**383**	**385**	**96**	
trans-Chlordane	27.2	373	375	95.9	
cis-Chlordane	28	373	375	95.9	
Octachlorostyrene	25.86	380	378	89.4	
Oxychlordane	26.04	185	115	120	

[1] REC = Recovery Standard, added just prior to Instrument analysis

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
$^{13}C_{10}$-trans-Nonachlor	**28.26**	**419**	**421**	**64**	
trans-Nonachlor	28.28	409	411	64	
cis-Nonachlor	31.5	409	411	64	
Mirex	37.98	272	270	52.1	
$^{13}C_{12}$-pp'-DDE	**29.21**	**330**	**328**	**78.2**	
o,p'-DDE	27.56	246	248	64	
p,p'-DDE	29.22	246	248	64	
$^{13}C_{12}$-PCB 138-REC[1]	**33.52**	**372**	**376**	**34**	
$^{13}C_{12}$-pp'-DDT	**33.32**	**247**	**249**	**64**	
o,p'-DDD	29.63	235	237	64	
p,p'-DDD	31.37	235	237	64	
o,p'-DDT	31.53	235	237	64.6	
p,p'-DDT	33.32	235	237	64	
$^{13}C_{12}$-PCB 52-REC[1]	**23.27**	**302**	**304**	**130**	
$^{13}C_{12}$-PCB 3	**15.03**	**200**	**202**	**31.9**	
PCB 1	13.63	188	190	32.1	
PCB 2	14.91	188	190	32.1	PCB 3
PCB 3	15.04	188	190	32.1	
$^{13}C_{12}$-PCB 8	**17.51**	**234**	**236**	**64**	
PCB 4/10	15.86	222	224	64.1	
PCB 7/9	16.84	222	224	64.1	PCB 8/5
PCB 6	17.28	222	224	64.1	PCB 8/5
PCB 8/5	17.52	222	224	64.1	
PCB 14	18.12	222	224	64.1	PCB 8/5
PCB 11	18.98	222	224	64.1	PCB 8/5
PCB-12/13	19.25	222	224	64.1	PCB 8/5
PCB 15	19.5	222	224	64.1	
$^{13}C_{12}$-PCB 28	**21.57**	**268**	**270**	**96**	
PCB 19	18.4	256	258	96.2	
PCB 30	18.77	256	258	96.2	PCB 18
PCB 18	19.4	256	258	96.2	
PCB 17	19.5	256	258	96.2	PCB 18
PCB 24/27	19.88	256	258	96.2	PCB 18
PCB 16/32	20.27	256	258	96.2	PCB 18
PCB 34/23	20.73	256	258	96.2	PCB 31
PCB 29	20.9	256	258	96.2	PCB 31
PCB 26	21.13	256	258	96.2	PCB 31
PCB 25	21.51	256	258	96.2	PCB 31
PCB 31	21.58	256	258	96.2	
PCB 28	22.03	256	258	96.2	
PCB 33/20/21	22.4	256	258	96.2	PCB 31

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 22	22.69	256	258	96.2	PCB 31
PCB 36	23.12	256	258	96.2	PCB 31
PCB 39	23.58	256	258	96.2	PCB 31
PCB 38	24.08	256	258	96.2	PCB 37
PCB 35	24.45	256	258	96.2	PCB 37
PCB 37	21.5	256	258	96.2	
$^{13}C_{12}$PCB 31- FS[1]	**21.51**	**270**	**272**	**33.2**	**PCB 31**
$^{13}C_{12}$-PCB 138-REC[1]	**33.52**	**372**	**376**	**34**	
$^{13}C_{12}$-PCB 101	**27.69**	**338**	**340**	**64**	
PCB 54	20.91	290	292	129.9	
PCB 50	21.53	290	292	129.9	PCB 54
PCB 53	22.09	290	292	129.9	PCB 52/73
PCB 51	22.32	290	292	129.9	PCB 52/73
PCB 45	22.66	290	292	129.9	PCB 52/73
PCB 46	23.05	290	292	129.9	PCB 52/73
PCB 69	23.18	290	292	129.9	PCB 52/73
PCB 52/73	23.29	290	292	129.9	
PCB 49/43	23.49	290	292	129.9	
PCB 47/48/75	23.65	290	292	129.9	PCB 52/73
PCB 65/62	23.82	290	292	129.9	PCB 52/73
PCB 44	24.3	290	292	129.9	
PCB 42/59	24.4	290	292	129.9	PCB 44
PCB 72	24.73	290	292	129.9	PCB 44
PCB 41/71/64/68	24.92	290	292	129.9	PCB 44
PCB 40	25.31	290	292	129.9	
PCB 57	25.39	290	292	129.9	PCB 40
PCB 67	25.62	290	292	129.9	PCB 40
PCB 58	25.79	290	292	129.9	PCB 40
PCB 63	25.91	290	292	129.9	PCB 66/80
PCB 74/61	26.11	290	292	129.9	PCB 66/80
PCB 70/76	26.28	290	292	129.9	PCB 66/80
PCB 66/80	26.45	290	292	129.9	
PCB 55	26.9	290	292	129.9	PCB 56/60
PCB 56/60	27.32	290	292	129.9	
PCB 79	28.03	290	292	129.9	PCB 56/60
PCB 78	28.55	290	292	129.9	PCB 56/60
PCB 81	29.06	290	292	129.9	PCB 56/60
PCB 77	29.56	290	292	129.9	PCB 56/60

[1] FS = Field Standard29.06

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 104	24.08	326	328	64.5	
PCB 96	25.14	326	328	64.5	PCB 95/93
PCB 103	25.34	326	328	64.5	PCB 95/93
PCB 100	25.62	326	328	64.5	PCB 95/93
PCB 94	26.03	326	328	64.5	PCB 95/93
PCB 98/102	26.39	326	328	64.5	PCB 95/93
PCB 95/93	26.51	326	328	64.5	
PCB 88/121	26.66	326	328	64.5	PCB 95/93
PCB 91	26.83	326	328	64.5	PCB 95/93
PCB 92	27.41	326	328	64.5	PCB 90/101/89
PCB 84	27.58	326	328	64.5	PCB 90/101/89
PCB 90/101/89	27.7	326	328	64.5	
PCB 113	27.88	326	328	64.5	PCB 90/101/89
PCB 99	27.97	326	328	64.5	
PCB 119	28.3	326	328	64.5	PCB 99
PCB 112	28.41	326	328	64.5	PCB 87/115/116
PCB 83/108	28.54	326	328	64.5	PCB 87/115/116
PCB 97/86	28.8	326	328	64.5	PCB 87/115/116
PCB 125	28.92	326	328	64.5	PCB 87/115/116
PCB 111/117	29	326	328	64.5	PCB 87/115/116
PCB 87/115/116	29.08	326	328	64.5	
PCB 85/120	29.28	326	328	64.5	PCB 87/115/116
PCB 110	29.58	326	328	64.5	
PCB 82	30.14	326	328	64.5	PCB 87/115/116
PCB 124	30.49	326	328	64.5	PCB 110
PCB 107/109	30.67	326	328	64.5	PCB 110
PCB 114	31.5	326	328	64.5	
PCB 122	31.63	326	328	64.5	PCB 114
PCB 105/127	32.3	326	328	64.5	
PCB 126	33.99	326	328	64.5	
$^{13}C_{12}$PCB 95- FS[1]	**26.52**	**338**	**340**	**64**	**PCB 95/93**
PCB 155	27.23	360	362	80.6	
PCB 150	28.33	360	362	80.6	PCB 149/139
PCB 152	28.71	360	362	80.6	PCB 149/139
PCB 145	29.04	360	362	80.6	PCB 149/139
PCB 148	29.26	360	362	80.6	PCB 149/139
PCB 136	29.41	360	362	80.6	PCB 149/139
PCB 154	29.64	360	362	80.6	PCB 149/139

[1] FS = Field Standard

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 151	30.22	360	362	80.6	
PCB 144/135	30.46	360	362	80.6	PCB 149/139
PCB 147	30.64	360	362	80.6	PCB 149/139
PCB 149/139	30.84	360	362	80.6	
PCB 140	30.99	360	362	80.6	PCB 149/139
PCB 134/143	31.31	360	362	80.6	PCB 149/139
PCB 133	31.5	360	362	80.6	PCB 149/139
PCB 131/142	31.63	360	362	80.6	PCB 149/139
PCB 165	31.72	360	362	80.6	PCB 153
PCB 146	31.82	360	362	80.6	PCB 153
PCB 161	31.91	360	362	80.6	PCB 153
$^{13}C_{12}$-PCB 118	**30.92**	**338**	**340**	**64**	
PCB 123	30.8	326	328	64.5	PCB 118/106
PCB 118/106	30.92	326	328	64.5	
$^{13}C_{12}$-PCB 180	**37.05**	**406**	**408**	**95**	
PCB 153	32.12	360	362	80.6	
PCB 132/168	32.25	360	362	80.6	PCB 153
PCB 141	32.77	360	362	80.6	PCB 138/163/164
PCB 137	33.12	360	362	80.6	PCB 138/163/164
PCB 130	33.26	360	362	80.6	PCB 138/163/164
PCB 138/163/164	33.52	360	362	80.6	
PCB 158/160	33.66	360	362	80.6	PCB 138/163/164
PCB 129	33.95	360	362	80.6	PCB 138/163/164
PCB 166	34.27	360	362	80.6	PCB 138/163/164
PCB 159	34.44	360	362	80.6	PCB 138/163/164
PCB 162	34.71	360	362	80.6	PCB 138/163/164
PCB 128	34.93	360	362	80.6	PCB 138/163/164
PCB 167	35.01	360	362	80.6	
PCB 156	36.17	360	362	80.6	
PCB 157	36.46	360	362	80.6	
PCB 169	37.91	360	362	80.6	
PCB 188	31.77	394	396	95.2	
PCB 184	32.12	394	396	95.2	PCB 188
PCB 179	32.85	394	396	95.2	PCB 188
PCB 176	33.22	394	396	95.2	PCB 188
PCB 186	33.67	394	396	95.2	PCB 187/182
PCB 178	34	394	396	95.2	PCB 187/182
PCB 175	34.29	394	396	95.2	PCB 187/182
PCB 187/182	34.42	394	396	95.2	
PCB 183	34.68	394	396	95.2	
PCB 185	35.23	394	396	95.2	PCB 183

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 174/181	35.68	394	396	95.2	PCB 183
PCB 177	35.89	394	396	95.2	PCB 183
PCB 171	36.12	394	396	95.2	PCB 180
PCB 173	36.42	394	396	95.2	PCB 180
PCB 172/192	36.75	394	396	95.2	PCB 180
PCB 180	37.06	394	396	95.2	
PCB 193	37.19	394	396	95.2	PCB 180
PCB 191	37.39	394	396	95.2	PCB 180
PCB 170/190	38.22	394	396	95.2	
PCB 189	38.98	394	396	95.2	
$^{13}C_{12}$-PCB 153- FS[1]	**32.12**	**372**	**376**	**34.1**	**PCB 153**
$^{13}C_{12}$-PCB 202	**36.07**	**440**	**442**	**112**	
PCB 202	36.08	428	430	112.4	
PCB 201	36.48	428	430	112.4	PCB 204
PCB 204	36.58	428	430	112.4	
PCB 197	36.86	428	430	112.4	PCB 204
PCB 200	37.61	428	430	112.4	PCB 204
PCB 198	38.35	428	430	112.4	PCB 196/203
PCB 199	38.45	428	430	112.4	PCB 196/203
PCB 196/203	38.6	428	430	112.4	
PCB 195	39.37	428	430	112.4	PCB 194
PCB 194	39.9	428	430	112.4	
PCB 205	40.03	428	430	112.4	
$^{13}C_{12}$-PCB 206	**40.82**	**474**	**476**	**128**	
PCB 208	39.35	462	464	128.2	
PCB 207	39.56	462	464	128.2	PCB 208
PCB 206	40.82	462	464	128.2	
$^{13}C_{12}$-PCB 209	**41.51**	**512**	**510**	**117**	
PCB 209	41.51	500	498	117	

[1] FS = Field Standard

Table 11. Typical Retention Times, Surrogates Used and RRF Determination for E2 Pesticides by GC/ECD

Compound Name	Typical Retention time (min)		Surrogate	RRF Determined From
	DB-5 column	DB-17 column		
delta-HCH	11.1	10.2	d_4-alpha-Endosulfan	delta-HCH
cis-Heptachlor epoxide	15.6	13.1	d_4-alpha-Endosulfan	cis-Heptachlor epoxide
alpha-Endosulfan	17.4	15.0	d_4-alpha-Endosulfan	alpha-Endosulfan
Dieldrin	18.9	17.0	d_4-beta-Endosulfan	Dieldrin
Endrin	20.2	19.5	d_4-beta-Endosulfan	Endrin
beta-Endosulfan	20.8	21.7	d_4-beta-Endosulfan	beta-Endosulfan
Endrin Aldehyde	22.1	24.4	d_4-beta-Endosulfan	Endrin Aldehyde
Endosulfan Sulfate	23.9	25.9	d_4-beta-Endosulfan	Endosulfan Sulfate
Endrin Ketone	27.5	32.5	d_4-beta-Endosulfan	Endrin Ketone
Methoxychlor	29.8	33.3	d_4-beta-Endosulfan	Methoxychlor
Labeled Surrogates			**Recovery Calculated Against**	
d_4-alpha-Endosulfan	17.3	14.9	$^{13}C_{12}$-PCB 153	
d_4-beta-Endosulfan	20.7	21.5	$^{13}C_{12}$-PCB 153	
Recovery Standard				
$^{13}C_{12}$-PCB 153	22.7	20.2		

Table 12. Surrogates Used and RRF Determination for Technical Toxaphene by GC/MS

Compound Name	Typical Retention Time (min)	Quantification Ion (mz)	Confirmation Ion (mz)	RRF Determined from
Toxaphene Peak T1	30.2	159	161	Toxaphene Peak T1
Toxaphene Peak T2	31.3	159	161	Toxaphene Peak T2
Toxaphene Peak T4	34.1	159	161	Toxaphene Peak T4
Toxaphene Peak T5	35.1	159	161	Toxaphene Peak T5
Toxaphene Peak T6	35.5	159	161	Toxaphene Peak T6
Surrogate Standard				
^{13}C-PCB 159	34.4	372	374	^{13}C-PCB 159
Recovery Standard				
^{13}C-PCB 138	33.5	372	374	

APPENDIX A

ECNI GC/LRMS ANALYSIS OF INDIVIDUAL TOXAPHENE CONGENERS/PARLARS

A ANALYSIS OF INDIVIDUAL TOXAPHENE CONGENERS/PARLARS BY ECNI GC/LRMS

The concentration of Toxaphene congeners/Parlars (identified with Parlar congener numbers where applicable) in the E1 extract may be determined by an additional analysis of the extract by electron capture negative ion (ECNI) GC/LRMS. $^{13}C_{12}$-PCB 180 is used as surrogate standard at nominal levels as shown in Table 3 of the main document and Table A-1 below. These levels may be adjusted depending on the composition of the used standard solutions or contract requirements. A recovery standard containing $^{13}C_{12}$-labeled PCB-138 is added prior to instrument analysis. Details of the typical concentration and composition of the authentic and calibration standards listed below are documented on Working Standard Data sheets.

A.1 INSTRUMENTAL ANALYSIS

Sensitivity

Sensitivity is demonstrated daily, by injection of a standard solution containing 25 pg/µL of each Toxaphene congener/Parlar just prior to injection of the calibration standard solution; the signal-to-noise ratio for each Toxaphene congener/Parlar compound in the sensitivity standard must be greater than or equal to 10:1, except for Parlar 62 where the signal-to-noise ratio must be greater than or equal to 5:1.

Calibration

Calibration RRFs are determined from analysis of a single concentration solution containing the individual Toxaphene congeners/Parlars, the surrogate and the recovery standards. The calibration standard is analyzed at least once every twelve hours, at the beginning and end of a bracket of samples. Mean RRFs from the beginning and end calibration runs are used to convert instrument responses to concentrations. The RSD for the beginning and end calibration runs must be less than 25%.

Table A1. Nominal Concentrations of Bracketing Calibration Standard Solution for Individual Toxaphene Congeners/Parlars (ng/mL)

Toxaphene Congener/Parlar Name	Calibration Standard Concentration (ng/mL)
2-exo,3-endo,6-exo,8,9,10-HexaCB	500
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	500
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	500
2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	1000
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	1000
2-exo,5-exo,6-endo,8,9,10,10-HeptaCB aka 2-endo,3-exo,6-exo,8,9,10,10-HeptaCB	500
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	500
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	500

2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	1000
2,2,5-endo,6-exo,8,8,9,10-OctaCB	500
2-exo,5,5,8,9,9,10,10-OctaCB	500
2,2,5-endo,6-exo,8,9,10,10-OctaCB	500
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	500
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	500
2,2,3-exo,5,5,9,9,10,10-NonaCB	500
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	1500
2,2,3-exo,5,5,8,9,10,10-NonaCB	500
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	500
2,2,5,5,8,9,9,10,10-NonaCB	500
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	500
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	500
Surrogate Standard	
$^{13}C_{12}$-PCB 180	400
Recovery Standard	
$^{13}C_{12}$-PCB 138	400

Linearity

Linearity of instrumental response across the working range is demonstrated separately from calibration by injection of multi-level solutions containing selected Toxaphene congeners/Parlars and the surrogate and recovery standards; this is performed upon initial instrument set up and repeated following major instrument maintenance. The RSD of the RRFs must be less than 25% across the working range. The range of linearity demonstrated is instrument specific and typically extends from approximately 25 pg/µL to 5000 pg/µL.

ECNI GC/LRMS Analysis

Gas chromatography/low resolution mass spectrometry (GC/LRMS) analysis of the E1 fraction for Toxaphene is done on a low-resolution mass spectrometer (LRMS) equipped with a capillary gas chromatograph using manufacturer's supplied software. A J&W DB-5 chromatography column (60 m, 0.25 mm i.d., 0.10 µm film thickness) is coupled directly to the MS source. The MS is operated at unit mass resolution in the electron capture negative ionization mode (ECNI) using multiple ion detection (MID) acquiring at least two characteristic ions for each target analyte and surrogate standard. A splitless/split injection sequence is used. The ions acquired are listed in Table A2.

Typical operating conditions for the GC/LRMS ECNI are presented below.

GC Temperature Program		**General GC Conditions**	
Temp (°C)	80	Injector Temp (°C)	170
Hold time (min)	1	Injector	Split/Splitless, 2 min
Rate 9 (°C min^{-1})	20	Carrier Gas	Helium, 28psi
Temp (°C)	200	Maximum Temp (°C)	325
Hold time (min)	0		
Rate (°C min^{-1})	1.5	**MS Conditions**	
Temp (°C)	230	Source Temp (°C)	142
Hold time (min)	0	Electron Energy (eV)	Variable
Rate (°C min^{-1})	10	Trap Current (µA)	–NA
Temp (°C)	300	Mass Resolution	unit
Hold time (min)	6.0	Detector Voltage (V)	Variable

The IA Chemist is referred to the Instrumental Method for further details of the instrumental analysis.

A.2 ANALYTE IDENTIFICATION

A peak is identified as a Toxaphene congener/Parlar only when the following criteria are met.

1. The peak must occur within 2 seconds of the retention time predicted from the calibration data and adjusted for the sample RT of the labeled retention time reference.
2. The ratio of confirming to quantification ions must be within 20% of the ratio found for that peak in the calibration standard.
3. The quantification and confirmation ions must co-maximize to within 2 seconds.

A.3 QUANTIFICATION PROCEDURES

Toxaphene congener/Parlar concentrations are determined by internal standard quantification against the labeled surrogate standard added prior to analysis; final results are automatically recovery corrected by this procedure.

Surrogate recoveries are calculated against the labeled recovery standard and are used as a general quality indicator.

The sample specific detection limit (SDL), for each analyte, is determined by converting the minimum detectable signal to a concentration using the calculations described in Section 9.0.

A.4 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, a procedural blank, a spiked reference sample and a duplicate sample where requested. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be substituted for duplicates on an individual contract basis. The batch is carried through the complete analytical process as a unit. For

sample data to be reportable, the batch QC data must meet the following established acceptance criteria.

- Concentration of target analytes in the procedural blanks must be less than 10 ng/sample.
- The percent recovery of the surrogate standards must be between 40–130%.
- The percent recovery of toxaphene congeners/Parlars in a spiked reference sample must be between 50–150%.
- The difference between target concentrations for MS/MSD samples (where performed) should be less than 40%.

Table A2. Analyte Ions Monitored, Surrogates Used, and RRF Determination for Toxaphene Congeners/Parlars
(No entry in the "RRF Used" field designates an RRF derived from that same compound)

IUPAC	Parlar Suite (Lab Code)	Quantified against labeled standard	Typical Retention Time	RT Win. (sec	mass1 (Qt)	mass2 (Cf)	%Cf/Qt ratio	Ion Ratio Limit (+/- %)
2-exo,3-endo,6-exo,8,9,10-HexaCB	Hex sed (2)	$^{13}C_{12}$-PCB-180	18.13	10	307	309	161	20
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	Hept sed (3)	$^{13}C_{12}$-PCB-180	19.13	10	341	343	192	20
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	(Peak 5)	$^{13}C_{12}$-PCB-180	21.31	10	341	343	192	20
2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	Parlar 32 (6)	$^{13}C_{12}$-PCB-180	21.79	10	341	343	192	20
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	(Peak 7)	$^{13}C_{12}$-PCB-180	22.03	10	341	343	192	20
2-endo,3-exo,6-exo,8,9,10,10-HeptaCB	(Peak 9)	$^{13}C_{12}$-PCB-180	23.54	10	341	343	192	20
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	Parlar 26 (4)	$^{13}C_{12}$-PCB-180	20.48	10	375	377	222	20
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	Parlar 39 (10)	$^{13}C_{12}$-PCB-180	23.76	10	375	377	222	20
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	Par 40/41 (11,12)	$^{13}C_{12}$-PCB-180	24.14	10	375	377	222	20
2,2,5-endo,6-exo,8,8,9,10-OctaCB	Parlar 42a (13)	$^{13}C_{12}$-PCB-180	24.39	10	375	377	222	20
2-exo,5,5,8,9,9,10,10-OctaCB	Parlar 44 (14)	$^{13}C_{12}$-PCB-180	24.75	10	375	377	222	20
2,2,5-endo,6-exo,8,9,10,10-OctaCB	(Peak 15)	$^{13}C_{12}$-PCB-180	26.08	10	375	377	222	20
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	(Peak 18)	$^{13}C_{12}$-PCB-180	26.99	10	375	377	222	20
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	Parlar 50 (16)	$^{13}C_{12}$-PCB-180	26.24	10	411	413	112	20
2,2,3-exo,5,5,9,9,10,10-NonaCB	(Peak 17)	$^{13}C_{12}$-PCB-180	26.84	10	411	413	112	20
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	Parlar 56 (19/20)	$^{13}C_{12}$-PCB-180	27.90	10	411	413	112	20
2,2,3-exo,5,5,8,9,10,10-NonaCB	Parlar 58 (21)	$^{13}C_{12}$-PCB-180	28.44	10	411	413	112	20
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	Parlar 59	$^{13}C_{12}$-PCB-180	28.72	10	411	413	112	20
2,2,5,5,8,9,9,10,10-NonaCB	Parlar 62 (22)	$^{13}C_{12}$-PCB-180	29.50	10	73	71	157	20
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	Parlar 63	$^{13}C_{12}$-PCB-180	29.93	10	411	413	112	20
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	Parlar 69	$^{13}C_{12}$-PCB-180	32.53	10	445	447	128	20
Labeled Compounds								
$^{13}C_{12}$-PCB-180		13C-PCB 138	28.28	20	406	408	91.9	20
$^{13}C_{12}$-PCB-138			24.89	20	372	370	78.7	20

APPENDIX B

HIGH RESOLUTION GC/MS ANALYSIS OF COPLANAR PCBs

B ANALYSIS OF COPLANAR PCBS BY HRGC/HRMS

The analysis of coplanar PCBs is carried out as a separate analysis of the E1 extract obtained by any of the extraction procedures described in Section 5.0. The analysis requires that an additional surrogate standard be added to the sample prior to extraction. The E1 extract is subject to additional chromatographic cleanup procedures prior to HRGC/HRMS analysis. The analysis is carried out by high-resolution gas chromatography with detection by high-resolution mass spectrometry (HRGC/HRMS).

The standard solutions required for the analysis of coplanar PCBs are the same as used for the analysis of all PCB congeners. The authentic standard solution is used to prepare the spiked matrix sample. The calibration standard is used for calibration of the GC/MS. Details of the concentration and composition of these standards are documented on Working Standard Data sheets.

B.1 EXTRACTION/CLEANUP PROCEDURES

Carry out the extraction procedure for the matrix as described in Section 5.0. Clean up the extract on Florisil as described in Section 6.1. Quantitatively split the E1 extract from the Florisil column in half and use ½ for the analysis of coplanar PCBs, cleaning up the split extract on carbon/Celite as described below. Alternatively, the entire extract can be subjected to the cleanup procedures below after the initial analysis for E1 fraction compounds.

B.2 PREPARATION OF CHROMATOGRAPHIC CLEANUP COLUMNS

Refer to MLA-010 section 3.5 for chromatographic cleanup column packing preparation instructions.

B.2.1 Carbon/Celite 545 Column for Coplanar PCB Analysis

A carbon/Celite column is prepared by packing 4.5% carbon/Celite mixture (0.22 g) into a 9” Pasteur pipette that is fitted with a filter paper disc, as described in SCH-005. The column is pre-eluted with toluene (15 mL) and hexane (15 mL), ensuring all the toluene has been eluted from the column.

B.2.2 Alumina Column for Coplanar PCB Analysis

Pack alumina (baked, 1% deactivated, 6 g) into a glass chromatographic column (1 cm x 25 cm with 100 mL reservoir) with hexane and cap with a 10 mm layer of anhydrous granular sodium sulfate.

B.3 CHROMATOGRAPHIC CLEANUP PROCEDURES

Carbon Column (Default Procedure)

Load the sample extract onto a pre-eluted carbon/Celite column (Section B.2.1) with hexane rinses (2 mL) and elute the column with cyclohexane:dichloromethane (1:1, 2 mL) followed by 10:1 ethyl acetate:toluene (2 mL). Discard each of these eluates. Invert the column and elute with toluene (60 mL). Collect this fraction and concentrate to just to dryness (by rotary evaporation followed by evaporation under a stream of nitrogen). Redissolve the residue in hexane (2 mL).

Alumina Column (Optional Procedure)

Additional cleanup on a prepared alumina column can be used to remove interferences associated with the matrix or the presence of interferences in the chromatogram. Requests for additional cleanup come from an IA Chemist or Manager.

Load the extract with hexane rinses onto an alumina column (6 g, 1% deactivated) (Section B.2.2). Elute the column with hexane (15 mL) and discard the eluate. Elute the column with dichloromethane:hexane (1:1, 35 mL) and retain the eluate. Concentrate to 1 mL by rotary evaporation and transfer the eluate to a centrifuge tube. Concentrate the extract under a stream of nitrogen and transfer to an autosampler vial with solvent rinses. Evaporate the solvent from the extract to just dryness under a stream of nitrogen and add aliquot of the recovery standard named in Table B1. Cap the autosampler vial. The extract is ready for GC/MS analysis.

B.4 INSTRUMENTAL ANALYSIS

Sensitivity

Signal-noise ratios must be greater than or equal to 3:1 for 0.05 pg of 3,3',4,4',5,5'-HxCB (PCB 169) injected.

GC Resolution

All peaks must be inspected for acceptable chromatography, but specifically, a maximum peak width of 15 sec is allowable for PCB 169.

Calibration

Initial calibration is performed using a series of five solutions that encompass the working concentration range of the instrument. The initial calibration solutions contain a suite of labeled surrogates, recovery standards and target compounds. The relative standard deviation of the RRFs must be $\leq$20% over the calibration range. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution The RRFs from the mid-point calibration standard must agree to ±20% over a 12-hour period.

Analysis

The E1 fraction is analyzed for coplanar PCBs using a high-resolution mass spectrometer (HRMS) equipped with a gas chromatograph (GC), and an autosampler, and operated using manufacturer's software for targeting and quantification. Chromatographic separation is achieved with a DB-1 capillary column (30 m, 0.25 mm id and 0.25 µm film thickness), refer to MLA-010 "Analytical Method for the Determination of 209 PCB Congeners by EPA Method 1668A, EPA Method 1668C or EPA Method CBC01.2", Appendix A, for further details. A splitless/split injection sequence is used.

The IA Chemist is referred to Instrument Method MIN-007 (COP) for further details of the instrumental analysis.

B.5 ANALYTE IDENTIFICATION

A chromatographic peak is identified as a target compound if the following criteria are met for the quantification and confirmation ions (where confirmation ions are available):

1. Peak responses must be at least three times the background noise level.
2. The retention time must be within five seconds of that predicted from the calibration run and the sample retention time reference.
3. Peak maxima for quantification and confirmation ions must coincide within two seconds.
4. The relative ion abundance ratios must be within 20% of the theoretical.

B.6 QUANTIFICATION PROCEDURES

Target concentrations are determined using the isotope dilution method of quantification, as described in Section 9.0.

B.7 QUALITY ACCEPTANCE CRITERIA

The quality acceptance criteria are presented in Table B1.

Table B1: QC Acceptance Criteria for Analysis of Coplanar PCBs by GC/HRMS

Analyte:	ACCEPTABLE DETECTION LIMITS					Procedural Blank Level pg	Acceptable Matrix Spike % Recovery	
	Solid pg/g	Aqueous pg/L	Tissue pg/g	Pulp pg/g	XAD Column pg/col		GC/LRMS	GC/HRMS
3,3',4,4'-TCB (#77)	0.2	2	0.2	0.2	2	<3	70-130	70-130
3,3'4,4',5-PCB (#126)	0.2	2	0.2	0.2	2	<2	70-130	70-130
3,3'4,4'5,5'-HCB (#169)	0.2	2	0.2	0.2	2	<3	70-130	70-130
Typical Sample Size:	10 g	1 L	10 g	10 g	1 col			
Final Vol, µL	20	20	20	20	20			

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
^{13}C-3,3',4,4'-TCB (#77)	40-130
^{13}C-3,3',4,4',5-PCB (#126)	40-130
^{13}C-3,3',4,4',5,5'-HCB (#169)	40-130

GC Parameter	Specification
ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
PROCEDURAL BLANK:	All analytes must be <3 pg or ≤1% of sample's analyte values.
INSTRUMENT SENSITIVITY:	S/N ratios should be ≥3:1 for 0.05 pg of 3,3',4,4',5,5'-HxCB injected.
INSTRUMENT LINEARITY:	Linearity determined by at least a 5-point calibration over the range of 10 pg/µL to 1000 pg/µL if all coplanar PCBs with an RSD of the RRFs are within ±20%.
RRF: Bracketing Cal	RRFs from calibration standards must agree to ±20% over a 12-hour period.
RRF: Cal Ver	RRFs from opening/closing calibration standards must be within ±25% of the mean RRFs from the initial calibration.
CHROMATOGRAM QUALITY: **Max Peak Width:** **Resolution:**	All chromatograms visually inspected but the 3,3',4,4',5,-PCB peak is examined specifically. 15 sec for PCB 169 Visually acceptable
ANALYTE/SURROGATE RATIOS:	Response must be within the calibrated range of the instrument. IA Chemists may use data from more than one chromatogram to get the responses in the calibrated range.

APPENDIX C

MASS SPECTROMETER DAILY TUNING CHECK USING PFTBA

C MASS SPECTROMETER DAILY TUNING CHECK USING PFTBA

Using the Chem Station Software follow the following steps to check the mass spectrometer mass calibration, mass resolution and peak relative response:

1) Click the “View” option in Chem Station and select “Manual Tune”. Then select “AdjParam” and then “Edit MS Params”. Select “Prof” for profile scan and let the tune gas stabilize for 1 minute. Then select “File” and “Print”. Press “Stop”. All evaluation will be done from the printout.

2) A. Check the instrument “Mass Resolution” as per SIN-029 Rev. 03.

   B. Check “Mass Calibration” by measuring the amount of peak drift from the expected mass for M/Z 69, 219 and 502. If the peak apex has shifted more than approx 0.4 (or approx 2 mm on the calibration printout) then the instrument will need to be re-calibrated.

   C. Check “Relative Peak Response” of m/z 219 and 502 against m/z 69 by dividing the abundance of the desired peak, listed on the calibration check printout, by the abundance of the m/z 69 peak. If the relative peak intensities are outside a 50 - 150 % range compared to the reference tune, corrective action is required.

Corrective actions

3) A. If the mass resolution fails against SIN-029 Rev. 03 then return to step one and print out another calibration test. If the resolution fails a second time the instrument will need to be re-tuned.

   B. If the mass calibration is outside the specification, then in the “Manual Tune” page select “Calibrate” and then “Mass Axis”. Then return to step 1 above and re-check all specifications.

   C. If the relative intensities are outside the given specification then re-attempt step 1 and re-evaluate the printout. If the relative mass intensities are still out of spec, a full tune may be required.

APPENDIX D

Summary of Key Attributes of Methods MLA-007, EPA 8270C/D, EPA 625, EPA 8081A/B and EPA 608

D SUMMARY OF KEY ATTRIBUTES OF METHODS MLA-007, EPA 8270C/D, EPA 8081A/D, EPA 625 AND EPA 608

Analysis by GC/LRMS, Key Attributes of SGS AXYS MLA-007, EPA 8270C/D and EPA 625				
	MLA-007	**EPA 8270C**	**EPA 8270D**	**EPA 625**
MS acquisition mode	SIM	Full Scan or optional SIM [1]	Full Scan or optional SIM [1]	Full Scan or alternate SIM [1]
Qualitative Identification Criteria	Retention time & ratio of 2 ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions	Retention time & ratio of 3 [2] ions
MS Ion Ratio Criteria	Within 20% of theoretical	Within 30% of reference spectrum	Within 30% of reference spectrum	Within 20% of reference spectrum
MS Tuning Type and Check Frequency	PTFBA, daily	DFTTP [1], 12 hrs	DFTTP [1], 12 hrs	DFTTP [1], 12 hrs
Quantification References	Isotopically Labeled Standards added prior to extraction	Internal Standards added just before instrumental analysis	Internal Standards added just before instrumental analysis	Internal Standards added just before instrumental analysis
Recovery correction of results	YES	NO	NO	NO
Calibration, minimum # levels	CCV Procedure: 5 levels OPTIONAL Single Point BRACKETING: 1 level	5	5	3
Initial Calibration Limit (% rsd)	20% (DDT is 25%)	15%	20%	35%
Calibration Verification Frequency	Initially and every 12 hrs	Initially and every 12 hrs	Initially and every 12 hrs	Daily
Calibration Verification Relative Response Limit (% diff.)	CCV Procedure: < 20% of I-CAL OPTIONAL BRACKETING Procedure based on runs before and after samples: <20%	< 20% of I-CAL	< 20% of I-CAL	< 20% of I-CAL
Calibration Verification IS area (% of I-CAL midpoint)	50–200%	50–200 %	50–200%	n.a.
Calibration verification IS RT (diff. from I-CAL midpoint)	n.a.	30 sec.	30 sec.	n.a.
Extraction	DCM, L/L (aqueous), DCM, sox. (solids)	Options specified externally	Options specified externally	DCM, L/L (aqueous), pH >11 or pH other [3]

Notes:

1 SIM acquisition is permitted in EPA8270 and under Federal Register Volume 77 Issue 97 (May 18, 2012) Part 136.6
2 Based on availability, use of fewer ions is permitted where necessary.
3 Modifications are permitted under Federal Register Volume 77 Issue 97 (May 18, 2012) Part 136.6

Analysis by GC/ECD, Key Attributes of SGS AXYS MLA-007, EPA 8081A/B and EPA 608

	MLA-007	EPA 8081A	EPA 8081B	EPA 608
Sample Preservation and Storage	0–4 ºC, dark No preservation required	≤6 ºC $Na_2S_2O_3$ if chlorinated	≤6 ºC $Na_2S_2O_3$ if chlorinated	4 ºC Adjust pH 5–9 if not extracted by 72 hrs
Sample/Extract Hold Time	7 days / not defined	7 / 40 days	7 / 40 days	7 / 40 days
GC Columns	Capillary	Capillary	Capillary	Packed columns, OPTIONAL capillary columns permitted
Detection	ECD	ECD	ECD	ECD
Qualitative Identification Criteria	Relative retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute retention times. Confirmation by dual column GC/ECD or by GC/MS	Absolute retention times. Confirmation by dual column GC/ECD or by GC/MS
Quantification Technique and References	Combination of isotope dilution and internal standard added prior to extraction	External standard or optional internal standard prior to instrument	External standard or optional internal standard prior to instrument	External standard or optional internal standard prior to instrument
Recovery correction of results	YES	NO	NO	NO
Calibration, minimum # levels	CCV Procedure: 5 levels OPTIONAL Single Point BRACKETING: 1 level	5	5	3
Initial Calibration Limit (% rsd)	20%	20%	20%	10% otherwise use regression
Calibration Verification Frequency	Initially, every 12 hrs, every 20 samples before and after samples	Initially, every 12 hrs, every 20 samples before and after samples	Initially, every 12 hrs, every 20 samples, before and after samples	Daily
Calibration Verification Relative Response Limit (% diff.)	< 20% of I-CAL (d-HCH is 25%)	< 15% of I-CAL	< 20% of I-CAL	< 15% of I-CAL
Extraction	DCM, L/L stirring DCM, Soxhlet	Options specified externally include DCM L/L sep. funnel and DCM Soxhlet	Options specified externally include DCM L/L sep. funnel and DCM Soxhlet	DCM L/L sep. funnel
Clean-up	Florisil	Not specified	Not specified	Florisil sulfur removal
Spiked Sample Requirement	Optional by contract	Not specified	Not specified	10% at 1–5 times sample conc.
QC spike frequency	1 per batch or 5%	Not specified	Not specified	When spiked sample test fails
IPR acceptance limit range	N/A	Not specified	Not specified	Varying
OPR acceptance limit range	60–130%, or narrower	Not specified	Not specified	EPA 608 limits are wider than MLA-007

AXYS Analytical Services Ltd.
Standard Operating Procedure

Title: Procedures for Homogenization of Solids and Tissues
Area: Laboratory Procedures

SOP #: SLA-013
Rev. No.: 09
Date: 28-Jul-2016

Purpose:

To homogenize samples thoroughly to enable a representative sub-sample to be taken for analysis.

Scope:

Accurate analysis results require that the sub-sample analyzed is representative of the larger sample being studied. Therefore, the sample must be thoroughly homogenized before subsampling. Chemists working in the Sample Preparation group are responsible for the homogenization of the following matrices.

- Animal tissue
- Plant tissue
- Pulps
- Solids (soils, sediments, biosolids, ashes, sludges, etc.)

This SOP describes the homogenizing procedures for the above matrices. Prior to homogenizing, samples may have to be dissected or composited. These procedures are described in SLA-011 “Compositing Samples” and SLA-012 “Dissection of Samples”, respectively.

Aqueous samples are homogenized according to procedures described in SLA-084 “Preparation of Aqueous Samples for Extraction”. These procedures are carried out by a Chemist in the Extraction Lab, just prior to analysis.

Equipment and Reagents:

1. Refer to Table 1 for the equipment used in homogenization procedures. Refer to Table 2 to determine approved equipment for homogenization procedures. Refer to Table 4 for guidance in selecting the appropriate homogenizing equipment.
2. Wear only polyethylene gloves that have been approved for use in the lab for homogenization procedures. Minimize the contact between a glove and sample and ensure that solvents do not come in contact with the gloves used for homogenization.
3. Clean all equipment before starting homogenization and between each use according to SOP SLA-037 “Cleaning of Sample Preparation Equipment used for Preparing Metals and Organic Samples”.
4. Solvents used must be proofed and approved for use. The list with approved solvents is found in LIMS under Reports → Reports to preview → Report → APPRVD SOLVENTS.
5. Only the dull side of aluminium foil may come in contact with a sample as the shiny side

may have a chemical coating that may be a source of contamination.

6. Do not allow samples to remain at room temperature for extended periods. Homogenize and then return to cold storage immediately.
7. Any new jars used must be approved for use as listed in LIMS (Reports → Reports to preview → Report → APPRVD BOTTLES_JARS). Containers may be restricted for use with only certain analyses, refer to the LIMS to determine which analyses have been requested for the sample.

Table 1: Homogenizing Equipment

Inventory No.	*LIMS Inventory No.*	*Item*	*Manufacturer*	*Model No. (Serial No.)*
G01	2050	Grinder	OMAS ½ hp	TS8 (8449)
G03	2052	Grinder	Butcher Boy 3 hp	TCA32 (S2457)
G04	2066	Grinder	Butcher Boy 0.75 hp	TCA12 (S368)
G05	2220	Grinder	OMAS ½ hp	TS8 (A1462)
	2154	Blender	Virtis	302968 (214235)
	3030	Blender	Virtis	302968 (212954)
	3493	Blender	Omni International	17105 (MX11302)
	Glass jars Spatulas Disposable spoons Scoopulas Scissors Disposable scissors Forceps Disposable forceps Sieve Knives Stainless steel bowls Scalpels Shears Centrifuge (Thermo Scientific Sorvall Legend RT+)			

Table 2: Use of Homogenizing Equipment

LIMS Inventory No.	*Item*	*Manufacturer*	*Model No. (Serial No.)*	*Approved Analysis*
2050	Grinder	OMAS ½ hp	TS8 (8449)	Routine Organics, PFCs and Metals
2052	Grinder	Butcher Boy 3 hp	TCA32 (S2457)	Routine Organics, PFCs and Metals
2066	Grinder	Butcher Boy 0.75 hp	TCA12 (S368)	Routine Organics, PFCs and Metals
2220	Grinder	OMAS ½ hp	TS8 (A1462)	Routine Organics, PFCs and Metals
2154	Blender	Virtis	302968 (214235)	Routine Organics, PFCs and Metals
3030	Blender	Virtis	302968 (212954)	Routine Organics, PFCs and Metals
3493	Blender	Omni International	17105 (MX11302)	Routine Organics, PFCs* and Metals

* Stainless steel bearings are required when used for PFC analysis

AXYS Analytical Services Ltd.
Standard Operating Procedure

Procedures:

1. INITIAL INSTRUCTIONS

1. Do not proceed with homogenization until the Project Manager has released samples in LIMS. Always print and review the Project Notes before starting homogenization. Resolve any ambiguity or questions before homogenizing.
2. Request the samples for homogenization from the Sample Receiving Chemist, according to procedures described in SLA-004 “Sample Control Procedures”. A request for the samples to be homogenized should be made prior to homogenization by the Scheduler, the Sample Preparation Lead Chemist, or designee.
3. Clean equipment required as outlined in SLA-037 “Cleaning of Sample Preparation Equipment used for Preparing Metals and Organic Samples”.
4. Agricultural Hazard or Human Biohazard sample transfers must be performed in a HEPA-filter equipped fume hood using the following SOP instructions:

   Handling of Human Biohazardous Samples (SLA-082)

   Agricultural Hazard Protocols for Soils (SLA-079)
5. Check the sample jars for any cracks. If any samples have broken jars, transfer the sample to a new container as per SLA-077 “Sample Transfer Procedures” and note on the Sample Preparation Record that the jars were broken. (A Sample Transfer Form (FSA-030) is not required when transferring a sample that is being homogenized at the same time.) Report compromised or lost samples to the Project Manager.
6. Any concerns regarding sample integrity must immediately be brought to the attention of a supervisor and Project Manager.
7. Concisely describe each sample (i.e., type or species of plant tissue, colour, texture, condition, etc.) on the Sample Preparation Record.
8. After homogenizing each sample, clean all equipment as outlined in SLA-037 “Cleaning of Sample Preparation Equipment used for Preparing Metals and Organic Samples”.
9. Request the samples be returned to the storage location according to procedures in SLA-004 “Sample Control Procedures”.
10. Write the empty jar weight on the AXYS label whenever possible.

2. RECORD KEEPING

A Sample Preparation Record must be completed for each sample at the time it is homogenized. Table 3 lists Sample Preparation Records available for use. Custom worksheets may be created for use with specific contracts and should be used where available. All fields on a Sample Preparation Record MUST be filled in.

Table 3: Available Sample Preparation Records

Worksheet ID	Title
FWO-104	Tissue Preparation Record
FWO-250	Tissue Composite Preparation Record
FWO-219	Solid Preparation Record
FWO-235	Lab Services Subsampling Record
FWO-306	Sample Weight and Moisture

1. Cross-reference the AXYS ID label and the Client label against the LIMS Chain of Custody. If there are any labelling discrepancies that could call into question sample identification, do not proceed until these are resolved and/or corrected.
2. Record the Client ID directly from the sample label. Use judgement to determine if additional label information would be useful to record.

3. HOMOGENIZING ANIMAL TISSUE

Equipment used to homogenize animal tissue includes scissors, a blender and commercial meat grinder(s). The type of equipment used depends on the quantity and texture of the sample to be homogenized.

1. Allow the samples to thaw as described in SLA-014 “Thawing Solid and Tissue Samples”.
2. If the sample requires dissection or shucking (molluscs), do this while the sample is still partially frozen. The dissection procedure is described in SLA-012 “Dissection of Samples”.
3. If shucking is required, use shucking tools and wear gloves. Shuck the samples on a foil-covered surface.
4. If samples are small and a significant proportion of the sample would be lost when using a powered homogenizing instrument, they may be homogenized by cutting with scissors. Cut the sample inside the jar using only scissors and stir vigorously and thoroughly with a spatula. If necessary, forceps may be used to hold the sample while cutting.

Procedure: Blender

1. Before using a blender, the chemist must use their judgement as to which blender is the appropriate homogenization tool. Sufficient sample size is required to avoid losses onto the aluminum foil by spray from the blender. There also needs to be enough liquid present for the blender to be appropriate. Too little liquid and the blender cannot properly homogenize a sample.

2. Select a homogenization jar. The homogenization jar should not be smaller than 60 mL nor larger than 500 mL. The jar should be no more than 2/3 full during homogenization. If it is not possible to fit the entire sample into one jar, two jars may be used. In some cases the original sample jar will be suitable for use with the blender.

3. If necessary, depending on which blender is chosen, chop the sample into small pieces using appropriate stainless steel tools. Cut the tissue on a foil-covered surface or directly into the jar that will be used to homogenize the sample. If skin is present, score it with a scalpel prior to chopping.

4. Transfer the sample to the homogenization jar using solvent rinsed utensils. Allow the sample to thaw completely.

5. Place aluminum foil around the jar and position the blade inside the jar in such a way that the blade is not touching either the bottom or the sides of the jar. Fold foil around the stem of the blender blade and cover the jar to avoid sample loss and contamination. On speed setting 1, turn on the blender for about 15 seconds. Hold the jar near the threaded portion and keep one hand on the on/off switch as a safety precaution.

6. Remove aluminum foil and lower the jar from the blade. With a spatula scrape the sample off the blade back into the jar. Scrape sample from the sides of the jar and mix. If the sample will be analyzed for metals, minimize the amount of metal scraping. Inspect for homogeneity.
   Repeat Steps 4 and 5 until the sample is completely homogenized.

7. If the sample has been moved to new jar(s), transfer the original labelling from the original jar to the one used for homogenizing. If necessary print a set of LIMS labels using module: Reports → Login → Labels → Sample Prep Labels. NEVER HAVE UNLABELLED SAMPLES IN THE LAB. If the sample required homogenization in more than one jar combine the sample into a jar large enough to hold the entire sample. Mix thoroughly and label this jar. If necessary, multiple jars may be used (labelled as 1 of X, 2 or X, as appropriate). Ensure that all client information is photocopied and placed on all jars.

8. Write your initials and the date on the sample label along with the word "homogenized" on all jars.

Procedure: (Commercial Meat Grinder)

The chemist homogenizing the sample will have to use their judgement when deciding which grinder is appropriate for use.

1. Thaw samples just enough to allow them to be cut with a knife. If a sample requires dissection, follow the procedure as described in SLA-012, "Dissection of Samples".

2. Use a knife to chop the frozen sample into pieces small enough to feed through the meat grinder. Place the pieces in a stainless steel mixing bowl. Wear polyethylene gloves while handling the sample. A chain-mail glove should be used while chopping samples and is worn between two polyethylene gloves.

3. Transfer the sample to the feed chute of the grinder using a polyethylene-gloved hand. Turn on the grinder and push the sample through the grinder with the stainless steel plunger. Do not use the plunger to force tissue through the grinder faster as it may push tissue back into the motor housing. Collect the ground sample in the mixing bowl. **Do not put a spatula or other object into the blender while it is running.**

4. After the entire sample has gone through the grinder, turn off the motor. Mix the ground tissue using a spoon. Transfer the ground sample back into the grinder and repeat grinding two more times.

5. If the sample is small (100 g) carefully disassemble the grinder and scrape out any remaining tissue between each grind. USE EXTREME CAUTION WHEN SCRAPING OUT GRINDER IF TRACE METALS ANALYSIS IS BEING DONE.

6. If the sample has been moved to new jar(s), transfer the original labelling from the original jar to the one used for homogenizing. If necessary print a set of LIMS labels using module: Reports → Login → Labels → Sample Prep Labels. NEVER HAVE UNLABELLED SAMPLES IN THE LAB. If necessary, multiple jars may be used (labelled as 1 of X, 2 or X, as appropriate). Ensure that all client information is photocopied and placed on all jars.

7. Write your initials and the date on the sample label along with the word "homogenized" on all jars.

4. HOMOGENIZING PLANT TISSUE

1. It is not necessary for plant tissues to thaw before homogenizing.

2. If the sample is grass, leaves or thin woody stalks, use scissors for homogenizing. Wear gloves while handling the sample. Cut the sample up while it is still frozen into one-centimetre long pieces. Woody stems should also be cut in half with the grain of the wood. Collect the cut sample on a piece of aluminum foil. Homogenize the cut material using scissors.

   Note: If large pieces of wood require homogenizing, use a drill with a large-gauge drill bit. Securely clamp the wood and drill random holes in the wood making sure to choose drill sites that best represent the composition of the submitted wood sample. Collect the sawdust on aluminum foil.

3. Transfer the sample to a jar.

4. If the sample has been moved to new jar(s), transfer the original labelling from the original jar to the one used for homogenizing. If necessary print a set of LIMS labels using module: Reports → Login → Labels → Sample Prep Labels. NEVER HAVE UNLABELLED SAMPLES IN THE LAB. If the sample required homogenization in more than one jar combine the sample into a jar large enough to hold the entire sample. Mix thoroughly and label this jar. If necessary, multiple jars may be used (labelled as 1 of X, 2 or X, as appropriate). Ensure that all client information is photocopied and placed on all jars.

5. Write your initials and the date on the LIMS label along with the word "homogenized" on all jars.

5. HOMOGENIZING PULPS

Dry Pulp

1. Randomly cut strips approximately 1 cm wide by 5 cm long from the sheet pulp and paper samples using scissors, shears or a scalpel. Cut these strips into 1 cm squares. Wear gloves while handling the sample.

2. Re-wrap the original sample in aluminum foil. Affix a label indicating the date, the Chemist's initials and the word "subsampled". Retain the original label on the foil.

3. Transfer the homogenized sample to a jar. Print a set of LIMS labels using module: Reports → Login → Labels → Sample Prep Labels. NEVER HAVE UNLABELLED SAMPLES IN THE LAB. Ensure that all client information is photocopied and placed on all jars.

4. Write your initials and the date on the AXYS sample label along with the word "subsampled" on the jar.

6. HOMOGENIZING SOLIDS

Solids include soils, sediments, biosolids, ashes and sludges.

When homogenizing solids, the analyst needs to use their judgement when choosing the appropriate tools required for the sample. In certain situations the samples may require homogenization tools that are not routinely used.

The chemist homogenizing will use judgement to decide what is representative of the sample and whether what the client wants to be removed can be, or if it will need to be homogenized as part of the sample.

1. If a client has requested the removal of standing water, use the following procedures as appropriate:

a) Settling/Decantation:

Without disturbing the fine particulate matter, inspect the free water for evidence of suspended or floating particles. If these are observed, consult the Project Manager for permission to proceed.

The decantation procedure must not result in loss of fine particulate matter. Decant carefully, removing only as much clear water as can be separated without disturbing the final material. For small volumes of water (<50 mL) a baked glass pipette is used with a bulb. For larger volumes (>50 mL), the pipette may be connected to a pump to remove the water more efficiently. (See the Lab Supervisor for details).

Note the weight of the water decanted on the Sample Preparation Record.

b) Centrifugation/Decantation:

Samples in which the fine particles cannot be removed from the free water by settling are centrifuged prior to decantation as follows:

Decant standing water into a solvent-rinsed, 50 mL centrifuge tube. Load into the Thermo Scientific Sorvall Legend RT+ centrifuge and spin at 3000 RPM for 10 min. Decant the standing water from the particulate and combine the particulate in the centrifuge tube with the original sediment sample.
Note the weight of the water decanted on the Sample Preparation Record.

c) The client may request that the standing water be decanted and retained. If the water is to be retained decant the water into a jar or bottle and label with the pre-logged in AXYS ID and the client information. The retained water **must not** have the same AXYS ID as the solid it has been decanted from. If the standing water is not to be retained discard the standing water as per SAD-014 “Sample Disposal”.

2. Due to the difficulty and expense of cleaning and proofing sieving equipment, sieving of solids is not recommended but maybe performed at the request of the client. If sieving is requested pass the sample through the appropriate sieve, into a clean bowl. Break up clumps with a disposable spoon. Ensure that the sample is not within 1” of the sides of the sieve in order to prevent sample contamination. Refer to the Project Notes for instructions regarding material retained on the sieve. **If samples appear oily or greasy or smell of hydrocarbons consult the Project Manager before sieving.** Sieves must be thoroughly cleaned and proofed before being used or reused.

3. If vegetation is present and is required to be retained, use disposable scissors to cut the vegetation into pieces no larger than 1 cm and return it to the sample.

4. If required by the client, remove rocks of greater than 0.4 cm in diameter and foreign particles using a disposable spoon or forceps.

5. Where possible, homogenize the sample by manual mixing with a disposable spoon, within the original sample container. The container should be no more than ¾ full to ensure room for mixing.

6. If it is impractical to mix the sample within its container: Transfer the sample to a new larger container while inspecting it for rocks, vegetation, invertebrates and foreign objects. Mix the sample thoroughly with a disposable spoon. Be sure to stir from the bottom to the top and in a circular motion along the sides of the jar. The homogenized sample should be even in colour with no layers present. Make a reasonable effort to break particles to less than 1 mm by pressing against the side of the container.

7. Grinding the sample in a mortar and pestle is not recommended due to the difficulty and

expense of cleaning and proofing equipment. A client may request that grinding be performed to ensure that particle size is less than an estimated 1 mm diameter. This must be employed where porous and pulverizable materials are observed at greater than 5% of the total sample mass as estimated by eye. Consult the Project Manager for instructions and authorization. After grinding, stir the sample using a solvent-rinsed spoon until it is completely homogeneous. Return the sample to its original jar. **After completion of this step the mortar and pestle must be isolated and identified as potentially high level contaminated – it will require special cleaning and proofing prior to being returned into service.**

8. After the sample is homogenized obtain a moisture evaluation if requested as per SLA-015 “Moisture Determination”.

9. If the sample is a wet solid it should be divided into multiple jars after homogenization to reduce breakage due to freezing and thawing. The sediment should be divided as follows:

    a. 1 X 120 mL or appropriate jar size for sample for primary use
    b. 1 X 120 mL or appropriate jar size for sample for secondary use
    c. 250 mL or 500 mL jars as required for back up

10. If the sample has been moved to new jar(s) transfer the labelling from the original jar to the one used for homogenization. If necessary, print a set of LIMS labels using module: Reports → Login → Labels → Sample Prep Labels. NEVER HAVE UNLABELLED SAMPLES IN THE LAB. If the sample required homogenization in more than one jar combine the sample into a jar large enough to hold the entire sample. Mix thoroughly and label this jar. If necessary, multiple jars may be used (labelled as 1 of X, 2 or X, as appropriate). Ensure that all client information is photocopied and placed on all jars.

11. Write your initials and the date on the sample label along with the word "homogenized" on all jars.

Table 4: Guidance for Homogenization

SAMPLE TYPE		*Prehomogenizing Preparation*	*Homogenizing\ Equipment Used*
Fish (whole or fillets)	under 100 g	cut into small pieces, place in jar, thaw thoroughly. Score skin with scalpel.	Scissors
Fish (whole or fillets)	over 30 g to 300 g	cut into small pieces, sample remains partially frozen, score skin with scalpel	Blender (Omni Mixer)
Fish (whole or fillets)	over 300 g	cut into small pieces, sample remains partially frozen	Small grinder (G01, G05 G04 or G03
Bivalves	under 250 g over 250 g	thaw thoroughly, shuck, place in jar, shuck, thaw	Blender (Omni Mixer)
Crab muscle		partially thaw, remove muscle tissue from shell, place in jar	Scissors or blender (Omni Mixer)
Crab hepatopancreas		thaw slightly, remove hepatopancreas from body, place in jar	Blender (Omni Mixer)
Plants (soft or woody stem)	under 75 g	cut leaves off stems, cut stems into small pieces, place in jar	Scissors
Plants (soft or woody stem)	over 75 g	cut leaves off stems, cut stems into small pieces	Scissors
Plants (grass any amount)		cut into small pieces, place in jar	Scissors
Mice and voles	1 to 2 whole animal(s)	while partially frozen, cut into very small pieces, place in jar, thaw	Scissors
Mice and voles	over 2 whole animals	while partially frozen, cut into very small pieces, remain partially frozen	Scissors
Other mammals	whole	use same guidelines as fish	Same rules as with fish
Liver	under 100 g	place in jar, thaw thoroughly	Scissors
Liver	over 100 g under 500 g	partially thaw, chop into small pieces, place in jar, thaw thoroughly	Blender (Omni Mixer)
Adipose	under 75 g	partially thaw, chop into small pieces, place in jar, thaw thoroughly	Scissors
Adipose	over 75 g	partially thaw, chop into small pieces	Knife, scissors, small grinder (G01 or G05), Blender (Omni Mixer)
Insects	under 3 g	partially thaw	Scissors
Insects	over 3 g	partially thaw	Scissors
Egg		thaw slightly, remove shell, place in jar, thaw thoroughly	Blender (Omni Mixer), scissors, spatula
Soil, sediments and sludges	wet or dry	thaw thoroughly	Spoon/forceps/scissors. 4 mm sieve if requested
Paper and pulp	dry sheets	none	Scissors
Ash	dry	none	Spoon/forceps/scissors.

AXYS Analytical Services Ltd.
Standard Operating Procedure

References:

FSA-030	Sample Transfer Form
FWO-104	Tissue Sample Preparation Record
FWO-219	Solid Sample Preparation Record
FWO-235	Sample Preparation Subsampling Record
FWO-250	Composite Sample Preparation Record
FWO-306	Sample Weight and Moisture
SLA-004	Sample Control Procedures
SLA-011	Compositing Samples
SLA-012	Dissection of Samples
SLA-014	Thawing Solid, Tissue and Serum Samples
SLA-015	Moisture Determination
SLA-037	Cleaning of Sample Preparation Equipment used for Preparing Metals and Organic Samples
SLA-077	Sample Transfer Procedures
SLA-079	Agricultural Hazard Protocols for Soils
SLA-082	Handling of Human Biohazardous Samples
SLA-084	Preparation of Aqueous Samples for Extraction
SAD-014	Sample Disposal

Approval:

Approved 04-Jul-2016	John Cosgrove, President and Senior Technical Director
	Shea Hewage, Director of Operations
	Dale Hoover, QA Manager

AXYS Analytical Services Ltd.
Standard Operating Procedure

Purpose:

To determine the lipid content (expressed as a percent of sample weight) of a tissue sample.

Scope:

When required by contract, a lipid determination on a tissue sample is performed on a tissue sample extract, prior to any chromatographic clean-up procedures. A Chemist working in the Extraction Lab performs the analysis. The percent lipid determination for a tissue sample is carried out in duplicate on the same extract. The Project Manager selects the analysis in which the lipid determination is carried out if more than one analysis is being performed. The data are entered into a spreadsheet that calculates the results. The data are saved in the LIMS (Laboratory Information Management System).

Equipment and Materials:

Top loading balance – accurate to 2 decimal places
Analytical balance – accurate to 4 decimal places
Drying oven (Fisher Isotemp Oven 200 Series, Lab-Line Heet-Cab No.3515) maintained at 105°C.
Solvents - high purity, distilled in glass, either HPLC grade or pesticide residue grade. Each lot number of solvent must be checked for impurities by performing a solvent proof prior to use
Rotary evaporator - equipped with a water bath
Pasteur pipettes
Glass Petri dishes

Glassware Cleaning

All glassware must be organically clean. Glassware must be washed and baked using standard operating procedures (SLA-001). If baked glassware is not available, glassware must be washed and solvent rinsed following standard operating procedures (SLA-018).

Procedure:

Record all weights on the sample worksheet.

1. Prior to beginning the sample extraction, weigh each *labelled* round-bottom flask (including anti-bumping granules) that will be used to collect the sample extract on a top loading balance. Record each weight to two decimal places.
2. If sodium sulphate is present, carefully transfer with rinses to another weighed, *labelled* round-bottom flask ensuring that no sodium sulphate or anti-bumping granules are transferred. Use hexane rinses for extracts in hexane or in 1:1 hexane:dichloromethane. Use dichloromethane for extracts in 100% dichloromethane.
3. When the extraction procedure is complete, concentrate the extract to 10-20 mL using rotary evaporation.
4. Label and accurately weigh two prebaked glass Petri dishes on the analytical balance. Record the weight of each dish to four decimal places.
5. Allow the flask to achieve room temperature. Ensure the outside of the flask is dry. Place the flask on a top loading balance.
6. Add sufficient solvent* to bring the weight of the extract to 30 g. Record the weight of the extract (W_{te}) to two decimal places.
   * Add hexane to extracts that are in hexane or in 1:1 hexane:dichloromethane.
   Add dichloromethane to extracts in 100% dichloromethane.
7. Place a Petri dish on the top loading balance and tare to zero. Transfer a 2 g subsample of extract to the Petri dish using a glass Pasteur pipette and bulb. Record the weight (W_{le}) of the extract to two decimal places.
8. Repeat Step 7 with the second Petri dish and subsample. The extract has now been subsampled twice, one subsample in each Petri dish.
9. Place the Petri dishes with extracts in a fumehood at room temperature until the solvent is evaporated, approximately 15 minutes.
10. Place the Petri dishes in the drying oven at 105°C for 30 minutes.
11. Remove the Petri dishes from the oven and allow to cool.
12. Weigh the Petri dishes on the analytical balance. Record the weight of the dish with lipid to four decimal places.
13. Concentrate the remaining 26 g of extract using the same procedure that would be used if lipid analysis on the extract had not been required.

Calculation of Results

Calculation of lipid content is completed by entering the recorded weights into an Excel spreadsheet (Figure 1). The results for all samples in the batch are saved, printed and attached to the Batch List.

1. Login to LIMS, Sample Management, Workstat, and click the Moist/SS/Lipid radio button.
2. Enter the WG and select lipid. Click OK.
3. Clicking on the first line of the data sheet will pop up a data entry screen. For each sample, fill in the weighing data in the spreadsheet fields displayed in the Workup Sheet Information window and click on "OK". Repeat for all samples. OK will move to the next sample for data entry.
4. The spreadsheet calculates the above values using the formulas below. These can be used to double-check the calculated values if necessary. The spreadsheet displays the data entered and results for:
   - Extract weight;
   - Dry lipid;
   - Percent lipid for each aliquot and average percent lipid;
   - Duplicate test results;
   - Recovery correction factor;

   To calculate lipid weight:

   $$W_l = (\text{weight of dish and lipid}) - (\text{weight of dish})$$

   To calculate the percent lipid:

   $$\%lipid = \frac{W_l}{W_s} \times \frac{W_{te}}{W_{le}} \times 100$$

   where W_l = weight of lipid (above);
   W_s = weight of sample taken for analysis;
   W_{te} = weight of total extract (Step 4); and
   W_{le} = weight of extract for lipid analysis (Step 6).
5. Average the percent lipid from the two analyses.

To calculate the lipid factor:

$$Factor = \frac{W_{te}}{W_{te} - W_{le}}$$

where W_{te} = weight of extract
W_{le} = weight of both lipid extracts

To calculate Relative Percent Difference (RPD)

$$RPD = \frac{|Result\,1 - Result\,2| \times 100}{\frac{(Result\,1 + Result\,2)}{2}}$$

6. Review the data for duplicate lipid determinations. Any discrepancies in entered data (i.e., {Dry Lipid + Tare} < Tare) are flagged with a colour code on the spreadsheet. Duplicate test results with a RPD greater than 20% are also flagged. Report RPD differences larger than 20% to the Lab Manager and halt the extraction procedures until a course of action has been determined.
7. If the raw data has been entered correctly, click on “Save Samples”. The results are saved under the workgroup number in the LIMS database. A copy of the spreadsheet is printed at the default printer.
8. All raw data required for the percent lipid calculation must be recorded on the workup sheet. This allows the calculations to be reproduced if necessary. Attach the printed copy of the lipid spreadsheet to the Batch List.

Approval:

Approved 03-Dec-2015 John Cosgrove, President and Senior Technical Director
Shea Hewage, Director of Operations
Dale Hoover, QA Manager

Figure 1. Gravimetric Lipid Determination Calculation Sheet

LIPIDS (gravimetric) Data
Work Group:
Analyst:

To begin: Get Samples

enter the data screen by screen.

To finish: Save Samples.

colours indicate: | less than tare | limits failed | less than dry

Sample #	Extraction Date	Wet Samp Weight	Flask	Flask + Extract	Extract	Aliquot 1 Weight	Tare 1	Dry Lipid +Tare 1	Dry Lipid 1	% Lipid 1	Aliquot 2 Weight	Tare 2	Dry Lipid +Tare 2	Dry Lipid 2	% Lipid 2	Average % Lipids	Duplicate Test	Recovery Correction

ATTACHMENT B

Canadian Association for Laboratory Accreditation Inc.

CALA

Certificate of Accreditation

SGS AXYS Analytical Services Ltd.
SGS Canada Inc.
2045 Mills Road
Sidney, British Columbia

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

CANADIAN ASSOCIATION FOR LABORATORY ACCREDITATION INC. CALA

Accreditation No.:	A2637
Issued On:	February 11, 2016
Accreditation Date:	January 3, 2005
Expiry Date:	August 11, 2018

President & CEO

ilac-MRA CALA

ATTACHMENT C

CALA
Canadian Association for
Laboratory Accreditation Inc.

CALA Directory of Laboratories

Membership Number: 2637
Laboratory Name: SGS AXYS Analytical Services Ltd.
Parent Institution: SGS Canada Inc.
Address: 2045 Mills Road Sidney BC V8L 5X2
Contact: Mr. Dale Hoover
Phone: (250) 655-5800
Fax: (250) 655-5811
Email: dhoover@axys.com

Standard: Conforms with requirements of ISO/IEC 17025
Clients Served:
Revised On: May 24, 2017
Valid To: August 11, 2018

Scope of Accreditation

Pulp (Organic)
Polychlorinated Dioxins/Furans (PCDD/PCDF) - Pulp (021)
MLA-017; modified from EPA 1613B
HIGH RESOLUTION GC/MS - EXTRACTION
1,2,3,4,6,7,8-Heptachlorodioxin
1,2,3,4,6,7,8-Heptachlorofuran
1,2,3,4,7,8-Hexachlorodioxin
1,2,3,4,7,8-Hexachlorofuran
1,2,3,4,7,8,9-Heptachlorofuran
1,2,3,6,7,8-Hexachlorodioxin
1,2,3,6,7,8-Hexachlorofuran
1,2,3,7,8-Pentachlorodioxin
1,2,3,7,8-Pentachlorofuran
1,2,3,7,8,9-Hexachlorodioxin
1,2,3,7,8,9-Hexachlorofuran
2,3,4,6,7,8-Hexachlorofuran
2,3,4,7,8-Pentachlorofuran
2,3,7,8-Tetrachlorodioxin
2,3,7,8-Tetrachlorofuran
Octachlorodioxin
Octachlorofuran

Serum (Organic)
Acylcarnitines, Glycerophospho-lipids, Sphingolipids - Serum/Plasma (063)
MLM-001; IN-HOUSE
FLOW INJECTION - MS/MS
Acetylcarnitine
Butenylcarnitine

† "OSDWA" indicates the appendix is used for the analysis of Ontario drinking water samples, which is subject to the rules and related regulations under the Ontario "Safe Drinking Water Act" (2002).

The list of tests and measurement capabilities for which a laboratory is accredited can change at any time due to circumstances such as scope extensions, voluntary withdrawal of tests by the laboratory and suspension. Scopes are published by the CALA via the Internet at http://www.cala.ca/cala_directories.html

Butyrylcarnitine
Carnitine
Decadienylcarnitine
Decanoylcarnitine
Decenoylcarnitine
Dodecanedioylcarnitine
Dodecanoylcarnitine
Dodecenoylcarnitine
Glutaconylcarnitine
Glutarylcarnitine (Hydroxyhexanoylcarnitine)
Hexadecadienylcarnitine
Hexadecanoylcarnitine
Hexadecenoylcarnitine
Hexanoylcarnitine (Fumarylcarnitine)
Hexenoylcarnitine
Hydroxyhexadecadienylcarnitine
Hydroxyhexadecanoylcarnitine
Hydroxyhexadecenoylcarnitine
Hydroxylbutyrylcarnitine
Hydroxyoctadecenoylcarnitine
Hydroxypropionylcarnitine
Hydroxysphingomyeline C14:1
Hydroxysphingomyeline C16:1
Hydroxysphingomyeline C22:1
Hydroxysphingomyeline C22:2
Hydroxysphingomyeline C24:1
Hydroxytetradecadienylcarnitine
Hydroxytetradecenoylcarnitine
Hydroxyvalerylcarnitine (Methylmalonylcarnitine)
lysoPhosphatidylcholine acyl C14:0
lysoPhosphatidylcholine acyl C16:0
lysoPhosphatidylcholine acyl C16:1
lysoPhosphatidylcholine acyl C17:0
lysoPhosphatidylcholine acyl C18:0
lysoPhosphatidylcholine acyl C18:1
lysoPhosphatidylcholine acyl C18:2
lysoPhosphatidylcholine acyl C20:3
lysoPhosphatidylcholine acyl C20:4
lysoPhosphatidylcholine acyl C24:0
lysoPhosphatidylcholine acyl C26:1
lysoPhosphatidylcholine acyl C28:0
lysoPhosphatidylcholine acyl C28:1
Methylglutarylcarnitine
Nonaylcarnitine
Octadecadienylcarnitine
Octadecanoylcarnitine
Octadecenoylcarnitine
Octanoylcarnitine
Phosphatidylcholine acyl-alkyl C30:0
Phosphatidylcholine acyl-alkyl C30:1
Phosphatidylcholine acyl-alkyl C30:2
Phosphatidylcholine acyl-alkyl C32:1
Phosphatidylcholine acyl-alkyl C32:2

† "OSDWA" indicates the appendix is used for the analysis of Ontario drinking water samples, which is subject to the rules and related regulations under the Ontario "Safe Drinking Water Act" (2002).

The list of tests and measurement capabilities for which a laboratory is accredited can change at any time due to circumstances such as scope extensions, voluntary withdrawal of tests by the laboratory and suspension. Scopes are published by the CALA via the Internet at http://www.cala.ca/cala_directories.html

Phosphatidylcholine acyl-alkyl C34:0
Phosphatidylcholine acyl-alkyl C34:1
Phosphatidylcholine acyl-alkyl C34:2
Phosphatidylcholine acyl-alkyl C34:3
Phosphatidylcholine acyl-alkyl C36:0
Phosphatidylcholine acyl-alkyl C36:1
Phosphatidylcholine acyl-alkyl C36:2
Phosphatidylcholine acyl-alkyl C36:3
Phosphatidylcholine acyl-alkyl C36:4
Phosphatidylcholine acyl-alkyl C36:5
Phosphatidylcholine acyl-alkyl C38:0
Phosphatidylcholine acyl-alkyl C38:1
Phosphatidylcholine acyl-alkyl C38:2
Phosphatidylcholine acyl-alkyl C38:3
Phosphatidylcholine acyl-alkyl C38:5
Phosphatidylcholine acyl-alkyl C38:6
Phosphatidylcholine acyl-alkyl C40:1
Phosphatidylcholine acyl-alkyl C40:2
Phosphatidylcholine acyl-alkyl C40:3
Phosphatidylcholine acyl-alkyl C40:4
Phosphatidylcholine acyl-alkyl C40:5
Phosphatidylcholine acyl-alkyl C40:6
Phosphatidylcholine acyl-alkyl C42:0
Phosphatidylcholine acyl-alkyl C42:1
Phosphatidylcholine acyl-alkyl C42:2
Phosphatidylcholine acyl-alkyl C42:3
Phosphatidylcholine acyl-alkyl C42:4
Phosphatidylcholine acyl-alkyl C42:5
Phosphatidylcholine acyl-alkyl C44:3
Phosphatidylcholine acyl-alkyl C44:4
Phosphatidylcholine acyl-alkyl C44:5
Phosphatidylcholine acyl-alkyl C44:6
Phosphatidylcholine diacyl C24:0
Phosphatidylcholine diacyl C26:0
Phosphatidylcholine diacyl C28:1
Phosphatidylcholine diacyl C30:0
Phosphatidylcholine diacyl C30:2
Phosphatidylcholine diacyl C32:0
Phosphatidylcholine diacyl C32:1
Phosphatidylcholine diacyl C32:2
Phosphatidylcholine diacyl C32:3
Phosphatidylcholine diacyl C34:1
Phosphatidylcholine diacyl C34:2
Phosphatidylcholine diacyl C34:3
Phosphatidylcholine diacyl C34:4
Phosphatidylcholine diacyl C36:0
Phosphatidylcholine diacyl C36:1
Phosphatidylcholine diacyl C36:2
Phosphatidylcholine diacyl C36:3
Phosphatidylcholine diacyl C36:4
Phosphatidylcholine diacyl C36:5
Phosphatidylcholine diacyl C36:6
Phosphatidylcholine diacyl C38:0

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Phosphatidylcholine diacyl C38:1
Phosphatidylcholine diacyl C38:3
Phosphatidylcholine diacyl C38:4
Phosphatidylcholine diacyl C38:5
Phosphatidylcholine diacyl C38:6
Phosphatidylcholine diacyl C40:1
Phosphatidylcholine diacyl C40:2
Phosphatidylcholine diacyl C40:3
Phosphatidylcholine diacyl C40:4
Phosphatidylcholine diacyl C40:5
Phosphatidylcholine diacyl C40:6
Phosphatidylcholine diacyl C42:0
Phosphatidylcholine diacyl C42:1
Phosphatidylcholine diacyl C42:2
Phosphatidylcholine diacyl C42:4
Phosphatidylcholine diacyl C42:5
Phosphatidylcholine diacyl C42:6
Pimelylcarnitine
Propenoylcarnitine
Propionylcarnitine
Sphingomyeline C16:0
Sphingomyeline C16:1
Sphingomyeline C18:0
Sphingomyeline C18:1
Sphingomyeline C20:2
Sphingomyeline C22:3
Sphingomyeline C24:0
Sphingomyeline C24:1
Sphingomyeline C26:0
Sphingomyeline C26:1
Tetradecadienylcarnitine
Tetradecanoylcarnitine
Tetradecenoylcarnitine
Tiglylcarnitine
Valerylcarnitine

Serum (Organic)
Amino Acids and Biogenic Amines - Serum/Plasma (062)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
3-hydroxytyrosine
Acetylornithine
Alanine
alpha-Aminoadipic acid
Arginine
Asparagine
Aspartate
Asymmetric dimethylarginine
Carnosine
Citrulline
Creatinine
Dopamine
Glutamate
Glutamine

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Glycine
Histamine
Histidine
Hydroxyproline
Isoleucine
Kynurenine
Leucine
Lysine
Methionine
Methioninesulfoxide
Nitrotyrosine
Ornithine
Phenylalanine
Phenylethylamine
Proline
Putrescine
Sarcosine
Serine
Serotonin
Spermidine
Spermine
Symmetric dimethylarginine
Taurine
Threonine
Total dimethylarginine
Tryptophan
Tyrosine
Valine

Serum (Organic)

Chlorinated dioxins and furans - Serum/Plasma (047)
MLA-017; modified from EPA 1613B

HI RESOLUTION GC/MS

1,2,3,4,6,7,8-Heptachlorodioxin
1,2,3,4,6,7,8-Heptachlorofuran
1,2,3,4,7,8-Hexachlorodioxin
1,2,3,4,7,8-Hexachlorofuran
1,2,3,4,7,8,9-Heptachlorofuran
1,2,3,6,7,8-Hexachlorodioxin
1,2,3,6,7,8-Hexachlorofuran
1,2,3,7,8-Pentachlorodioxin
1,2,3,7,8-Pentachlorofuran
1,2,3,7,8,9-Hexachlorodioxin
1,2,3,7,8,9-Hexachlorofuran
2,3,4,6,7,8-Hexachlorofuran
2,3,4,7,8-Pentachlorofuran
2,3,7,8-Tetrachlorodioxin
2,3,7,8-Tetrachlorofuran
Octachlorodioxin
Octachlorofuran

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Serum (Organic)
HBCDD - Serum/Plasma (051)
MLA-070; IN-HOUSE
LC-MS/MS
alpha-hexabromocyclododecane (a-HBCDD)
beta-hexabromocyclododecane (b-HBCDD)
gamma-hexabromocyclododecane (g-HBCDD)

Serum (Organic)
Pesticides - Serum/Plasma (046)
MLA-028; IN-HOUSE
HI RESOLUTION GC/MS
Aldrin
alpha-Hexachlorocyclohexane (a-HCH, a-BHC)
beta-Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
delta- Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulphan I (alpha-Endosulphan)
Endosulphan II (beta-Endosulphan)
Endosulphan Sulphate
Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor
Heptachlor Epoxide
Hexachlorobenzene
Lindane (gamma BHC)
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
p,p'-Methoxychlor
trans-Chlordane
trans-Nonachlor

Serum (Organic)
Pesticides - Serum/Plasma (055)
MLA-007; IN-HOUSE
GC/ECD
delta- Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulphan I (alpha-Endosulphan)
Endosulphan II (beta-Endosulphan)
Endosulphan Sulphate
Endrin
Endrin Ketone
Heptachlor Epoxide
p,p'-Methoxychlor

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Serum (Organic)
Pesticides - Serum/Plasma (056)
MLA-007; IN-HOUSE
LOW RESOLUTION - GC/MS
Aldrin
alpha-Hexachlorocyclohexane (a-HCH, a-BHC)
beta-Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
Heptachlor
Hexachlorobenzene
Lindane (gamma BHC)
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
trans-Chlordane
trans-Nonachlor

Serum (Organic)
PFC - Serum/Plasma (050)
MLA-042; IN-HOUSE
LC-MS/MS
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate (PFBA)
Perfluorodecanoate (PFDA)
Perfluorododecanoate (PFDoA)
Perfluorohepanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanoate (PFNA)
Perfluorooctane sulfonamide (PFOSA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanoate (PFOA)
Perfluoropentanoate (PFPeA)
Perfluoroundecanoate (PFUnA)

Serum (Organic)
Polybrominated diphenylethers - Serum/Plasma (045)
MLA-033; modified from EPA 1614
HI RESOLUTION GC/MS
2',3,4-tribromodiphenylether (BDE 33)
2,2',3,3',4,4',5,5',6-nonabromodiphenylether (BDE 206)
2,2',3,3',4,4',5,5',6,6' -decabromodiphenylether (BDE 209)
2,2',3,3',4,5,5',6,6' -nonabromodiphenylether (BDE 208)
2,2 3,3,4,4,5,6,6 -nonabromodiphenylether (BDE 207)
2,2',3,4,4',5',6-heptabromodiphenylether (BDE 183)
2,2',3,4,4',5,6-heptabromodiphenylether (BDE 181)
2,2',3,4,4',6' -hexabromodiphenylether (BDE 140)
2,2',3,4,4' -pentabromodiphenylether (BDE 85)
2,2',4-tribromodiphenylether (BDE 17)

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2,2',4,4',5',6-hexabromodiphenylether (BDE 154)
2,2',4,4',5-pentabromodiphenylether (BDE 99)
2,2',4,4',5,5' -hexabromodiphenylether (BDE 153)
2,2',4,4',6-pentabromodiphenylether (BDE 100)
2,2',4,4',6,6' -hexabromodiphenylether (BDE 155)
2,2',4,4' -tetrabromodiphenylether (BDE 47)
2,2',4,5' -tetrabromodiphenylether (BDE 49)
2,3',4-tribromodiphenylether (BDE 25)
2,3',4,4',6-pentabromodiphenylether (BDE 119)
2,3',4,4' -tetrabromodiphenylether (BDE 66)
2,3,3',4,4',5,6-heptabromodiphenylether (BDE 190)
2,3,3',4,4' -pentabromodiphenylether (BDE 105)
2,3,4,4',5,6-hexabromodiphenylether (BDE 166)
2,3,4,5,6-pentabromodiphenylether (BDE 116)
2,4'-dibromodiphenylether (BDE 8)
2,4-dibromodiphenylether (BDE 7)
2,4,4',6-tetrabromodiphenylether (BDE 75)
2,4,4' -tribromodiphenylether (BDE 28)
2,4,6-tribromodiphenylether (BDE 30)
2,6-dibromodiphenylether (BDE 10)
3,3',4-tribromodiphenylether (BDE 35)
3,3',4,4',5-pentabromodiphenylether (BDE 126)
3,3',4,4' -tetrabromodiphenylether (BDE 77)
3,3'-dibromodiphenylether (BDE 11)
3,4'-dibromodiphenylether (BDE 13)
3,4-dibromodiphenylether (BDE 12)
3,4,4'-tribromodiphenylether (BDE 37)
4,4'-dibromodiphenylether (BDE 15)

Serum (Organic)
Polychorinated Biphenyls (PCB) - Serum/Plasma (048)
MLA-010; modified from EPA 1668A
HI RESOLUTION GC/MS
PCB 001
PCB 002
PCB 003
PCB 004
PCB 005
PCB 006
PCB 007
PCB 008
PCB 009
PCB 010
PCB 011
PCB 012
PCB 013
PCB 014
PCB 015
PCB 016
PCB 017
PCB 018
PCB 019
PCB 020
PCB 021

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PCB 022
PCB 023
PCB 024
PCB 025
PCB 026
PCB 027
PCB 028
PCB 029
PCB 030
PCB 031
PCB 032
PCB 033
PCB 034
PCB 035
PCB 036
PCB 037
PCB 038
PCB 039
PCB 040
PCB 041
PCB 042
PCB 043
PCB 044
PCB 045
PCB 046
PCB 047
PCB 048
PCB 049
PCB 050
PCB 051
PCB 052
PCB 053
PCB 054
PCB 055
PCB 056
PCB 057
PCB 058
PCB 059
PCB 060
PCB 061
PCB 062
PCB 063
PCB 064
PCB 065
PCB 066
PCB 067
PCB 068
PCB 069
PCB 070
PCB 071
PCB 072
PCB 073
PCB 074

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PCB 075
PCB 076
PCB 077
PCB 078
PCB 079
PCB 080
PCB 081
PCB 082
PCB 083
PCB 084
PCB 085
PCB 086
PCB 087
PCB 088
PCB 089
PCB 090
PCB 091
PCB 092
PCB 093
PCB 094
PCB 095
PCB 096
PCB 097
PCB 098
PCB 099
PCB 100
PCB 101
PCB 102
PCB 103
PCB 104
PCB 105
PCB 106
PCB 107
PCB 108
PCB 109
PCB 110
PCB 111
PCB 112
PCB 113
PCB 114
PCB 115
PCB 116
PCB 117
PCB 118
PCB 119
PCB 120
PCB 121
PCB 122
PCB 123
PCB 124
PCB 125
PCB 126
PCB 127

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PCB 128
PCB 129
PCB 130
PCB 131
PCB 132
PCB 133
PCB 134
PCB 135
PCB 136
PCB 137
PCB 138
PCB 139
PCB 140
PCB 141
PCB 142
PCB 143
PCB 144
PCB 145
PCB 146
PCB 147
PCB 148
PCB 149
PCB 150
PCB 151
PCB 152
PCB 153
PCB 154
PCB 155
PCB 156
PCB 157
PCB 158
PCB 159
PCB 160
PCB 161
PCB 162
PCB 163
PCB 164
PCB 165
PCB 166
PCB 167
PCB 168
PCB 169
PCB 170
PCB 171
PCB 172
PCB 173
PCB 174
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 180

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PCB 181
PCB 182
PCB 183
PCB 184
PCB 185
PCB 186
PCB 187
PCB 188
PCB 189
PCB 190
PCB 191
PCB 192
PCB 193
PCB 194
PCB 195
PCB 196
PCB 197
PCB 198
PCB 199
PCB 200
PCB 201
PCB 202
PCB 203
PCB 204
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Monochloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs

Serum (Organic)
Polychorinated Biphenyls (PCB) - Serum/Plasma (049)
MLA-901; IN-HOUSE
LOW RESOLUTION GC/MS
PCB 118
PCB 138
PCB 146
PCB 153
PCB 156
PCB 170
PCB 180
PCB 187
PCB 194
PCB 74
PCB 99

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Serum (Organic)
Sugars, Bile Acids, Fatty Acids - Serum/Plasma (061)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
chenodeoxycholic acid
cholic acid
deoxycholic acid
glycochenodeoxycholic acid
glycocholic acid
glycodeoxycholic acid
Hexose (sum isomers)
lithocholic acid
taurochenodeoxycholic acid
taurocholic acid
taurodeoxycholic acid
taurolithocholic acid
tauroursodexoycholic acid
ursodexoycholic acid

Serum (Organic)
TBBPA - Serum/Plasma (052)
MLA-079; IN-HOUSE
LC-MS/MS
Tetrabromobisphenol A (TBBPA)

Solids (Organic)
Dioxins, Furans (PCDD/PCDF) - Soil, Sediment (019)
MLA-017; EPA 1613B
HIGH RESOLUTION GC/MS - EXTRACTION
1,2,3,4,6,7,8-Heptachlorodioxin
1,2,3,4,6,7,8-Heptachlorofuran
1,2,3,4,7,8-Hexachlorodioxin
1,2,3,4,7,8-Hexachlorofuran
1,2,3,4,7,8,9-Heptachlorofuran
1,2,3,6,7,8-Hexachlorodioxin
1,2,3,6,7,8-Hexachlorofuran
1,2,3,7,8-Pentachlorodioxin
1,2,3,7,8-Pentachlorofuran
1,2,3,7,8,9-Hexachlorodioxin
1,2,3,7,8,9-Hexachlorofuran
2,3,4,6,7,8-Hexachlorofuran
2,3,4,7,8-Pentachlorofuran
2,3,7,8-Tetrachlorodioxin
2,3,7,8-Tetrachlorofuran
Octachlorodioxin
Octachlorofuran

Solids (Organic)
Organochlorine Pesticides (OCP) - Soil/Sediment (004)
MLA-007; modified from EPA 8081A
GC/ECD - EXTRACTION
delta-Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulfan I (alpha-Endosulphan)
Endosulfan II (beta-Endosulphan)
Endosulphan Sulphate

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Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor Epoxide
p,p'-Methoxychlor

Solids (Organic)
Per- and Polyfluoroalkyl Substances (PFAS) in Solid Samples - Solids (068)
MLA-110; IN-HOUSE
LC-MS/MS
4:2 fluorotelomersulfonate (4:2 FTS)
6:2 fluorotelomersulfonate (6:2 FTS)
8:2 fluorotelomersulfonate (8:2 FTS)
N-Ethylperfluorooctanesulfonamide (N-EtFOSA)
N-Ethylperfluorooctanesulfonamidoethanol (N-EtFOSE)
N-MEthylperfluorooctanesulfonamidoacetic acid (N-
N-Methylperfluorooctanesulfonamidoacetic acid (N-
N-Methylperfluorooctanesulfonamide (N-MeFOSA)
N-Methylperfluorooctanesulfonamidoethanol (N-MeFOSE)
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate (PFBA)
Perfluorodecanesulfonate (PFDS)
Perfluorodecanoate (PFDA)
Perfluorododecanesulfonate (PFDoS)
Perfluorododecanoate (PFDoA)
Perfluoroheptanesulfonate (PFHpS)
Perfluoroheptanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanesulfonate (PFNS)
Perfluorononanoate (PFNA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanesulfonamide (PFOSA), a.k.a FOSA
Perfluorooctanoate (PFOA)
Perfluoropentanesulfonate (PFPeS)
Perfluoropentanoate (PFPeA)
Perfluoroundecanoate (PFUnA)

Solids (Organic)
Perfluorinated Organics - Soil, Sediment (033)
MLA-041; IN-HOUSE
LC-MS/MS - EXTRACTION
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate PFBA
Perfluorodecanoate (PFDA)
Perfluorododecanoate (PFDoA)
Perfluorohepanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanoate (PFNA)
Perfluorooctane sulfonamide (PFOSA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanoate (PFOA)
Perfluoropentanoate (PFPeA)
Perfluoroundecanoate (PFUnA)

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Solids (Organic)
Pesticides - Soil, Sediment (030)
MLA-028; IN-HOUSE

HIGH RESOLUTION GC/MS
Aldrin
alpha-Hexachlorocyclohexane (a-HCH, a-BHC)
beta-Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
delta- Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulphan I (alpha-Endosulphan)
Endosulphan II (beta-Endosulphan)
Endosulphan Sulphate
Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor
Heptachlor Epoxide
Hexachlorobenzene
Lindane (gamma-BHC)
Methoxychlor
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
trans-Chlordane (gamma-chlordane)
trans-Nonachlor

Solids (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Solids (039)
MLA-075; EPA 1694

LC-MS/MS - ACID EXTRACTION, FORMIC ACID
10-hydroxy-amitriptyline
1,7-Dimethylxanthine
Acetaminophen
Alprazolam
Amitriptyline
Amlodipine
Azithromycin
Benzoylecgonine
Benztropine
Betamethasone
Caffeine
Carbadox
Carbamazepine
Cefotaxime
Ciprofloxacin
Clarithromycin
Clinafloxacin

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Cloxacillin
Cocaine
DEET (N,N-diethyl-m-toluamide)
Dehydronifedipine
Desmethyldiltiazem
Diazepam
Digoxigenin
Digoxin
Diltiazem
Diphenhydramine
Enrofloxacin
Erythromycin
Flumequine
Fluocinonide
Fluoxetine
Fluticasone propionate
Hydrocortisone
Lincomycin
Lomefloxacin
Meprobamate
Methylprednisolone
Metoprolol
Miconazole
Norfloxacin
Norfluoxetine
Norgestimate
Norverapamil
Ofloxacin
Ormetoprim
Oxacillin
Oxolinic acid
Paroxetine
Penicillin G
Penicillin V
Prednisolone
Prednisone
Promethazine
Propoxyphene
Propranolol
Roxithromycin
Sarafloxacin
Sertraline
Simvastatin
Sulfachloropyridazine
Sulfadiazine
Sulfadimethoxine
Sulfamerazine
Sulfamethazine
Sulfamethizole
Sulfamethoxazole
Sulfanilamide
Sulfathiazole
Theophylline

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Thiabendazole
Trenbolone
Trenbolone acetate
Trimethoprim
Tylosin
Valsartan
Verapamil
Virginiamycin

Solids (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Solids (040)
MLA-075; EPA 1694
LC-MS/MS - ACID EXTRACTION, ACN
4-Epianhydrochlortetracycline (EACTC)
4-Epianhydrotetracycline (EATC)
4-Epichlortetracycline (ECTC)
4-Epioxytetracycline (EOTC)
4-Epitetracycline (ETC)
Anhydrochlortetracycline (ACTC)
Anhydrotetracycline (ATC)
Chlortetracycline (CTC)
Demeclocycline
Doxycycline
Isochlortetracycline (ICTC)
Minocycline
Oxytetracycline (OTC)
Tetracycline (TC)

Solids (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Solids (041)
MLA-075; EPA 1694
LC-MS/MS - ACID EXTRACTION, AMMONIUM ACETATE
2-hydroxy-ibuprofen
Bisphenol A
Furosemide
Gemfibrozil
Glipizide
Glyburide
Hydrochlorothiazide
Ibuprofen
Naproxen
Triclocarban
Triclosan
Warfarin

Solids (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Solids (042)
MLA-075; EPA 1694
LC-MS/MS - BASE EXTRACTION
Albuterol
Amphetamine
Atenolol
Atorvastatin
Cimetidine
Clonidine
Codeine

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Cotinine
Enalapril
Hydrocodone
Metformin
Oxycodone
Ranitidine
Triamterene

Solids (Organic)

Polybrominated Diphenylethers (PBDE) - Soil, Sediment (026)
MLA-033; EPA 1614

HIGH RESOLUTION GC/MS

1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE)
2',3,4-tribromodiphenylether (BDE 33)
2,2',3,3',4,4',5,5',6-nonabromodiphenylether (BDE 206)
2,2',3,3',4,4',5,5',6,6' -decabromodiphenylether (BDE 209)
2,2',3,3',4,5,5',6,6' -nonabromodiphenylether (BDE 208)
2,2 3,3,4,4,5,6,6 -nonabromodiphenylether (BDE 207)
2,2',3,4,4',5',6-heptabromodiphenylether (BDE 183)
2,2',3,4,4',5,6-heptabromodiphenylether (BDE 181)
2,2',3,4,4',6' -hexabromodiphenylether (BDE 140)
2,2',3,4,4' -pentabromodiphenylether (BDE 85)
2,2',4-tribromodiphenylether (BDE 17)
2,2',4,4',5',6-hexabromodiphenylether (BDE 154)
2,2',4,4',5-pentabromodiphenylether (BDE 99)
2,2',4,4',5,5' -hexabromodiphenylether (BDE 153)
2,2',4,4',6-pentabromodiphenylether (BDE 100)
2,2',4,4',6,6' -hexabromodiphenylether (BDE 155)
2,2',4,4' -tetrabromodiphenylether (BDE 47)
2,2',4,5' -tetrabromodiphenylether (BDE 49)
2,3',4-tribromodiphenylether (BDE 25)
2,3',4,4',6-pentabromodiphenylether (BDE 119)
2,3',4,4' -tetrabromodiphenylether (BDE 66)
2,3,3',4,4',5,6-heptabromodiphenylether (BDE 190)
2,3,3',4,4' -pentabromodiphenylether (BDE 105)
2,3,4,4',5,6-hexabromodiphenylether (BDE 166)
2,3,4,5,6-pentabromodiphenylether (BDE 116)
2,4'-dibromodiphenylether (BDE 8)
2,4-dibromodiphenylether (BDE 7)
2,4,4',6-tetrabromodiphenylether (BDE 75)
2,4,4' -tribromodiphenylether (BDE 28)
2,4,6-tribromodiphenylether (BDE 30)
2,6-dibromodiphenylether (BDE 10)
3,3',4-tribromodiphenylether (BDE 35)
3,3',4,4',5-pentabromodiphenylether (BDE 126)
3,3',4,4' -tetrabromodiphenylether (BDE 77)
3,3'-dibromodiphenylether (BDE 11)
3,4'-dibromodiphenylether (BDE 13)
3,4-dibromodiphenylether (BDE 12)
3,4,4'-tribromodiphenylether (BDE 37)
4,4'-dibromodiphenylether (BDE 15)
Decabromodiphenylethane (DBDPE)
Hexabromobenzene (HBB)
Pentabromoethylbenzene (PBEB)

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Solids (Organic)
Polychlorinated Biphenyls (PCB) - Soil, Sediment (023)
MLA-010; EPA 1668A
HIGH RESOLUTION GC/MS
PCB 001
PCB 002
PCB 003
PCB 004
PCB 005
PCB 006
PCB 007
PCB 008
PCB 009
PCB 010
PCB 011
PCB 012
PCB 013
PCB 014
PCB 015
PCB 016
PCB 017
PCB 018
PCB 019
PCB 020
PCB 021
PCB 022
PCB 023
PCB 024
PCB 025
PCB 026
PCB 027
PCB 028
PCB 029
PCB 030
PCB 031
PCB 032
PCB 033
PCB 034
PCB 035
PCB 036
PCB 037
PCB 038
PCB 039
PCB 040
PCB 041
PCB 042
PCB 043
PCB 044
PCB 045
PCB 046
PCB 047
PCB 048
PCB 049

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PCB 050
PCB 051
PCB 052
PCB 053
PCB 054
PCB 055
PCB 056
PCB 057
PCB 058
PCB 059
PCB 060
PCB 061
PCB 062
PCB 063
PCB 064
PCB 065
PCB 066
PCB 067
PCB 068
PCB 069
PCB 070
PCB 071
PCB 072
PCB 073
PCB 074
PCB 075
PCB 076
PCB 077
PCB 078
PCB 079
PCB 080
PCB 081
PCB 082
PCB 083
PCB 084
PCB 085
PCB 086
PCB 087
PCB 088
PCB 089
PCB 090
PCB 091
PCB 092
PCB 093
PCB 094
PCB 095
PCB 096
PCB 097
PCB 098
PCB 099
PCB 100
PCB 101
PCB 102

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PCB 103
PCB 104
PCB 105
PCB 106
PCB 107
PCB 108
PCB 109
PCB 110
PCB 111
PCB 112
PCB 113
PCB 114
PCB 115
PCB 116
PCB 117
PCB 118
PCB 119
PCB 120
PCB 121
PCB 122
PCB 123
PCB 124
PCB 125
PCB 126
PCB 127
PCB 128
PCB 129
PCB 130
PCB 131
PCB 132
PCB 133
PCB 134
PCB 135
PCB 136
PCB 137
PCB 138
PCB 139
PCB 140
PCB 141
PCB 142
PCB 143
PCB 144
PCB 145
PCB 146
PCB 147
PCB 148
PCB 149
PCB 150
PCB 151
PCB 152
PCB 153
PCB 154
PCB 155

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PCB 156
PCB 157
PCB 158
PCB 159
PCB 160
PCB 161
PCB 162
PCB 163
PCB 164
PCB 165
PCB 166
PCB 167
PCB 168
PCB 169
PCB 170
PCB 171
PCB 172
PCB 173
PCB 174
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 180
PCB 181
PCB 182
PCB 183
PCB 184
PCB 185
PCB 186
PCB 187
PCB 188
PCB 189
PCB 190
PCB 191
PCB 192
PCB 193
PCB 194
PCB 195
PCB 196
PCB 197
PCB 198
PCB 199
PCB 200
PCB 201
PCB 202
PCB 203
PCB 204
PCB 205
PCB 206
PCB 207
PCB 208

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PCB 209
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Monochloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs

Solids (Organic)
Polychlorinated Biphenyls (PCB)/Organochlorine Pesticides (OCP) - Soil, Sediment (003)
MLA-007; modified from EPA 8270C
GC/MS - EXTRACTION
Aldrin
alpha-hexachlorocyclohexane (a-HCH, A-BHC)
Aroclor 1221
Aroclor 1232
Aroclor 1242/1016
Aroclor 1248
Aroclor 1254
Aroclor 1260
Aroclor 1268
beta-hexachlorocyclohexane (b-BHC, a-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
Heptachlor
Hexachlorobenzene
Lindane (gamma-BHC)
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
PCB 101/90/89
PCB 105/127
PCB 107/109
PCB 110
PCB 114
PCB 118/106
PCB 126
PCB 128
PCB 129
PCB 130
PCB 131/142
PCB 134/143
PCB 136
PCB 137
PCB 138/163/164
PCB 141

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PCB 144/135
PCB 146
PCB 149/139
PCB 15
PCB 151
PCB 153
PCB 156
PCB 157
PCB 158/160
PCB 16/32
PCB 169
PCB 17
PCB 170/190
PCB 171
PCB 172/192
PCB 174/181
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 18
PCB 180
PCB 183
PCB 185
PCB 187/182
PCB 189
PCB 19
PCB 191
PCB 193
PCB 194
PCB 195
PCB 196/203
PCB 197
PCB 198
PCB 199
PCB 201
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
PCB 22
PCB 24/27
PCB 25
PCB 26
PCB 28
PCB 31
PCB 33/20/21
PCB 40
PCB 41/71/64/68
PCB 42/59
PCB 44

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PCB 45
PCB 46
PCB 47/48/75
PCB 49/43
PCB 52/73
PCB 56/60
PCB 66/80
PCB 70/76
PCB 74/61
PCB 77
PCB 8/5
PCB 83/108
PCB 84
PCB 85/120
PCB 87/115/116
PCB 91
PCB 95/93
PCB 97/86
PCB 99
Total Decachloro-PCBs
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total PCB
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs
Toxaphene
trans-Chlordane (gamma-chlordane)
trans-Nonachlor

Solids (Organic)

Polycyclic Aromatic Hydrocarbons (PAH) - Soil, Sediment (011)
MLA-021; modified from EPA 1625

GC/MS - DIGESTION

1-Methylchrysene
1-Methylnaphthalene
1-Methylphenanthrene
1,2-Dimethylnaphthalene
1,2,6-Trimethylphenanthrene
1,4,6,7-Tetramethylnaphthalene
1,7-Dimethylfluorene
1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene
2-Methylanthracene
2-Methylfluorene
2-Methylnaphthalene
2-Methylphenanthrene
2/3-Methyldibenzothiophenes
2,3,5-Trimethylnaphthalene
2,3,6-Trimethylnaphthalene
2,4-Dimethyldibenzothiophene

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2,6-Dimethylnaphthalene
2,6-Dimethylphenanthrene
3-Methylfluoranthene/ Benzo(a)fluorene
3-Methylphenanthrene
3,6-Dimethylphenanthrene
5/6-Methylchrysenes
5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene
9/4-Methylphenanthrenes
Acenaphthene
Acenaphthylene
Anthracene
Benz (e) pyrene
Benzo (a) anthracene
Benzo (a) pyrene
Benzo (b) fluoranthene
Benzo (g,h,i) perylene
Biphenyl
C1-Acenaphthenes
C1-Benz(a)anthracenes/chrysenes
C1-Benzofluoranthenes/ Benzopyrenes
C1-Biphenyls
C1-Dibenzothiophene
C1-Fluoranthenes/Pyrenes
C1-Fluorenes
C1-Naphthalenes
C1-Phenanthrenes/Anthracenes
C2-Benz(a)anthracenes/Chrysenes
C2-Benzofluoranthenes/ Benzopyrenes
C2-Biphenyls
C2-Dibenzothiophene
C2-Fluoranthenes/Pyrenes
C2-Fluorenes
C2-Naphthalenes
C2-Phenanthrenes/Anthracenes
C3-Benz(a)anthracenes/Chrysenes
C3-Dibenzothiophene
C3-Fluoranthenes/Pyrenes
C3-Fluorenes
C3-Naphthalenes
C3-Phenanthrenes/Anthracenes
C4-Benz(a)anthracenes/Chrysenes
C4-Dibenzothiophene
C4-Fluoranthenes/Pyrenes
C4-Naphthalenes
C4-Phenanthrenes/Anthracenes
Chrysene
Dibenzo (a,h) anthracene
Dibenzothiophene
Fluoranthene
Fluorene
Indeno (1,2,3 - cd) pyrene
Naphthalene

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Perylene
Phenanthrene
Pyrene
Retene

Tissue
Perfluorinated Organics - Tissue (034)
MLA-043; IN-HOUSE
LC-MS/MS - EXTRACTION
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate PFBA
Perfluorodecanoate (PFDA)
Perfluorododecanoate (PFDoA)
Perfluorohepanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanoate (PFNA)
Perfluorooctane sulfonamide (PFOSA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanoate (PFOA)
Perfluoropentanoate (PFPeA)
Perfluoroundecanoate (PFUnA)

Tissue (Organic)
Acylcarnitines, Glycerophospho-lipids, Sphingolipids - Tissue (060)
MLM-001; IN-HOUSE
FLOW INJECTION - MS/MS
Acetylcarnitine
Butenylcarnitine
Butyrylcarnitine
Carnitine
Decadienylcarnitine
Decanoylcarnitine
Decenoylcarnitine
Dodecanedioylcarnitine
Dodecanoylcarnitine
Dodecenoylcarnitine
Glutaconylcarnitine
Glutarylcarnitine (Hydroxyhexanoylcarnitine)
Hexadecadienylcarnitine
Hexadecanoylcarnitine
Hexadecenoylcarnitine
Hexanoylcarnitine (Fumarylcarnitine)
Hexenoylcarnitine
Hydroxyhexadecadienylcarnitine
Hydroxyhexadecanoylcarnitine
Hydroxyhexadecenoylcarnitine
Hydroxylbutyrylcarnitine
Hydroxyoctadecenoylcarnitine
Hydroxypropionylcarnitine
Hydroxysphingomyeline C14:1
Hydroxysphingomyeline C16:1
Hydroxysphingomyeline C22:1
Hydroxysphingomyeline C22:2
Hydroxysphingomyeline C24:1

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Hydroxytetradecadienylcarnitine
Hydroxytetradecenoylcarnitine
Hydroxyvalerylcarnitine (Methylmalonylcarnitine)
lysoPhosphatidylcholine acyl C14:0
lysoPhosphatidylcholine acyl C16:0
lysoPhosphatidylcholine acyl C16:1
lysoPhosphatidylcholine acyl C17:0
lysoPhosphatidylcholine acyl C18:0
lysoPhosphatidylcholine acyl C18:1
lysoPhosphatidylcholine acyl C18:2
lysoPhosphatidylcholine acyl C20:3
lysoPhosphatidylcholine acyl C20:4
lysoPhosphatidylcholine acyl C24:0
lysoPhosphatidylcholine acyl C26:1
lysoPhosphatidylcholine acyl C28:0
lysoPhosphatidylcholine acyl C28:1
Methylglutarylcarnitine
Nonaylcarnitine
Octadecadienylcarnitine
Octadecanoylcarnitine
Octadecenoylcarnitine
Octanoylcarnitine
Phosphatidylcholine acyl-alkyl C30:0
Phosphatidylcholine acyl-alkyl C30:1
Phosphatidylcholine acyl-alkyl C30:2
Phosphatidylcholine acyl-alkyl C32:1
Phosphatidylcholine acyl-alkyl C32:2
Phosphatidylcholine acyl-alkyl C34:0
Phosphatidylcholine acyl-alkyl C34:1
Phosphatidylcholine acyl-alkyl C34:2
Phosphatidylcholine acyl-alkyl C34:3
Phosphatidylcholine acyl-alkyl C36:0
Phosphatidylcholine acyl-alkyl C36:1
Phosphatidylcholine acyl-alkyl C36:2
Phosphatidylcholine acyl-alkyl C36:3
Phosphatidylcholine acyl-alkyl C36:4
Phosphatidylcholine acyl-alkyl C36:5
Phosphatidylcholine acyl-alkyl C38:0
Phosphatidylcholine acyl-alkyl C38:1
Phosphatidylcholine acyl-alkyl C38:2
Phosphatidylcholine acyl-alkyl C38:3
Phosphatidylcholine acyl-alkyl C38:5
Phosphatidylcholine acyl-alkyl C38:6
Phosphatidylcholine acyl-alkyl C40:1
Phosphatidylcholine acyl-alkyl C40:2
Phosphatidylcholine acyl-alkyl C40:3
Phosphatidylcholine acyl-alkyl C40:4
Phosphatidylcholine acyl-alkyl C40:5
Phosphatidylcholine acyl-alkyl C40:6
Phosphatidylcholine acyl-alkyl C42:0
Phosphatidylcholine acyl-alkyl C42:1
Phosphatidylcholine acyl-alkyl C42:2
Phosphatidylcholine acyl-alkyl C42:3

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Phosphatidylcholine acyl-alkyl C42:4
Phosphatidylcholine acyl-alkyl C42:5
Phosphatidylcholine acyl-alkyl C44:3
Phosphatidylcholine acyl-alkyl C44:4
Phosphatidylcholine acyl-alkyl C44:5
Phosphatidylcholine acyl-alkyl C44:6
Phosphatidylcholine diacyl C24:0
Phosphatidylcholine diacyl C26:0
Phosphatidylcholine diacyl C28:1
Phosphatidylcholine diacyl C30:0
Phosphatidylcholine diacyl C30:2
Phosphatidylcholine diacyl C32:0
Phosphatidylcholine diacyl C32:1
Phosphatidylcholine diacyl C32:2
Phosphatidylcholine diacyl C32:3
Phosphatidylcholine diacyl C34:1
Phosphatidylcholine diacyl C34:2
Phosphatidylcholine diacyl C34:3
Phosphatidylcholine diacyl C34:4
Phosphatidylcholine diacyl C36:0
Phosphatidylcholine diacyl C36:1
Phosphatidylcholine diacyl C36:2
Phosphatidylcholine diacyl C36:3
Phosphatidylcholine diacyl C36:4
Phosphatidylcholine diacyl C36:5
Phosphatidylcholine diacyl C36:6
Phosphatidylcholine diacyl C38:0
Phosphatidylcholine diacyl C38:1
Phosphatidylcholine diacyl C38:3
Phosphatidylcholine diacyl C38:4
Phosphatidylcholine diacyl C38:5
Phosphatidylcholine diacyl C38:6
Phosphatidylcholine diacyl C40:1
Phosphatidylcholine diacyl C40:2
Phosphatidylcholine diacyl C40:3
Phosphatidylcholine diacyl C40:4
Phosphatidylcholine diacyl C40:5
Phosphatidylcholine diacyl C40:6
Phosphatidylcholine diacyl C42:0
Phosphatidylcholine diacyl C42:1
Phosphatidylcholine diacyl C42:2
Phosphatidylcholine diacyl C42:4
Phosphatidylcholine diacyl C42:5
Phosphatidylcholine diacyl C42:6
Pimelylcarnitine
Propenoylcarnitine
Propionylcarnitine
Sphingomyeline C16:0
Sphingomyeline C16:1
Sphingomyeline C18:0
Sphingomyeline C18:1
Sphingomyeline C20:2
Sphingomyeline C22:3

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Sphingomyeline C24:0
Sphingomyeline C24:1
Sphingomyeline C26:0
Sphingomyeline C26:1
Tetradecadienylcarnitine
Tetradecanoylcarnitine
Tetradecenoylcarnitine
Tiglylcarnitine
Valerylcarnitine

Tissue (Organic)
Amino Acids and Biogenic Amines - Tissue (059)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
3-hydroxytyrosine
Acetylornithine
Alanine
alpha-Aminoadipic acid
Arginine
Asparagine
Aspartate
Asymmetric dimethylarginine
Carnosine
Citrulline
Creatinine
Dopamine
Glutamate
Glutamine
Glycine
Histamine
Histidine
Hydroxyproline
Isoleucine
Kynurenine
Leucine
Lysine
Methionine
Methioninesulfoxide
Nitrotyrosine
Ornithine
Phenylalanine
Phenylethylamine
Proline
Putrescine
Sarcosine
Serine
Serotonin
Spermidine
Spermine
Symmetric dimethylarginine
Taurine
Threonine
Total dimethylarginine
Tryptophan

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Tyrosine
Valine

Tissue (Organic)
Organochlorine Pesticides (OCP) - Tissue (008)
MLA-007; modified from EPA 8081A
GC/ECD - EXTRACTION
delta-Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulfan I (alpha-Endosulphan)
Endosulfan II (beta-Endosulphan)
Endosulphan Sulphate
Endrin
Endrin Ketone
Heptachlor Epoxide
p,p'-Methoxychlor

Tissue (Organic)
Pesticides - Tissue (029)
MLA-028; IN-HOUSE
HIGH RESOLUTION GC/MS
Aldrin
alpha-Hexachlorocyclohexane (a-HCH, a-BHC)
beta-Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
delta- Hexachlorocyclohexane
Dieldrin
Endosulphan I (alpha-endosulphan)
Endosulphan II (beta-endosulphan)
Endosulphan Sulphate
Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor
Heptachlor Epoxide
Hexachlorobenzene
Lindane (gamma BHC)
Methoxychlor
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
trans-Chlordane (gamma-chlordane)
trans-Nonachlor

Tissue (Organic)
Polybrominated Diphenylethers - Tissue (025)
MLA-033; modified from EPA 1614
HIGH RESOLUTION GC/MS
1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE)
2',3,4-tribromodiphenylether (BDE 33)

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2,2',3,3',4,4',5,5',6-nonabromodiphenylether (BDE 206)
2,2',3,3',4,4',5,5',6,6' -decabromodiphenylether (BDE 209)
2,2',3,3',4,5,5',6,6' -nonabromodiphenylether (BDE 208)
2,2 3,3,4,4,5,6,6 -nonabromodiphenylether (BDE 207)
2,2',3,4,4',5',6-heptabromodiphenylether (BDE 183)
2,2',3,4,4',5,6-heptabromodiphenylether (BDE 181)
2,2',3,4,4',6' -hexabromodiphenylether (BDE 140)
2,2',3,4,4' -pentabromodiphenylether (BDE 85)
2,2',4-tribromodiphenylether (BDE 17)
2,2',4,4',5',6-hexabromodiphenylether (BDE 154)
2,2',4,4',5-pentabromodiphenylether (BDE 99)
2,2',4,4',5,5' -hexabromodiphenylether (BDE 153)
2,2',4,4',6-pentabromodiphenylether (BDE 100)
2,2',4,4',6,6' -hexabromodiphenylether (BDE 155)
2,2',4,4' -tetrabromodiphenylether (BDE 47)
2,2',4,5' -tetrabromodiphenylether (BDE 49)
2,3',4-tribromodiphenylether (BDE 25)
2,3',4,4',6-pentabromodiphenylether (BDE 119)
2,3',4,4' -tetrabromodiphenylether (BDE 66)
2,3,3',4,4',5,6-heptabromodiphenylether (BDE 190)
2,3,3',4,4' -pentabromodiphenylether (BDE 105)
2,3,4,4',5,6-hexabromodiphenylether (BDE 166)
2,3,4,5,6-pentabromodiphenylether (BDE 116)
2,4'-dibromodiphenylether (BDE 8)
2,4-dibromodiphenylether (BDE 7)
2,4,4',6-tetrabromodiphenylether (BDE 75)
2,4,4' -tribromodiphenylether (BDE 28)
2,4,6-tribromodiphenylether (BDE 30)
2,6-dibromodiphenylether (BDE 10)
3,3',4-tribromodiphenylether (BDE 35)
3,3',4,4',5-pentabromodiphenylether (BDE 126)
3,3',4,4' -tetrabromodiphenylether (BDE 77)
3,3'-dibromodiphenylether (BDE 11)
3,4'-dibromodiphenylether (BDE 13)
3,4-dibromodiphenylether (BDE 12)
3,4,4'-tribromodiphenylether (BDE 37)
4,4'-dibromodiphenylether (BDE 15)
Decabromodiphenylethane (DBDPE)
Hexabromobenzene (HBB)
Pentabromoethylbenzene (PBEB)

Tissue (Organic)
Polychlorinated Biphenyls (PCB) - Tissue (022)
MLA-010; EPA 1668A
HIGH RESOLUTION GC/MS
PCB 001
PCB 002
PCB 003
PCB 004
PCB 005
PCB 006
PCB 007
PCB 008
PCB 009

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PCB 010
PCB 011
PCB 012
PCB 013
PCB 014
PCB 015
PCB 016
PCB 017
PCB 018
PCB 019
PCB 020
PCB 021
PCB 022
PCB 023
PCB 024
PCB 025
PCB 026
PCB 027
PCB 028
PCB 029
PCB 030
PCB 031
PCB 032
PCB 033
PCB 034
PCB 035
PCB 036
PCB 037
PCB 038
PCB 039
PCB 040
PCB 041
PCB 042
PCB 043
PCB 044
PCB 045
PCB 046
PCB 047
PCB 048
PCB 049
PCB 050
PCB 051
PCB 052
PCB 053
PCB 054
PCB 055
PCB 056
PCB 057
PCB 058
PCB 059
PCB 060
PCB 061
PCB 062

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PCB 063
PCB 064
PCB 065
PCB 066
PCB 067
PCB 068
PCB 069
PCB 070
PCB 071
PCB 072
PCB 073
PCB 074
PCB 075
PCB 076
PCB 077
PCB 078
PCB 079
PCB 080
PCB 081
PCB 082
PCB 083
PCB 084
PCB 085
PCB 086
PCB 087
PCB 088
PCB 089
PCB 090
PCB 091
PCB 092
PCB 093
PCB 094
PCB 095
PCB 096
PCB 097
PCB 098
PCB 099
PCB 100
PCB 101
PCB 102
PCB 103
PCB 104
PCB 105
PCB 106
PCB 107
PCB 108
PCB 109
PCB 110
PCB 111
PCB 112
PCB 113
PCB 114
PCB 115

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PCB 116
PCB 117
PCB 118
PCB 119
PCB 120
PCB 121
PCB 122
PCB 123
PCB 124
PCB 125
PCB 126
PCB 127
PCB 128
PCB 129
PCB 130
PCB 131
PCB 132
PCB 133
PCB 134
PCB 135
PCB 136
PCB 137
PCB 138
PCB 139
PCB 140
PCB 141
PCB 142
PCB 143
PCB 144
PCB 145
PCB 146
PCB 147
PCB 148
PCB 149
PCB 150
PCB 151
PCB 152
PCB 153
PCB 154
PCB 155
PCB 156
PCB 157
PCB 158
PCB 159
PCB 160
PCB 161
PCB 162
PCB 163
PCB 164
PCB 165
PCB 166
PCB 167
PCB 168

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PCB 169
PCB 170
PCB 171
PCB 172
PCB 173
PCB 174
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 180
PCB 181
PCB 182
PCB 183
PCB 184
PCB 185
PCB 186
PCB 187
PCB 188
PCB 189
PCB 190
PCB 191
PCB 192
PCB 193
PCB 194
PCB 195
PCB 196
PCB 197
PCB 198
PCB 199
PCB 200
PCB 201
PCB 202
PCB 203
PCB 204
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Monochloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs

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Tissue (Organic)

Polychlorinated Biphenyls (PCB)/Organochlorine Pesticides (OCP) - Tissue (007)

MLA-007; modified from EPA 8270C

GC/MS - EXTRACTION

Aldrin
alpha-hexachlorocyclohexane (a-HCH, a-BHC)
Aroclor 1221
Aroclor 1232
Aroclor 1242/1016
Aroclor 1248
Aroclor 1254
Aroclor 1260
Aroclor 1268
beta - Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Chlordane (alpha-chlordane)
cis-Nonachlor
Heptachlor
Hexachlorobenzene
Lindane (gamma-BHC)
Mirex
o,p'-DDD
o,p'-DDE
o,p'-DDT
Oxychlordane
p,p'-DDD
p,p'-DDE
p,p'-DDT
PCB 101/90/89
PCB 105/127
PCB 107/109
PCB 110
PCB 114
PCB 118/106
PCB 126
PCB 128
PCB 129
PCB 130
PCB 131/142
PCB 134/143
PCB 136
PCB 137
PCB 138/163/164
PCB 141
PCB 144/135
PCB 146
PCB 149/139
PCB 15
PCB 151
PCB 153
PCB 156
PCB 157
PCB 158/160
PCB 16/32

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PCB 169
PCB 17
PCB 170/190
PCB 171
PCB 172/192
PCB 174/181
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 18
PCB 180
PCB 183
PCB 185
PCB 187/182
PCB 189
PCB 19
PCB 191
PCB 193
PCB 194
PCB 195
PCB 196/203
PCB 197
PCB 198
PCB 199
PCB 201
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
PCB 22
PCB 24/27
PCB 25
PCB 26
PCB 28
PCB 31
PCB 33/20/21
PCB 40
PCB 41/71/64/68
PCB 42/59
PCB 44
PCB 45
PCB 46
PCB 47/48/75
PCB 49/43
PCB 52/73
PCB 56/60
PCB 66/80
PCB 70/76
PCB 74/61
PCB 77

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PCB 8/5
PCB 83/108
PCB 84
PCB 85/120
PCB 87/115/116
PCB 91
PCB 95/93
PCB 97/86
PCB 99
Total Decachloro-PCBs
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total PCBs
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs
Toxaphene
trans-Chlordane (gamma-chlordane)
trans-Nonachlor

Tissue (Organic)
Polychlorinated Dioxins/Furans - Tissue (020)
MLA-017; EPA 1613B
HIGH RESOLUTION GC/MS - EXTRACTION
1,2,3,4,6,7,8-Heptachlorodioxin
1,2,3,4,6,7,8-Heptachlorofuran
1,2,3,4,7,8-Hexachlorodioxin
1,2,3,4,7,8-Hexachlorofuran
1,2,3,4,7,8,9-Heptachlorofuran
1,2,3,6,7,8-Hexachlorodioxin
1,2,3,6,7,8-Hexachlorofuran
1,2,3,7,8-Pentachlorodioxin
1,2,3,7,8-Pentachlorofuran
1,2,3,7,8,9-Hexachlorodioxin
1,2,3,7,8,9-Hexachlorofuran
2,3,4,6,7,8-Hexachlorofuran
2,3,4,7,8-Pentachlorofuran
2,3,7,8-Tetrachlorodioxin
2,3,7,8-Tetrachlorofuran
Octachlorodioxin
Octachlorofuran

Tissue (Organic)
Polycyclic Aromatic Hydrocarbons (PAH) - Tissue (014)
MLA-021; modified from EPA 8270C
GC/MS - DIGESTION
Acenaphthene
Acenaphthylene
Anthracene
Benz (a) anthracene
Benz (e) pyrene
Benzo (a) pyrene

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Benzo (b) fluoranthene
Benzo (g,h,i) perylene
Benzo (k) fluoranthene
Chrysene
Dibenz (a,h) anthracene
Fluoranthene
Fluorene
Indeno (1,2,3-cd) pyrene
Naphthalene
Perylene
Phenanthrene
Pyrene

Tissue (Organic)
Sugars, Bile Acids, Fatty Acids - Tissue (058)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
11, 14, 17-eicosatrienoic acid (eicosatrienoic acid)
11, 14-eicosadienoic acid
C22:5 ISOMER 1 (tentatively all-cis-4, 8, 12, 15, 19-
C22:5 ISOMER 2 (all-cis-7,10,13,16,19-docosapentaenoic
C22:5 ISOMER 3 (tentatively all-cis-4, 7, 10, 13, 16-
chenodeoxycholic acid
cholic acid
decanoic acid (capric acid)
deoxycholic acid
docosahexaenoic acid (DHA)
docosatetraenoic acid (adrenic acid)
eicosapentaenoic acid (EPA)
Eicosatetraenoic acid (arachidonic acid)
eicosatrienoic acid (dihomo-linolenic acid)
glycochenodeoxycholic acid
glycocholic acid
glycodeoxycholic acid
hexadecanoic acid (palmitic acid)
hexadecenoic acid (palmitoleic acid)
Hexose (sum isomers)
lithocholic acid
octadecadienoic acid (linoleic acid)
octadecanoic acid (stearic acid)
octadecatrienoic acid (
taurochenodeoxycholic acid
taurocholic acid
taurodeoxycholic acid
taurolithocholic acid
tauroursodexoycholic acid
tetradecanoic acid (myristic acid)
ursodexoycholic acid

Urine (Organic)
Acylcarnitines, Glycerophospho-lipids, Sphingolipids - Urine (066)
MLM-001; IN-HOUSE
FLOW INJECTION - MS/MS
Acetylcarnitine
Butenylcarnitine

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Butyrylcarnitine
Carnitine
Decadienylcarnitine
Decanoylcarnitine
Decenoylcarnitine
Dodecanedioylcarnitine
Dodecanoylcarnitine
Dodecenoylcarnitine
Glutaconylcarnitine
Glutarylcarnitine (Hydroxyhexanoylcarnitine)
Hexadecadienylcarnitine
Hexadecanoylcarnitine
Hexadecenoylcarnitine
Hexanoylcarnitine (Fumarylcarnitine)
Hexenoylcarnitine
Hydroxyhexadecadienylcarnitine
Hydroxyhexadecanoylcarnitine
Hydroxyhexadecenoylcarnitine
Hydroxylbutyrylcarnitine
Hydroxyoctadecenoylcarnitine
Hydroxypropionylcarnitine
Hydroxysphingomyeline C14:1
Hydroxysphingomyeline C16:1
Hydroxysphingomyeline C22:1
Hydroxysphingomyeline C22:2
Hydroxysphingomyeline C24:1
Hydroxytetradecadienylcarnitine
Hydroxytetradecenoylcarnitine
Hydroxyvalerylcarnitine (Methylmalonylcarnitine)
lysoPhosphatidylcholine acyl C14:0
lysoPhosphatidylcholine acyl C16:0
lysoPhosphatidylcholine acyl C16:1
lysoPhosphatidylcholine acyl C17:0
lysoPhosphatidylcholine acyl C18:0
lysoPhosphatidylcholine acyl C18:1
lysoPhosphatidylcholine acyl C18:2
lysoPhosphatidylcholine acyl C20:3
lysoPhosphatidylcholine acyl C20:4
lysoPhosphatidylcholine acyl C24:0
lysoPhosphatidylcholine acyl C26:1
lysoPhosphatidylcholine acyl C28:0
lysoPhosphatidylcholine acyl C28:1
Methylglutarylcarnitine
Nonaylcarnitine
Octadecadienylcarnitine
Octadecanoylcarnitine
Octadecenoylcarnitine
Octanoylcarnitine
Phosphatidylcholine acyl-alkyl C30:0
Phosphatidylcholine acyl-alkyl C30:1
Phosphatidylcholine acyl-alkyl C30:2
Phosphatidylcholine acyl-alkyl C32:1
Phosphatidylcholine acyl-alkyl C32:2

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Phosphatidylcholine acyl-alkyl C34:0
Phosphatidylcholine acyl-alkyl C34:1
Phosphatidylcholine acyl-alkyl C34:2
Phosphatidylcholine acyl-alkyl C34:3
Phosphatidylcholine acyl-alkyl C36:0
Phosphatidylcholine acyl-alkyl C36:1
Phosphatidylcholine acyl-alkyl C36:2
Phosphatidylcholine acyl-alkyl C36:3
Phosphatidylcholine acyl-alkyl C36:4
Phosphatidylcholine acyl-alkyl C36:5
Phosphatidylcholine acyl-alkyl C38:0
Phosphatidylcholine acyl-alkyl C38:1
Phosphatidylcholine acyl-alkyl C38:2
Phosphatidylcholine acyl-alkyl C38:3
Phosphatidylcholine acyl-alkyl C38:5
Phosphatidylcholine acyl-alkyl C38:6
Phosphatidylcholine acyl-alkyl C40:1
Phosphatidylcholine acyl-alkyl C40:2
Phosphatidylcholine acyl-alkyl C40:3
Phosphatidylcholine acyl-alkyl C40:4
Phosphatidylcholine acyl-alkyl C40:5
Phosphatidylcholine acyl-alkyl C40:6
Phosphatidylcholine acyl-alkyl C42:0
Phosphatidylcholine acyl-alkyl C42:1
Phosphatidylcholine acyl-alkyl C42:2
Phosphatidylcholine acyl-alkyl C42:3
Phosphatidylcholine acyl-alkyl C42:4
Phosphatidylcholine acyl-alkyl C42:5
Phosphatidylcholine acyl-alkyl C44:3
Phosphatidylcholine acyl-alkyl C44:4
Phosphatidylcholine acyl-alkyl C44:5
Phosphatidylcholine acyl-alkyl C44:6
Phosphatidylcholine diacyl C24:0
Phosphatidylcholine diacyl C26:0
Phosphatidylcholine diacyl C28:1
Phosphatidylcholine diacyl C30:0
Phosphatidylcholine diacyl C30:2
Phosphatidylcholine diacyl C32:0
Phosphatidylcholine diacyl C32:1
Phosphatidylcholine diacyl C32:2
Phosphatidylcholine diacyl C32:3
Phosphatidylcholine diacyl C34:1
Phosphatidylcholine diacyl C34:2
Phosphatidylcholine diacyl C34:3
Phosphatidylcholine diacyl C34:4
Phosphatidylcholine diacyl C36:0
Phosphatidylcholine diacyl C36:1
Phosphatidylcholine diacyl C36:2
Phosphatidylcholine diacyl C36:3
Phosphatidylcholine diacyl C36:4
Phosphatidylcholine diacyl C36:5
Phosphatidylcholine diacyl C36:6
Phosphatidylcholine diacyl C38:0

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Phosphatidylcholine diacyl C38:1
Phosphatidylcholine diacyl C38:3
Phosphatidylcholine diacyl C38:4
Phosphatidylcholine diacyl C38:5
Phosphatidylcholine diacyl C38:6
Phosphatidylcholine diacyl C40:1
Phosphatidylcholine diacyl C40:2
Phosphatidylcholine diacyl C40:3
Phosphatidylcholine diacyl C40:4
Phosphatidylcholine diacyl C40:5
Phosphatidylcholine diacyl C40:6
Phosphatidylcholine diacyl C42:0
Phosphatidylcholine diacyl C42:1
Phosphatidylcholine diacyl C42:2
Phosphatidylcholine diacyl C42:4
Phosphatidylcholine diacyl C42:5
Phosphatidylcholine diacyl C42:6
Pimelylcarnitine
Propenoylcarnitine
Propionylcarnitine
Sphingomyeline C16:0
Sphingomyeline C16:1
Sphingomyeline C18:0
Sphingomyeline C18:1
Sphingomyeline C20:2
Sphingomyeline C22:3
Sphingomyeline C24:0
Sphingomyeline C24:1
Sphingomyeline C26:0
Sphingomyeline C26:1
Tetradecadienylcarnitine
Tetradecanoylcarnitine
Tetradecenoylcarnitine
Tiglylcarnitine
Valerylcarnitine

Urine (Organic)

Amino Acids and Biogenic Amines - Urine (065)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
3-hydroxytyrosine
Acetylornithine
Alanine
alpha-Aminoadipic acid
Arginine
Asparagine
Aspartate
Asymmetric dimethylarginine
Carnosine
Citrulline
Creatinine
Dopamine
Glutamate
Glutamine

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Glycine
Histamine
Histidine
Hydroxyproline
Isoleucine
Kynurenine
Leucine
Lysine
Methionine
Methioninesulfoxide
Nitrotyrosine
Ornithine
Phenylalanine
Phenylethylamine
Proline
Putrescine
Sarcosine
Serine
Serotonin
Spermidine
Spermine
Symmetric dimethylarginine
Taurine
Threonine
Total dimethylarginine
Tryptophan
Tyrosine
Valine

Urine (Organic)
BPA and MPE - Urine (053)
MLA-059; IN-HOUSE
LC-MS/MS
4,4'-dihydroxy-2,2-diphenylpropane (Bisphenol A) (BPA)
Mono-(2-ethyl-5-hydroxyhexyl) phthalate (MEHHP)
Mono-(2-ethyl-5-oxohexyl) phthalate (MEOHP)
Mono-(3-carboxypropyl) phthalate (MCPP)
Mono-2-ethylhexyl phthalate (MEHP)
Mono-benzyl phthalate (MBzP)
Mono-butyl phthalate (MBP) (n + iso)
Mono-cyclohexyl phthalate (MCHP)
Mono-ethyl phthalate (MEP)
Mono-iso-nonyl phthalate (MiNP)
Mono-methyl phthalate (MMP)

Urine (Organic)
Sugars, Bile Acids, Fatty Acids - Urine (064)
MLM-001; IN-HOUSE
LC-ESI-MS/MS
chenodeoxycholic acid
cholic acid
deoxycholic acid
glycochenodeoxycholic acid
glycocholic acid
glycodeoxycholic acid

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Hexose (sum isomers)
lithocholic acid
taurochenodeoxycholic acid
taurocholic acid
taurodeoxycholic acid
taurolithocholic acid
tauroursodexoycholic acid
ursodexoycholic acid

Water (Organic)
Dioxins and Furans (PCDD/PCDF) - Water (018)
MLA-017; EPA 1613B and EPS1/RM/19
HIGH RESOLUTION GC/MS-EXTRACTION
1,2,3,4,6,7,8-Heptachlorodioxin
1,2,3,4,6,7,8-Heptachlorofuran
1,2,3,4,7,8-Hexachlorodioxin
1,2,3,4,7,8-Hexachlorofuran
1,2,3,4,7,8,9-Heptachlorofuran
1,2,3,6,7,8-Hexachlorodioxin
1,2,3,6,7,8-Hexachlorofuran
1,2,3,7,8-Pentachlorodioxin
1,2,3,7,8-Pentachlorofuran
1,2,3,7,8,9-Hexachlorodioxin
1,2,3,7,8,9-Hexachlorofuran
2,3,4,6,7,8-Hexachlorofuran
2,3,4,7,8-Pentachlorofuran
2,3,7,8-Tetrachlorodioxin
2,3,7,8-Tetrachlorofuran
Octachlorodioxin
Octachlorofuran

Water (Organic)
Organochlorine Pesticides (OC) - Water (002)
MLA-007; modified from EPA 608
GC/ECD - EXTRACTION
delta-Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulfan I
Endosulfan II
Endosulphan Sulphate
Endrin
Endrin Aldehyde
Endrin Ketone
Heptachlor Epoxide
p,p' Methoxychlor

Water (Organic)
Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples - Water (067)
MLA-110; IN-HOUSE
LC-MS/MS
4:2 fluorotelomersulfonate (4:2 FTS)
6:2 fluorotelomersulfonate (6:2 FTS)
8:2 fluorotelomersulfonate (8:2 FTS)
N-Ethylperfluorooctanesulfonamide (N-EtFOSA)
N-Ethylperfluorooctanesulfonamidoethanol (N-EtFOSE)
N-MEthylperfluorooctanesulfonamidoacetic acid (N-

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N-Methylperfluorooctanesulfonamidoacetic acid (N-
N-Methylperfluorooctanesulfonamide (N-MeFOSA)
N-Methylperfluorooctanesulfonamidoethanol (N-MeFOSE)
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate (PFBA)
Perfluorodecanesulfonate (PFDS)
Perfluorodecanoate (PFDA)
Perfluorododecanesulfonate (PFDoS)
Perfluorododecanoate (PFDoA)
Perfluoroheptanesulfonate (PFHpS)
Perfluoroheptanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanesulfonate (PFNS)
Perfluorononanoate (PFNA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanesulfonamide (PFOSA), a.k.a FOSA
Perfluorooctanoate (PFOA)
Perfluoropentanesulfonate (PFPeS)
Perfluoropentanoate (PFPeA)
Perfluorotetradecanoate (PFTeDA)
Perfluorotridecanoate (PFTrDA)
Perfluoroundecanoate (PFUnA)

Water (Organic)
Perfluorinated Organics - Water (032)
MLA-060; IN-HOUSE
LC-MS/MS - EXTRACTION
Perfluorobutanesulfonate (PFBS)
Perfluorobutanoate PFBA
Perfluorodecanoate (PFDA)
Perfluorododecanoate (PFDoA)
Perfluorohepanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)
Perfluorononanoate (PFNA)
Perfluorooctane sulfonamide (PFOSA)
Perfluorooctanesulfonate (PFOS)
Perfluorooctanoate (PFOA)
Perfluoropentanoate (PFPeA)
Perfluoroundecanoate (PFUnA)

Water (Organic)
Pesticides - Water (043)
MLA-028; IN-HOUSE
HI RESOLUTION GC/MS
A -BHC
a - Chlordane
Aldrin
beta-Hexachlorocyclohexane (b-HCH, b-BHC)
cis-Nonachlor
delta- Hexachlorocyclohexane (d-HCH, d-BHC)
Dieldrin
Endosulfan I
Endosulfan II

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Endosulphan Sulphate
Endrin
Endrin Aldehyde
Endrin Ketone
g - Chlordane
Heptachlor
Heptachlor Epoxide
Hexachlorobenzene
Lindane (gamma-BHC)
Mirex
o,p' - DDT
o,p'-DDD
o,p'-DDE
Oxychlordane
p,p' - DDT
p,p' Methoxychlor
p,p'-DDD
p,p'-DDE
trans-Nonachlor

Water (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Water (035)
MLA-075; EPA 1694

LC-MS/MS - ACID EXTRACTION, FORMIC ACID
10-hydroxy-amitriptyline
1,7-Dimethylxanthine
Acetaminophen
Alprazolam
Amitriptyline
Amlodipine
Azithromycin
Benzoylecgonine
Benztropine
Betamethasone
Caffeine
Carbadox
Carbamazepine
Cefotaxime
Ciprofloxacin
Clarithromycin
Clinafloxacin
Cloxacillin
Cocaine
DEET (N,N-diethyl-m-toluamide)
Dehydronifedipine
Desmethyldiltiazem
Diazepam
Digoxigenin
Digoxin
Diltiazem
Diphenhydramine
Enrofloxacin
Erythromycin
Flumequine

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Fluocinonide
Fluoxetine
Fluticasone propionate
Hydrocortisone
Lincomycin
Lomefloxacin
Meprobamate
Methylprednisolone
Metoprolol
Miconazole
Norfloxacin
Norfluoxetine
Norgestimate
Norverapamil
Ofloxacin
Ormetoprim
Oxacillin
Oxolinic acid
Paroxetine
Penicillin G
Penicillin V
Prednisolone
Prednisone
Promethazine
Propoxyphene
Propranolol
Roxithromycin
Sarafloxacin
Sertraline
Simvastatin
Sulfachloropyridazine
Sulfadiazine
Sulfadimethoxine
Sulfamerazine
Sulfamethazine
Sulfamethizole
Sulfamethoxazole
Sulfanilamide
Sulfathiazole
Theophylline
Thiabendazole
Trenbolone
Trenbolone acetate
Trimethoprim
Tylosin
Valsartan
Verapamil
Virginiamycin

Water (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Water (036)
MLA-075; EPA 1694
LC-MS/MS - ACID EXTRACTION, ACN
4-Epianhydrochlortetracycline (EACTC)

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4-Epianhydrotetracycline (EATC)
4-Epichlortetracycline (ECTC)
4-Epioxytetracycline (EOTC)
4-Epitetracycline (ETC)
Anhydrochlortetracycline (ACTC)
Anhydrotetracycline (ATC)
Chlortetracycline (CTC)
Demeclocycline
Doxycycline
Isochlortetracycline (ICTC)
Minocycline
Oxytetracycline (OTC)
Tetracycline (TC)

Water (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Water (037)
MLA-075; EPA 1694
LC-MS/MS - ACID EXTRACTION, AMMONIUM ACETATE
2-hydroxy-ibuprofen
Bisphenol A
Furosemide
Gemfibrozil
Glipizide
Glyburide
Hydrochlorothiazide
Ibuprofen
Naproxen
Triclocarban
Triclosan
Warfarin

Water (Organic)
Pharmaceuticals and Personal Care Products (PPCP) - Water (038)
MLA-075; EPA 1694
LC-MS/MS - BASE EXTRACTION
Albuterol
Amphetamine
Atenolol
Atorvastatin
Cimetidine
Clonidine
Codeine
Cotinine
Enalapril
Hydrocodone
Metformin
Oxycodone
Ranitidine
Triamterene

Water (Organic)
Polybrominated Diphenylethers (PBDE) - Water (044)
MLA-033; EPA 1614
HI RESOLUTION GC/MS
1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE)
2',3,4-tribromodiphenylether (BDE 33)

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2,2',3,3',4,4',5,5',6-nonabromodiphenylether (BDE 206)
2,2',3,3',4,4',5,5',6,6' -decabromodiphenylether (BDE 209)
2,2',3,3',4,5,5',6,6' -nonabromodiphenylether (BDE 208)
2,2 3,3,4,4,5,6,6 -nonabromodiphenylether (BDE 207)
2,2',3,4,4',5',6-heptabromodiphenylether (BDE 183)
2,2',3,4,4',5,6-heptabromodiphenylether (BDE 181)
2,2',3,4,4',6' -hexabromodiphenylether (BDE 140)
2,2',3,4,4' -pentabromodiphenylether (BDE 85)
2,2',4-tribromodiphenylether (BDE 17)
2,2',4,4',5',6-hexabromodiphenylether (BDE 154)
2,2',4,4',5-pentabromodiphenylether (BDE 99)
2,2',4,4',5,5' -hexabromodiphenylether (BDE 153)
2,2',4,4',6-pentabromodiphenylether (BDE 100)
2,2',4,4',6,6' -hexabromodiphenylether (BDE 155)
2,2',4,4' -tetrabromodiphenylether (BDE 47)
2,2',4,5' -tetrabromodiphenylether (BDE 49)
2,3',4-tribromodiphenylether (BDE 25)
2,3',4,4',6-pentabromodiphenylether (BDE 119)
2,3',4,4' -tetrabromodiphenylether (BDE 66)
2,3,3',4,4',5,6-heptabromodiphenylether (BDE 190)
2,3,3',4,4' -pentabromodiphenylether (BDE 105)
2,3,4,4',5,6-hexabromodiphenylether (BDE 166)
2,3,4,5,6-pentabromodiphenylether (BDE 116)
2,4'-dibromodiphenylether (BDE 8)
2,4-dibromodiphenylether (BDE 7)
2,4,4',6-tetrabromodiphenylether (BDE 75)
2,4,4' -tribromodiphenylether (BDE 28)
2,4,6-tribromodiphenylether (BDE 30)
2,6-dibromodiphenylether (BDE 10)
3,3',4-tribromodiphenylether (BDE 35)
3,3',4,4',5-pentabromodiphenylether (BDE 126)
3,3',4,4' -tetrabromodiphenylether (BDE 77)
3,3'-dibromodiphenylether (BDE 11)
3,4'-dibromodiphenylether (BDE 13)
3,4-dibromodiphenylether (BDE 12)
3,4,4'-tribromodiphenylether (BDE 37)
4,4'-dibromodiphenylether (BDE 15)
Decabromodiphenylethane (DBDPE)
Hexabromobenzene (HBB)
Pentabromoethylbenzene (PBEB)

Water (Organic)
Polychlorinated Biphenyls (PCB) - Water (024)
MLA-010; EPA 1668A
HIGH RESOLUTION GC/MS
PCB 001
PCB 002
PCB 003
PCB 004
PCB 005
PCB 006
PCB 007
PCB 008
PCB 009

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PCB 010
PCB 011
PCB 012
PCB 013
PCB 014
PCB 015
PCB 016
PCB 017
PCB 018
PCB 019
PCB 020
PCB 021
PCB 022
PCB 023
PCB 024
PCB 025
PCB 026
PCB 027
PCB 028
PCB 029
PCB 030
PCB 031
PCB 032
PCB 033
PCB 034
PCB 035
PCB 036
PCB 037
PCB 038
PCB 039
PCB 040
PCB 041
PCB 042
PCB 043
PCB 044
PCB 045
PCB 046
PCB 047
PCB 048
PCB 049
PCB 050
PCB 051
PCB 052
PCB 053
PCB 054
PCB 055
PCB 056
PCB 057
PCB 058
PCB 059
PCB 060
PCB 061
PCB 062

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PCB 063
PCB 064
PCB 065
PCB 066
PCB 067
PCB 068
PCB 069
PCB 070
PCB 071
PCB 072
PCB 073
PCB 074
PCB 075
PCB 076
PCB 077
PCB 078
PCB 079
PCB 080
PCB 081
PCB 082
PCB 083
PCB 084
PCB 085
PCB 086
PCB 087
PCB 088
PCB 089
PCB 090
PCB 091
PCB 092
PCB 093
PCB 094
PCB 095
PCB 096
PCB 097
PCB 098
PCB 099
PCB 100
PCB 101
PCB 102
PCB 103
PCB 104
PCB 105
PCB 106
PCB 107
PCB 108
PCB 109
PCB 110
PCB 111
PCB 112
PCB 113
PCB 114
PCB 115

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PCB 116
PCB 117
PCB 118
PCB 119
PCB 120
PCB 121
PCB 122
PCB 123
PCB 124
PCB 125
PCB 126
PCB 127
PCB 128
PCB 129
PCB 130
PCB 131
PCB 132
PCB 133
PCB 134
PCB 135
PCB 136
PCB 137
PCB 138
PCB 139
PCB 140
PCB 141
PCB 142
PCB 143
PCB 144
PCB 145
PCB 146
PCB 147
PCB 148
PCB 149
PCB 150
PCB 151
PCB 152
PCB 153
PCB 154
PCB 155
PCB 156
PCB 157
PCB 158
PCB 159
PCB 160
PCB 161
PCB 162
PCB 163
PCB 164
PCB 165
PCB 166
PCB 167
PCB 168

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PCB 169
PCB 170
PCB 171
PCB 172
PCB 173
PCB 174
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 180
PCB 181
PCB 182
PCB 183
PCB 184
PCB 185
PCB 186
PCB 187
PCB 188
PCB 189
PCB 190
PCB 191
PCB 192
PCB 193
PCB 194
PCB 195
PCB 196
PCB 197
PCB 198
PCB 199
PCB 200
PCB 201
PCB 202
PCB 203
PCB 204
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
Total dichloro-PCBs
Total heptachloro-PCBs
Total hexachloro-PCBs
Total monochloro-PCBs
Total nonachloro-PCBs
Total octachloro-PCBs
Total pentachloro-PCBs
Total tetrachloro-PCBs
Total trichloro-PCBs

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Water (Organic)

Polychlorinated Biphenyls (PCB)/Organochlorine Pesticides (OCP) - Water (001)

MLA-007; modified from EPA 625

GC/MS - EXTRACTION

A -BHC
a - Chlordane
Aldrin
Aroclor 1221
Aroclor 1232
Aroclor 1242/1016
Aroclor 1248
Aroclor 1254
Aroclor 1260
Aroclor 1268
beta-hexachlorocyclohexane (b-HCH, b-BHC)
cis-Nonachlor
Heptachlor
Hexachlorobenzene
Lindane (gamma-BHC)
Mirex
o,p' - DDT
o,p'-DDD
o,p'-DDE
Oxychlordane
p,p' - DDT
p,p'-DDD
p,p'-DDE
PCB 101/90/89
PCB 105/127
PCB 107/109
PCB 110
PCB 114
PCB 118/106
PCB 126
PCB 128
PCB 129
PCB 130
PCB 131/142
PCB 134/143
PCB 136
PCB 137
PCB 138/163/164
PCB 141
PCB 144/135
PCB 146
PCB 149/139
PCB 15
PCB 151
PCB 153
PCB 156
PCB 157
PCB 158/160
PCB 16/32

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PCB 169
PCB 17
PCB 170/190
PCB 171
PCB 172/192
PCB 174/181
PCB 175
PCB 176
PCB 177
PCB 178
PCB 179
PCB 18
PCB 180
PCB 183
PCB 185
PCB 187/182
PCB 189
PCB 19
PCB 191
PCB 193
PCB 194
PCB 195
PCB 196/203
PCB 197
PCB 198
PCB 199
PCB 201
PCB 205
PCB 206
PCB 207
PCB 208
PCB 209
PCB 22
PCB 24/27
PCB 25
PCB 26
PCB 28
PCB 31
PCB 33/20/21
PCB 40
PCB 41/71/64/68
PCB 42/59
PCB 44
PCB 45
PCB 46
PCB 47/48/75
PCB 49/43
PCB 52/73
PCB 56/60
PCB 66/80
PCB 70/76
PCB 74/61
PCB 77

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PCB 8/5
PCB 83/108
PCB 84
PCB 85/120
PCB 87/115/116
PCB 91
PCB 95/93
PCB 97/86
PCB 99
Total Decachloro-PCBs
Total Dichloro-PCBs
Total Heptachloro-PCBs
Total Hexachloro-PCBs
Total Nonachloro-PCBs
Total Octachloro-PCBs
Total PCB
Total Pentachloro-PCBs
Total Tetrachloro-PCBs
Total Trichloro-PCBs
Toxaphene
trans-Chlordane (gamma-chlordane)

Water (Organic)

Polycyclic Aromatic Hydrocarbons (PAH) - Water (057)
MLA-021; modified from EPA 1625B

GC/MS

1-Methylchrysene
1-Methylnaphthalene
1-Methylphenanthrene
1,2-Dimethylnaphthalene
1,2,6-Trimethylphenanthrene
1,4,6,7-Tetramethylnaphthalene
1,7-Dimethylfluorene
1,7-Dimethylphenanthrene
1,8-Dimethylphenanthrene
2-Methylanthracene
2-Methylfluorene
2-Methylnaphthalene
2-Methylphenanthrene
2/3-Methyldibenzothiophenes
2,3,5-Trimethylnaphthalene
2,3,6-Trimethylnaphthalene
2,4-Dimethyldibenzothiophene
2,6-Dimethylnaphthalene
2,6-Dimethylphenanthrene
3-Methylfluoranthene/Benzo(a)fluorene
3-Methylphenanthrene
3,6-Dimethylphenanthrene
5/6-Methylchrysenes
5,9-Dimethylchrysene
7-Methylbenzo(a)pyrene
9/4-Methylphenanthrenes
Acenaphthene
Acenaphthylene

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Anthracene
Benzo (a) anthracene
Benzo (a) pyrene
Benzo (b) fluoranthene
Benzo(e)pyrene
Benzo (g,h,i) perylene
Benzo(j/k)fluoranthenes
Benzo (k) fluoranthene
Biphenyl
C1-Acenaphthenes
C1-Benz(a)anthracenes/ chrysenes
C1-Benzofluoranthenes/Benzopyrenes
C1-Biphenyls
C1-Dibenzothiophene
C1-Fluoranthenes/Pyrenes
C1-Fluorenes
C1-Naphthalenes
C1-Phenanthrenes/Anthracenes
C2-Benz(a)anthracenes/Chrysenes
C2-Benzofluoranthenes/Benzopyrenes
C2-Biphenyls
C2-Dibenzothiophene
C2-Fluoranthenes/Pyrenes
C2-Fluorenes
C2-Naphthalenes
C2-Phenanthrenes/Anthracenes
C3-Benz(a)anthracenes/Chrysenes
C3-Dibenzothiophene
C3-Fluoranthenes/Pyrenes
C3-Fluorenes
C3-Naphthalenes
C3-Phenanthrenes/Anthracenes
C4-Benz(a)anthracenes/Chrysenes
C4-Dibenzothiophene
C4-Fluoranthenes/Pyrenes
C4-Naphthalenes
C4-Phenanthrenes/Anthracenes
Chrysene
Dibenzo (a,h) anthracene
Dibenzothiophene
Fluoranthene
Fluorene
Indeno (1,2,3 - cd) pyrene
Naphthalene
Perylene
Phenanthrene
Pyrene
Retene

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