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AXYS ANALYTICAL SERVICES LTD.

ANALYTICAL METHOD FOR THE DETERMINATION OF:
AROCLORS
TOTAL PCBs
CHLORINATED PESTICIDES
PCB CONGENERS
COPLANAR PCBs (NON-ORTHO-SUBSTITUTED, NOS)
TOXAPHENE
CHLOROBENZENES

FINAL

SCOPE

This document describes the analytical method for the quantitative determination of total PCBs (as Aroclor equivalents or as totals by PCB congener group), chlorobenzenes, chlorinated pesticides and individual PCB congeners in sediment, soil, sludge, ash, particulate filter, pulp, tissue (including blood and milk), water, effluent, XAD-2 columns (resin and filters), air samples and solvent extracts. Samples are spiked with isotopically labelled 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).

Data for solids are reported on a dry weight basis. Tissue data are reported on a wet weight basis. Water and effluent data are reported on a volume basis. Air sample and XAD-2 column data are reported on a per sample or volume basis.

This method can be adapted to the analysis of all target analytes by high-resolution gas chromatography with high-resolution mass spectrometric detection.

This method also allows for the optional analysis of toxaphene (as total toxaphene, total homologue groups, or as a suite of individual chlorobornanes), non-ortho substituted (NOS, coplanars) PCBs and chlorinated pesticides and PCBs by GC/ECD. These options are described in the appendices.

This method may be used for analysis of samples where USEPA Methods 608, 625, 1625, 8081, 8082, 8270, or Method SM18 6630B have been requested provided that the modifications described in this document are permitted by contract.

Approved by:_____ Date: _____
Dale Hoover, Quality Manager

Approved by:_____ Date:_____
Jill Hirschbold, Manager, Laboratory

Approved by :_____ Date:_____
Shea Hewage, Senior Manager, Production

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REFERENCES:

USEPA Method 608, Organochlorine Pesticides and PCB's.

USEPA Method 625, Acids and Base/Neutrals.

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

USEPA Method 8081, Organochlorine Pesticides by GC.

USEPA Method 8082, PCB's by GC.

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

SM18, Part 6630B, Liquid-Liquid Extraction Gas Chromatographic Method I

Smith, L.M. "Carbon Dispersion Glass Fibers as An Adsorbent for Contaminant Enrichment and Fractionation", Anal. Chem. (1981), **53**, p. 2154-56.

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.

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AMENDMENT PAGE

The following amendments have been made to this document. The signatories on the previous page have approved all amendments.

Date	Page	Amendment

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INTRODUCTION

1.0 ANALYTES OF INTEREST

A broad range of non-polar chlorinated organic compounds is extracted from sample matrices using the extraction procedures described in this document. The analysis code, as described below, indicates the group of target compounds analyzed.

Analysis Code	Target Group
A	Total PCBs (as Aroclor equivalents)
T	Total PCBs (as Congener groups)
P	Pesticides
C	PCB Congeners (POS, poly-ortho-substituted)
CB	Chlorobenzenes
Tx	Toxaphene
N	Coplanar PCBs (NOS, non-ortho-substituted)

This document describes the determination of the following compounds:

PCBs as Aroclor Equivalents

(Code A)

Aroclor 1221
Aroclor 1232
Aroclor 1242/1016
Aroclor 1248
Aroclor 1254
Aroclor 1260

PCBs as Congener Groups

(Code T)

Total Dichloro-PCBs
Total Trichloro-PCBs
Total Tetrachloro-PCBs
Total Pentachloro-PCBs
Total Hexachloro-PCBs
Total Heptachloro-PCBs
Total Octachloro-PCBs
Total Nonachlor-PCBs
Total Decachloro-PCBs

Total PCBs can be quantified as the sum of individual Aroclors, or as the total of PCB congener groups.

Chlorobenzenes (Code CB)

1,3-Dichlorobenzene
1,4- Dichlorobenzene
1,2- Dichlorobenzene
1,3,5-Trichlorobenzene
1,2,4- Trichlorobenzene
1,2,3- Trichlorobenzene
1,2,3,5/1,2,4,5-Tetrachlorobenzene
1,2,3,4-Tetrachlorobenzene
Pentachlorobenzene
Hexachlorobenzene

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Pesticides (Code P)

E1 Pesticides by GC/MS

Hexachlorobutadiene
Hexachlorobenzene
alpha-HCH
beta- BHC
gamma-HCH
delta-HCH
Heptachlor
Aldrin
Octachlorostyrene
Oxychlordane
Trans-Chlordane
Cis-Chlordane
o,p'-DDE
p,p'-DDE
trans-Nonachlor
cis-Nonachlor
o,p'-DDD
p,p'-DDD
o,p'-DDT (GC/MS only)
p,p'-DDT
Mirex
Photomirex (upon request)
Toxaphene (upon request as part of organochlorine scan)

E2 Pesticides by GC/ECD

Heptachlor Epoxide
alpha-Endosulphan
delta-HCH
Dieldrin
Endrin
Methoxychlor
Endosulphan Sulphate
Endrin Ketone
beta-Endosulphan
Endrin Aldehyde

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PCB Congeners by GC/MS (Code C)

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.

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2.0 OPTIONAL ANALYSES

The following optional analysis (indicated by analysis codes) may also be carried out and are described in the appendices at the end of this document.

2.1 Code Tx -Toxaphene by GC/MS-ECNI (Appendix A)

Total Toxaphene - (sum of 19 peaks)

Individual Components in Toxaphene (selected chlorobornanes, Parlar II Suite)

2,2,5-endo,6-exo,8,9,10-Heptachlorobornane
2-exo,3-endo,5-exo,6-endo,8,8,10,10-Octachlorobornane
Octachloro-derivative
2-exo,3-endo,5-exo,6-endo,8,8,9,10,10-Nonachlorobornane
Nonachloro-derivative
2,2,5-endo,6-exo,8,8,9,10,10-Nonachlorobornane
2-exo,3-exo,5-endo,6-exo,8,8,9,10,10-nonachlorobornane
2,2,3-exo,5-endo,6-exo,8,8,9,10,10-Decachlorobornane
2,2,5,5,6-exo,8,8,9,10,10-Decachlorobornane
2,2,3-exo,5,5,8,8,9,10,10-Decachlorobornane

2.2 Code N - Analysis of Coplanar PCBs by HRGC/HRMS (Appendix B)

PCB 81 (upon request)
PCB 77
PCB 126
PCB 169

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2.3 Code A,P,C,T - Analysis of PCBs/Pesticides by GC/ECD (Appendix C)

The GC/ECD analysis is suitable for the list of pesticides on page 5 and the following suite of PCB Congeners.

PCB 18
PCB 15/17
PCB 54
PCB 31
PCB 28
PCB 52
PCB 49
PCB 44
PCB 40/103
PCB 61/94/74
PCB 66/80/95
PCB 121
PCB 56/60
PCB 90/101
PCB 86/97
PCB 87
PCB 77/54/110
PCB 151
PCB 135/144
PCB 149
PCB 118
PCB 143
PCB 114
PCB 132/153
PCB 105
PCB 141/179
PCB 137
PCB 138/158
PCB 129/126
PCB 187/182/159
PCB 183
PCB 128
PCB 185
PCB 174/181
PCB 202/171/156
PCB 173/201
PCB 180
PCB 191
PCB 170
PCB 199
PCB 189
PCB 203/196
PCB 208/195
PCB 207
PCB 194
PCB 205
PCB 206

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3.0 Pollution Prevention and Waste Management

Axys Analytical Services Ltd. complies with all federal, provincial and municipal regulations governing waste management, including land disposal restrictions and sewage discharge regulations. 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 Axys' "Laboratory Safety Practices Guide and to the standard operating procedure SLA-077 "Sample Disposal" for procedures for disposing of laboratory wastes.

4.0 Safety

Refer to Axys "Laboratory Safety Practices Guide" for safety procedures.

5.0 Definitions

Refer to Axys Analytical Services QA/QC Policies and Procedures Manual for definitions of terms used in this document.

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ANALYSIS PROCEDURES

1.0 SAMPLE STORAGE

Sample storage requirements are summarized in the Sample Requirements Table. Samples other than XAD-2 columns and large tissue samples are stored in tightly sealed glass containers with foil or Teflon lined screw cap lids. Where adherence to USEPA Method holding times is required the storage times summarized in the EPA Methods Storage Requirements Table apply.

- Blood plasma and blood serum are stored frozen at -20°C.
- Milk samples are stored frozen at -20°C.
- Particulate filters are stored frozen, at -20°C.
- Ambient air samples, received as polyurethane foam plugs (PUF) and filters are stored frozen in screw top, airtight containers, at less than -20°C.
- Dried pulp samples are stored in a cupboard at room temperature.
- Wet pulp samples are stored at 4°C prior to drying. Excess wet pulp is stored frozen at -20°C.
- Soil, sediment, sludge, and ash samples are stored frozen, at -20°C.
- Stack gas sample train recovery containers and components are stored in sealed glass ampoules in the cooler at 4°C
- Tissue samples including fish and animal tissue, animal fat, and vegetation are stored frozen, at -20°C.
- Water, effluent and solvent extract samples are stored in the cooler, at 4°C.
- XAD-2 columns are stored in the refrigerator or cooler at 4°C.
- XAD-2 resin, removed from XAD-2 columns, is stored at 4°C.
- Particulate filters associated with XAD-2 columns are stored frozen at -20°C.
- Wound glass filter cartridges associated with XAD-2 columns are stored at 4°C.
- Microvialed sample extracts are to be stored in the freezer at –20°C.

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PCB/Pesticide Analysis Sample Storage Requirements

Matrix	Sample Size (per analysis)	Sample Container[1]	Storage Conditions[2]	Holding Time[3]	Extract Holding Time	Preservation
Air Sample	PUF & Filter	Glass	-20°C	2 months	2 months	none required
Ash	5 g dry	Glass	-20°C	2 months	2 months	none required
Blood	5-20 g	Glass	-20°C	2 months	2 months	none required
Milk	50 g	Glass	-20°C	2 months	2 months	none required
Particulate Filter Paper	whole filter	Glass or foil wrapped	-20°C	2 months	2 months	none required
Pulp (dry)	20 g dry	Glass or foil wrapped	ambient	2 months	2 months	none required
Pulp (wet)	N/A	Glass	4°C	2 months	2 months	none required
Sediment/Soil	10-15 g wet	Glass	-20°C	2 months	2 months	none required
Sludge	10 g wet	Glass	-20°C	2 months	2 months	none required
Solvent Extract	one	Glass	4°C	2 months	2 months	none required
Stack Gas	Containers A-F	Glass	4°C	30 days from sampling	2 months	none required
Tissue	5-10 g wet	Glass or foil wrapped	-20°C	2 months	2 months	none required
Water/Effluent	1 litre	Brown glass	4°C, dark	7 days	2 months	none required
XAD-2 Columns	one column	As is	4°C	2 months	2 months	none required
	resin	Glass	4°C	2 months	2 months	none required
	particulate filter(s)	Foil wrapped	-20°C	2 months	2 months	none required
	wound glass filter	Foil wrapped	4°C	2 months	2 months	none required

[1]All glass containers should be organically clean; i.e., baked or solvent-rinsed. All containers must be tightly sealed with screw cap lids (Teflon 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 at a nominal temperature of –20°C may be stored colder than this temperature but no more than 2°C warmer.

[3]There is no evidence to indicate that properly stored samples are not stable for longer periods of time. Client requests for specific holding times or other method-specific holding times are adhered to.

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EPA Method Sample Storage Requirements

Analysis	Matrix	Sample Size	Container[1]	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	4°C, dark	4°C, dark	(7 days)	40 days	Adjust to pH 5.9	Add $Na_2S_2O_3$
EPA 625									
Chlorinated Pesticides	Aqueous	1 L	Amber glass	4°C, dark	4°C, dark	(7 days)	40 days	Not required	Add 80mg/L $Na_2S_2O_3$
EPA 1625C									Add 80mg/L
PAHs	Aqueous	1 L	Amber glass	4°C, dark	4°C, dark	(7 days)	40 days	Not required	$Na_2S_2O_3$
Chlorinated Hydrocarbons	Solid	2 – 30 g	Glass	4°C, dark	4°C, dark	(14 days)	40 days	Not required	Not required
EPA 8081B									Add 80mg/L
Chlorinated Pesticides	Aqueous	1 L	Amber glass	4°C, dark	4°C, dark	(7 days)	40 days	Not required	$Na_2S_2O_3$
	Solid	2 – 30 g	Glass	4°C	4°C	(14 days)	40 days	Not required	Not required
EPA 8082A									Add 80mg/L
PCB's	Aqueous	1 L	Amber glass	4°C, dark	4°C, dark	(7 days)	40 days	Not required	$Na_2S_2O_3$
	Solid	2 – 30 g	Glass	4°C	4°C	(14 days)	40 days	Not required	Not required
EPA 8270D									Add 80mg/L
PAHs, PCBs	Aqueous	1 L	Amber glass	4°C, dark	4°C, dark	(7 days)	40 days	Not required	$Na_2S_2O_3$
Pesticides	Solid	2 – 30 g	Glass	-10°C, dark	-10°C, dark	(14 days)	40 days	Not required	Not required
Chlorinated Hydrocarbons									

[1] All glass containers should be organically clean; i.e., baked or solvent-rinsed. All containers must be tightly sealed with screw cap lids (Teflon 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 at a nominal temperature of –20°C may be stored colder than this temperature but no more than 2°C 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 Axys Analytical.

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2.0 SAMPLE PRETREATMENT

Refer to the following standard operating procedures:

AXYS ID	QPulse ID	Title
LAB-11	SLA-011	Compositing Samples
LAB-12	SLA-012	Dissection of Samples
LAB-13	SLA-013	Homogenization of Samples
LAB-14	SLA-014	Thawing of Samples Prior to Analysis
LAB-15	SLA-015	Moisture Determination
LAB-39	SLA-039	Air Drying of Pulp and Sludge

Blood Serum/Blood Plasma

Allow blood sample to thaw at room temperature prior to subsampling for analysis. Homogenize by vortex mixing.

Milk

Allow milk to thaw at room temperature prior to subsampling for analysis. Homogenize the sample by shaking well.

Particulate Filter Papers

Handle particulate filter papers while frozen. Break or cut the particulate filter into small pieces.

Ambient Air (PUF and Filter)

If received in the sampling apparatus, use solvent rinsed forceps to withdraw the plug and associated filter from the sampler.

Pulp

Air dry wet pulp samples according to standard operating procedures. Determine the moisture content on the dried pulp.

Sediment/Soils/Ash/Sludge/Gravel

Using standard operating procedures, allow the sample to thaw at room temperature prior to analysis.

Using standard operating procedures, homogenize the entire sample before subsampling for analysis.

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Determine moisture content on an accurately weighed subsample (1 g, depending upon sample available) according to standard operating procedures.

Tissue

Using standard operating procedures, allow the tissue sample to thaw at room temperature prior to analysis.

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 standard operating procedures.

If required, determine moisture content of an accurately weighed subsample of (1 g) according to standard operating procedures.

XAD-2 Columns and Filters

Refer to the following standard operating procedure:

AXYS ID	QPulse ID	Title
LAB-43	SLA-043	Handling and Preparation of XAD Resin Columns and Associated Filters, Section 1.

for details of removing the resin from the column.

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3.0 MATERIALS AND REAGENTS

Refer to the following standard operating procedures:

AXYS ID	QPulse ID	Title
LAB-1	SLA-001	Cleaning Laboratory Items
LAB-2	SLA-002	Glassware and Laboratory Equipment Proofs
LAB-09	SLA-009	Preparation of Standards
LAB-18	SLA-018	Solvent Rinsing of Glassware for Organic Analysis
LAB-19	SLA-019	Solvent Proofs
LAB-22	SLA-022	Use of Drying Ovens and Muffle Furnace
LAB-23	SLA-023	Use of Balances
LAB-35	SLA-035	Cleaning Glass Wool
LAB-41	SLA-041	Reagent Preparation
LAB-44	SLA-044	Activation of Copper Turnings
QAQC-9	SQA-009	Storage and Control of Standards
QAQC-3	SQA-003	Standard Solution Validation

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

The analysis of chlorinated pesticides, PCBs and chlorobenzenes by low resolution GC/MS and of the most 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 maintained by the Technical Specialist.

Surrogate Standard

Samples are spiked with a suite of isotopically labelled surrogate standards prior to extraction procedures. The surrogate standard solution is prepared to have the nominal concentrations of surrogates presented in Table 1. An aliquot of surrogate standard solution is added to each sample prior to extraction. Typically, 40 µL, equivalent to approximately 40 ng, is added to the sample. 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.

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Recovery Standard

Samples extracts are spiked with isotopically labelled recovery standards prior to instrumental analysis. The recovery standard solutions are prepared to have the nominal concentrations of recovery standards presented in Table 1. 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.

Calibration Solutions

A series of five calibration solutions containing native analytes, labelled surrogates standards, and labelled recovery standards are used to establish linearity of the analytical instrument. The calibration solutions are prepared to have the nominal concentrations presented in Tables 2 and 3. 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 solution is analyzed every 12 hours.

Two additional calibration GC/ECD solutions are prepared. One contains delta-HCH (80 ng/mL) and the recovery standard ^{13}C-PCB 153 (75 ng/mL). This solution is analyzed by GC/ECD, once every 12 hours, following the analysis of the mid-level calibration solution and is used to determine the RRF of delta-HCH. A second solution, containing endrin at a concentration of 250 ng/mL is prepared in hexane. This solution is analyzed once every 12 hours and is used to monitor the extent of endrin breakdown.

Authentic Spiking Standard

The authentic spiking solution is prepared to have the nominal concentrations presented in Table 1. 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, equivalent to 40 ng of each target analyte, is added to the matrix.

Retention Time Markers

The surrogate standard solution and calibration solutions contain a suite of isotopically labelled compounds that are used as retention time (RT) markers.

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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. 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.

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Table 1: Analysis of Chlorobenzenes, Pesticides and PCBs Nominal Concentrations of Standard Solutions (ng/mL)

Compound Name	Nominal Conc. in Authentic Std Solution
Mono-Cl-Benzene	40
1,3-DiCl-Benzene	40
1,4-DiCl-Benzene	40
1,2-DiCl-Benzene	40
1,3,5-TriCl-Benzene	40
1,2,4-TriCl-Benzene	40
1,2,3-TriCl-Benzene	40
1235/1245-TetraCl-Benzene	80
1,2,3,4-TetraCl-Benzene	40
PentaCl-Benzene	40
HexaCl-Benzene	40
alpha-HCH	80
beta-HCH	80
gamma-HCH	80
delta-HCH	80
Heptachlor	40
Aldrin	80
trans-Chlordane	80
cis-Chlordane	80
trans-Nonachlor	80
cis-Nonachlor	80
op'-DDE	40
pp'-DDE	40
op'-DDT	40
pp'-DDT	40
Octachlorostyrene	40
Oxychlordane	80
op'-DDD	40
pp'-DDD	40
Mirex	40
Dieldrin	40
alpha-Endosulphan	40
beta-Endosulphan	40
Endosulphan Sulphate	40
Endrin	40
Methoxychlor	40
Endrin Aldehyde	40
Endrin Ketone	40
Heptachlor Epoxide	40

FINAL

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Table 1 cont'd/....

Compound Name	Nominal Concentration in Authentic Std Solution	Compound Name	Nominal Concentration in Authentic Std Solution
PCB 1	40	PCB 114	40
PCB 3	40	PCB 118	40
PCB 4	40	PCB 123	40
PCB 8	36	PCB 126	40
PCB 15	40	PCB 138	36
PCB 18	36	PCB 149	36
PCB 19	40	PCB 151	36
PCB 28	36	PCB 153	36
PCB 31	36	PCB 155	40
PCB 34/23	80	PCB 156	40
PCB 37	40	PCB 157	40
PCB 40	36	PCB 167	40
PCB 44	36	PCB 169	40
PCB 49	36	PCB 170	40
PCB 52	36	PCB 180	40
PCB 54	40	PCB 183	36
PCB 56	36	PCB 187/182	80
PCB 66	36	PCB 188	40
PCB 77	40	PCB 189	40
PCB 81	40	PCB 194	36
PCB 87	36	PCB 196	36
PCB 95	36	PCB 202	40
PCB 99	36	PCB 205	40
PCB 101	36	PCB 208	40
PCB 104	40	PCB 206	40
PCB 110	36	PCB 209	40
PCB 105	40		

CONTROLLED

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Table 1 cont'd/....

Labelled Surrogates	Nominal Conc. in Surrogate Std Solution
$^{13}C_6$-Mono-Cl-Benzene	900
$^{13}C_6$-1,4-DiCl-Benzene	900
$^{13}C_6$-1,2,3-TriCl-Benzene	900
$^{13}C_6$-1,2,3,4-TetCl-Benzene	900
$^{13}C_6$-PentaCl-Benzene	900
$^{13}C_6$-HexaCl-Benzene	900
$^{13}C_6$-beta BHC	900
$^{13}C_6$-gamma BHC	900
$^{13}C_{10}$-Heptachlor	900
$^{13}C_{12}$-Aldrin	900
$^{13}C_{10}$-trans-Chlordane	900
$^{13}C_{10}$-trans-Nonachlor	900
$^{13}C_{10}$-Oxychlordane (RT marker)	900
$^{13}C_{12}$-pp'-DDE	900
$^{13}C_{12}$-pp'-DDT	900
$^{13}C_{12}$-op'-DDT	900
d_4-alpha-Endosulphan	1000
d_4-beta-Endosulphan	1000
$^{13}C_{12}$-PCB 3	900
$^{13}C_{12}$-PCB 15	900
$^{13}C_{12}$-PCB 37	900
$^{13}C_{12}$-PCB 54	900
$^{13}C_{12}$-PCB 77	900
$^{13}C_{12}$-PCB 118	900
$^{13}C_{12}$-PCB 126	900
$^{13}C_{12}$-PCB 167	900
$^{13}C_{12}$-PCB 169	900
$^{13}C_{12}$ PCB 180	900
$^{13}C_{12}$-PCB 189	900
$^{13}C_{12}$-PCB 202	900
$^{13}C_{12}$-PCB 206	900
$^{13}C_{12}$-PCB 209	900
Recovery Standards	**Nominal Conc. in Recovery Std Solution**
$^{13}C_{12}$-PCB 52	900
$^{13}C_{12}$-PCB 138	900
$^{13}C_{12}$ PCB 153	1000
Extraction Standard	
TCMX –EX	900

ORIGINAL

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Table 2: Analysis of E1 Chlorobenzenes, Pesticides and PCBs Nominal Concentrations of Calibration Standard Solutions (ng/mL)

Compound Name	Calibration Standards				
	Level B	Level C	Level E (Mid-Level)	Level F	Level H
Mono-Cl-Benzene	20	40	160	400	2000
1,3-DiCl-Benzene	20	40	160	400	2000
1,4-DiCl-Benzene	20	40	160	400	2000
1,2-DiCl-Benzene	20	40	160	400	2000
1,3,5-TriCl-Benzene	20	40	160	400	2000
1,2,4-TriCl-Benzene	20	40	160	400	2000
1,2,3-TriCl-Benzene	20	40	160	400	2000
1235/1245-TetraCl-Benzene	40	80	320	800	4000
1,2,3,4-TetraCl-Benzene	20	40	160	400	2000
PentaCl-Benzene	20	40	160	400	2000
HexaCl-Benzene	20	40	160	400	2000
alpha-HCH	40	80	320	800	4000
beta-HCH	40	80	320	800	4000
gamma-HCH	40	80	320	800	4000
delta-HCH	40	80	320	800	4000
Heptachlor	20	40	160	400	2000
Aldrin	40	80	320	800	4000
trans-Chlordane	40	80	320	800	4000
cis-Chlordane	40	80	320	800	4000
trans-Nonachlor	40	80	320	800	4000
cis-Nonachlor	40	80	320	800	4000
op'-DDE	20	40	160	400	2000
pp'-DDE	20	40	160	400	2000
op'-DDT	20	40	160	400	2000
pp'-DDT	20	40	160	400	2000
Octachlorostyrene	20	40	160	400	2000
Oxychlordane	40	80	320	800	4000
op'-DDD	20	40	160	400	2000
pp'-DDD	20	40	160	400	2000
Mirex	20	40	160	400	2000
PCB 1	20	40	160	400	2000
PCB 3	20	40	160	400	2000
PCB 4/10	20	40	160	400	2000
PCB 8/5	20	40	160	400	2000
PCB 15	20	40	160	400	2000
PCB 18	20	40	160	400	2000
PCB 19	20	40	160	400	2000
PCB 28	20	40	160	400	2000

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Compound Name	Calibration Standards				
	Level B	Level C	Level E (Mid-Level)	Level F	Level H
PCB 31	20	40	160	400	2000
PCB 37	20	40	160	400	2000
PCB 40	20	40	160	400	2000
PCB 44	20	40	160	400	2000
PCB 49/43	40	80	320	800	4000
PCB 52/73	40	80	320	800	4000
PCB 54	20	40	160	400	2000
PCB 56/60	40	80	320	800	4000
PCB 66/80	40	80	320	800	4000
PCB 87/115/116	60	120	480	600	3000
PCB 90/101/89	60	120	480	600	3000
PCB 95/93	40	80	320	800	4000
PCB 99	20	40	160	400	2000
PCB 104	20	40	160	400	2000
PCB 105/127	40	80	320	800	4000
PCB 110	20	40	160	400	2000
PCB 114	20	40	160	400	2000
PCB 118/106	40	80	320	800	4000
PCB 126	20	40	160	400	2000
PCB 138/163/164	60	120	480	600	3000
PCB 149/139	40	80	320	800	4000
PCB 151	20	40	160	400	2000
PCB 153	20	40	160	400	2000
PCB 155	20	40	160	400	2000
PCB 156	20	40	160	400	2000
PCB 157	20	40	160	400	2000
PCB 167	20	40	160	400	2000
PCB 169	20	40	160	400	2000
PCB 170/190	40	80	320	800	4000
PCB 180	20	40	160	400	2000
PCB 183	20	40	160	400	2000
PCB 187/182	40	80	320	800	4000
PCB 188	20	40	160	400	2000
PCB 189	20	40	160	400	2000
PCB 194	20	40	160	400	2000
PCB 196/203	40	80	320	800	4000
PCB 202	20	40	160	400	2000
PCB 205	20	40	160	400	2000
PCB 206	20	40	160	400	2000
PCB 208	20	40	160	400	2000
PCB 209	20	40	160	400	2000

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Compound Name	Calibration Standards				
	Level B	Level C	Level E (Mid-Level)	Level F	Level H
Labelled Surrogates					
$^{13}C_6$-1,4-DiCl-Benzene	400	400	400	400	400
$^{13}C_6$-1,2,3-TriCl-Benzene	400	400	400	400	400
$^{13}C_6$-1,2,3,4-TetCl-Benzene	400	400	400	400	400
$^{13}C_6$-PentaCl-Benzene	400	400	400	400	400
$^{13}C_6$-HexaCl-Benzene	400	400	400	400	400
$^{13}C_6$-beta BHC	400	400	400	400	400
$^{13}C_6$-gamma BHC	400	400	400	400	400
$^{13}C_{10}$-Heptachlor	400	400	400	400	400
$^{13}C_{12}$-Aldrin	400	400	400	400	400
$^{13}C_{10}$-trans-Chlordane	400	400	400	400	400
$^{13}C_{10}$-trans-Nonachlor	400	400	400	400	400
$^{13}C_{12}$-pp'-DDE	400	400	400	400	400
$^{13}C_{12}$-pp'-DDT	400	400	400	400	400
$^{13}C_{12}$-PCB 3	400	400	400	400	400
$^{13}C_{12}$-PCB 15	400	400	400	400	400
$^{13}C_{12}$-PCB 37	400	400	400	400	400
$^{13}C_{12}$-PCB 54	400	400	400	400	400
$^{13}C_{12}$-PCB 118	400	400	400	400	400
$^{13}C_{12}$-PCB 167	400	400	400	400	400
$^{13}C_{12}$ PCB 180	400	400	400	400	400
$^{13}C_{12}$-PCB 202	400	400	400	400	400
$^{13}C_{12}$-PCB 206	400	400	400	400	400
$^{13}C_{12}$-PCB 209	400	400	400	400	400
Recovery Standards					
$^{13}C_{12}$-PCB 52	400	400	400	400	400
$^{13}C_{12}$-PCB 138	400	400	400	400	400
Extraction Standard					
TCMX-EX	400	400	400	400	400

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Table 3: GC/ECD Analysis of E2 Pesticides
Nominal Concentrations of Calibration Solutions
(ng/mL)

Compound Name	Calibration Standards				
	Level B	Level C	Level D (Mid-Level)	Level E	Level F
Dieldrin	8	40	80	160	400
alpha-Endosulphan	8	40	80	160	400
beta-Endosulphan	8	40	80	160	400
Endosulphan Sulphate	8	40	80	160	400
Endrin	8	40	80	160	400
Methoxychlor	8	40	80	160	400
Endrin Aldehyde	8	40	80	160	400
Endrin Ketone	8	40	80	160	400
Heptachlor Epoxide	8	40	80	160	400
Surrogates					
d_4-alpha-Endosulphan	75	75	75	75	75
d_4-beta-Endosulphan	75	75	75	75	75
Recovery Standard					
$^{13}C_{12}$-PCB 153	75	75	75	75	75

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3.2 Glassware Cleaning

All glassware used in the preparation of reagents and in the analytical procedures must be organically clean. Glassware must be washed and baked using standard operating procedures.

If baked glassware is not available, glassware must be washed and solvent rinsed following standard operating procedures.

Solvent Rinse Sequence

Acetone (3 times) followed by toluene (3 times) followed by hexane (3 times).
Allow glassware to dry.

3.3 Preparation of Reagents

Activated Copper Turnings are freshly prepared according to standard operating procedure LAB-44.

Anhydrous Sodium Sulphate (Na_2SO_4, AnalaR, powdered) is cleaned by heating at 325°C overnight. The baked sodium sulphate is stored in a glass reagent bottle. Label the bottle with contents and date.

Glass Fibre Filters (Whatman 934 A/H) are cleaned by baking overnight at 325°C.

Saturated Sodium Chloride Solution (NaCl) is prepared by dissolving sodium chloride (300 g, Sifto pickling salt) in Seastar water (1 L). The solution is extracted by shaking twice with dichloromethane (100 mL) and once with hexane (100 mL).

Seastar Water (Seastar Chemicals, contaminant free) is used as received.

Sodium Chloride Solution (3% NaCl) is prepared by diluting a saturated sodium chloride solution 1:10 with Seastar water.

Sodium Hydroxide (NaOH, 1 M) is prepared by dissolving sodium hydroxide pellets (AR grade, 40 g) in distilled water (1 L). Extract the solution by shaking with dichloromethane (2 x 100 mL) followed by hexane (100 mL). Store the solution in a stoppered Erlenmeyer flask.

Solvent-Extracted Distilled Water is prepared by shaking (2 -3 minutes) distilled water (4 L) with dichloromethane (2 x 250 mL) and hexane (250 mL) before use.

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

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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.

Soxhlet Thimbles: used thimbles are soaked in Contrad 70 cleaning solution and rinsed with water until the pH of the rinse water is pH 6-7. Thimbles are baked overnight at 325°C.

3.4 Preparation of Chromatography Materials

Refer to the following Standard Operating Procedures:

AXYS ID	QPulse ID	Title
CHROM-1	SCH-001	Deactiviation/Activation Procedures
CHROM-2	SCH-002	Column Packing Procedures
CHROM-3	SCH-003	Column Cutpoint Procedures
CHROM-4	SCH-004	Layered Silica Gel Chromatography Procedures
CHROM-5	SCH-005	Preparation of Carbon/Celite Column
CHROM-6	SCH-006	Preparation and Maintenance of Biobead Columns

Alumina 1% Deactivated (Fisher, Basic Brockman Activity 1, 60-325 mesh) is baked overnight at 450°C. When cool, deactivate the alumina with Seastar water (1% by weight) and allow to equilibrate for 24 hours. Store alumina under nitrogen in a stoppered flask. Wrap Teflon tape around the stopper to seal the flask.

Acidic Silica (22% H_2SO_4) Add concentrated sulphuric acid (H_2SO_4, Seastar, quartz-distilled, 2.8 g) to silica (baked, 10 g) and agitate until homogeneous. Store the mixture in a stoppered round bottom flask.

Acidic Silica (44% H_2SO_4) Add concentrated sulphuric acid (H_2SO_4, Seastar, quartz-distilled 7.9 g) to silica (baked, 10 g) and agitate until homogeneous. Store the mixture in a stoppered round bottom flask.

Basic Silica (28% NaOH) Add sodium hydroxide solution (NaOH, 1 M, 39 g) to silica (baked, 100 g) and agitate until the mixture is homogeneous. Store the silica in a stoppered round bottom flask.

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

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

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Florisil (Fisher, 60-100 mesh) is activated by heating at 450°C for a minimum of 6 hours. Remove from the muffle furnace to a metal cooling rack and cover loosely with aluminum foil. Allow to cool for 15-20 minutes, transfer to a nitrogen flushed flask, and allow to cool to room temperature in the stoppered flask. When cool, deactivate the Florisil with Seastar water (2.1% by weight) and allow to equilibrate for 24 h.

Silica Gel (Mallinckrodt, SilicAR, 100-200 mesh) is heated for 8 hours at 450°C and stored in a stoppered reagent bottle.

3.5 Preparation of Chromatography Columns

3.5.1 Florisil Column

Fill a glass column (1 cm x 25 cm with 100 mL reservoir) with hexane and slurry pack the Florisil (8 g, 2.1% deactivated). Cap the Florisil with a 10 mm layer of anhydrous sodium sulphate. Elute the column with hexane (40 mL).

3.5.2 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 SOP CHROM-6 for the preparation, use, and cleaning of Biobead columns.

3.5.3 Layered 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 in hexane the following:

silica (1 g), silica (28% NaOH, 2 g), silica (1 g), silica (44% H_2SO_4, 4 g), and silica (22% H_2SO_4, 4 g).

3.5.4 Alumina Column for Extra Cleanup Procedures, Section 6.2

Dry 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 powdered sodium sulphate.

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4.0 QUALITY ASSURANCE/QUALITY CONTROL

Refer to the following Standard Operating Procedure:

AXYS ID	QPulse ID	Title
LAB-16	SLA-016	Preparation of QA/QC Samples

All samples are analyzed in batches. An analysis batch is assigned to an analyst by the Chemist 3. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.
- Blanks - One procedural blank is analyzed for each batch.
- Duplicates – Duplicate samples are analyzed as required by contract. For some matrices (i.e. XAD-2 columns, particulate filters) only field duplicates can be analyzed.
- Reference Samples – Spiked samples are relied on to demonstrate the accuracy of the data. Spiked sediments, tissues, and spiked waters are analyzed at regular intervals. In-house sediment and tissue samples are used to prepare spiked samples. Seastar water is used to prepare spiked water samples. The type of reference material to be analyzed is assigned to the analyst when the batch is assigned. A certified reference material, N.I.S.T. SRM 1588 Cod Oil, is commercially available and used to validate and periodically check the tissue method.
- 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.

The batch composition may vary should batch or quality control requirements be specified by a client.

All elements of the QA/QC program at Axys Analytical Services are documented in the most recent version of the Quality Manual "QA/AC Policies and Procedures Manual.

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

AXYS ID	Title
Specification Table CL/03	E2 Chlorinated Pesticides by GC/ECD
Specification Table CL/04	E1 Chlorinated Pesticides by GC/MS
Specification Table CL/07	PCB Congeners by GC/MS

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SPECIFICATION TABLE CL/03: Analysis of E2 Chlorinated Pesticides by GC/ECD Method

ACCEPTABLE DETECTION LIMITS									
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD column	Procedural Blank Level	Acceptable Matrix Spike % Recovery[1]
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng		
Heptachlor Epoxide	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
alpha-Endosulphan	0.1	1	1	0.1	0.1	1	1	<1 ng	70-130
Dieldrin	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
Endrin	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
Endosulphan sulphate	0.1	1	1	0.1	0.1	1	1	<1 ng	70-130
Endrin ketone	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
beta-Endosulphan	0.1	1	1	0.1	0.1	1	1	<1 ng	70-130
Endrin Aldehyde	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
Methoxychlor	0.5	5	5	0.5	0.5	5	5	<5	60-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 column		
Final Vol, TL	100	100	100	100	100	100	100		
Authentic Spike, ng	40	40	40	40	40	40	40		

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

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
d4-alpha endosulphan	40-130
d4-beta endosulphan	40-130

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Procedural Blank	See table CL/03 or <10% of analyte value.
Matrix Spike Recovery	See table CL/03.
Surrogate Std Recovery	See above
Instrument Sensitivity	S/N ratio ≤3:1 for 50 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 RRF's ≤20%.
RRF: Bracketing Cal	RRF's from calibration standards must agree to ±20% over a 12-hour period.
RRF: Continuing Cal Ver	RRF's from opening/closing calibration standards must be within ±25% of the mean RRF's from initial calibration.
Chromatogram Quality (GC Resolution)	1. Valley height between d_4-alpha endosulphan and alpha endosulphan must be ≤50% of d_4-alpha endosulphan peak height or the separation of d_4-alpha endosulphan and alpha endosulphan must be >0.15 minutes. 2. Endrin breakdown must be ≤20%
Analyte /Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

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SPECIFICATION TABLE CL/04: Analysis of E1 Chlorinated Pesticides by GC/MS Method

ACCEPTABLE DETECTION LIMITS* - SAMPLE SPECIFIC										
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level	Acceptable Matrix Spike % Recovery	
									GC/MS	GC/HRMS
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng	ng		
Dichlorobenzenes	1	10	10	1.0	1.0	10	10	<10	60-130	60-130
Trichlorobenzenes	1	10	10	1.0	1.0	10	10	<19	60-130	60-130
Tetrachlorobenzenes	0.5	5	5	0.5	0.5	5	5	<5	60-130	60-130
Pentachlorobenzene	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
Hexachlorobenzene	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
alpha-HCH	0.2	2	2	0.2	0.2	2	2	<2	70-130	70-130
beta-HCH	0.2	2	2	0.2	0.2	2	2	<2	70-130	70-130
gamma-HCH	0.2	2	2	0.2	0.2	2	2	<2	70-130	70-130
delta-HCH	0.2	2	2	0.2	0.2	2	2	<2	70-130	70-130
Heptachlor	0.5	5	5	0.5	0.5	5	5	<5	70-130	70-130
Aldrin	0.5	5	5	0.5	0.5	5	5	<5	70-130	70-130
Oxychlordane	0.5	5	5	0.5	0.5	5	5	<5	60-130	70-130
trans-Chlordane	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Chlordane	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDE	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDE	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
trans-Nonachlor	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
cis-Nonachlor	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDD	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDD	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
o,p'-DDT	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
p,p'-DDT	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
Mirex	0.1	1	1	0.1	0.1	1	1	<1	70-130	70-130
Toxaphene[1]	0.2	2	2	0.2	0.2	2	2	<10	60-130	60-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 column			
Final Vol, TL	100	100	100	100	100	100	100	100		
Authentic Spike GCMS ng	40	40	40	40	40	40	40			
Authentic Spike HRGCMS ng	2	2	2	2	2	2	2			

*DETECTION LIMITS ARE APPROXIMATELY 10 TIMES LOWER USING GC/HRMS

[1]Toxaphene is determined by Quadrupole GC/MS Electron Capture Negative Ion Analysis. Detection limits are based on minimum area method using the m/e 413 channel.

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Table CL/04 cont'd...

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
$^{13}C_{12}$-1,4-Dichlorobenzene	20-130
$^{13}C_{12}$-1,2,3-Trichlorobenzene	20-130
$^{13}C_{12}$-1,2,3,4-Tetrachlorobenzene	20-130
$^{13}C_{12}$-Pentachlorobenzene	30-130
$^{13}C_{12}$-Hexachlorobenzene	30-130
$^{13}C_{12}$-beta-HCH	30-130
$^{13}C_{12}$-gamma-HCH	40-130
$^{13}C_{12}$-Heptachlor	30-130
$^{13}C_{12}$-Aldrin	30-130
$^{13}C_{12}$-tran-Chlordane	30-130
$^{13}C_{12}$-trans-Nonachlor	30-130
$^{13}C_{12}$-p,p'-DDE	40-130
$^{13}C_{12}$-p,p'-DDT	40-130
$^{13}C_{12}$-PCB 15	40-130

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Procedural Blank	See Table CL/04 or <10% of analyte value <10 ng for toxaphene
Matrix Spike Reovery	See Table CL/04
Surrogate Std Recovery	See above.
Instrument Sensitivity	S/N ≥3:1 for 10 pg HCB, 10 pg pp-DDT, 10 pg PCB 118 and 20 pg oxychlordane S/N ≥2:1 for 2.8 ng of Toxaphene with a minimum of 5 peaks detected
Instrument Linearity	For a minimum 5-point calibration, a relative standard deviation of the RRF's ≤20% for all compounds
RRF: Bracketing Cal	RRF's from calibration standards must agree to ±20% over a 12-hour period.
RRF: Continuing CAL Ver	RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration for all compounds
Chromatogram Quality **Max Peak Width:** **Resolution:**	1. Peak width at half height for p,p'-DDT is 5 sec. 2. Baseline separation between p,p'-DDD and o,p-DDT. 3. PCB 209 peak must be symmetrical with negligible tailing, 20 sec. 4. pp-DDT breakdown must be ≤15%.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window	RRT must be ±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 labelled analogue

FINAL

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SPECIFICATION TABLE CL/07: Analysis: PCB Congeners by GC/MS Method

ACCEPTABLE DETECTION LIMITS*										
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery	
Congener (IUPAC):	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/col		GC/MS	HRC/HRMS
Monochlorinated (1-3)	0.5	5.0	5	0.5	0.5	50	5	<1	60-130	70-130
Dichlorinated (4-15)	0.5	5.0	5	0.5	0.5	50	5	<1	60-130	70-130
Trichlorinated (16-39)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Tetrachlorinated (40-81)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Pentachlorinated (82-127)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Hexachlorinated (128-169)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Heptachlorinated (170-193)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Octachlorinated (194-205)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Nonachlorinated (206-208)	0.1	1	1	0.1	0.1	10	1	<1	70-130	70-130
Decachlorinated (209)	0.5	5	5	0.5	0.5	50	5	<1	70-130	70-130
Aroclor Equivalents	1	10	10	1	1	100	10	<1	60-130	70-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 col			
Final Vol, TL	100	100	100	100	100	100	100			
Authentic Spike GCMS, pg	40	40	40	40	40	40	40			
Authentic Spike HRGCMS pg	2	2	2	2	2	2	2			

* Detection Limits are approximately 10 times lower by GC/HRMS.

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
^{13}C PCB 3	15-130
^{13}C PCB 15	20-130
^{13}C PCB 37	40-130
^{13}C PCB 54	40-130
^{13}C PCB 118	40-130
^{13}C PCB 167	40-130
^{13}C PCB 180	40-130
^{13}C PCB 202	40-130
^{13}C PCB 206	40-130
^{13}C PCB 209	40-130

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
Procedural Blank	See table CL/07 or <10% of analyte value.
Matrix Spike Recovery	See Table CL/07
Surrogate Std Recovery	See above
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 RRF's ≤20%.
RRF: Bracketing Cal	RRF's from calibration standards must agree to ±20% over a 12-hour period.

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Table CL/07 Cont'd....

RRF Continuing CAL VER	RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration.
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 ≤80% of smallest peak height of PCB 28/31 pair.
Analyte/Surrogagte Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Time Window	RRT must be ±3 sec of the predicted retention time determined from the calibration standard and adjusted relative to the peak retention time reference (labelled surrogate) Authentic compound must elute after its labelled analogue

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5.0 EXTRACTION PROCEDURES

Samples are analyzed in batches. The Chemist 3 assigns analysis batches to an analyst. The samples to be analyzed, the batch QC samples, the name and volume of the surrogate, recovery and calibration standards required, and any additional information concerning the analysis are documented on a batch list given to an analyst.

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

AXYS ID	QPulse ID	Title
LAB-4	SLA-004	Sample Control Procedures
LAB-5	SLA-005	Rotary Evaporation Concentration Technique
LAB-6	SLA-006	Nitrogen Blowdown Concentration Technique
LAB-7	SLA-007	Kuderna-Danish Concentration Technique
LAB-8	SLA-008	Preparing Extracts for Instrumental Analysis
LAB-17	SLA-017	Spiking Procedure
LAB-20	SLA-020	Lipid Determination by Weight of Extract Subsample
LAB-21	SLA-021	Lipid Determination by Volume of Extract Subsample
LAB-27	SLA-027	Completing a Worksheet
LAB-28	SLA-028	Independent Gravimetric Determination of Lipid Content
LAB-30	SLA-030	Micro-Lipid Determination
LAB-33	SLA-033	Assigning Analysis Batches
LAB-45	SLA-045	Removal of Sulphur From Extracts Using Activated Copper
LAB-72	SQA-72	Computer Preparation of Labels
LAB-67	SLA-067	Use of Nitrogen Cylinders
ADMIN-4	SAD-004	Chain of Custody Procedures

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5.1 Sediment/Soil/Ash/Sludge/Gravel/Particulate Filter Extraction

5.1.1 Shaker Table Extraction Procedure

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

Matrix	Typical Sample Size
Sediment/Soil	15 g (wet)
Filter	Whole Filter
Sludge	10 g (wet)
Ash	5 g (dry)

Accurately weigh a sample into a round bottom flask and add an aliquot(s) of the appropriate surrogate standard solution(s). Add dichloromethane:methanol (1:1, 80 mL) to the flask, stopper the flask, and secure the stopper with a plastic clamp. Extract the sample by shaking the flask on a shaker table for 20 minutes. Remove the flask from the shaker table and allow the mixture to separate. Decant the solvent through solvent rinsed filter paper (Whatman #1, Whatman 54 (12. 5 cm), or Whatman fluted filter paper) into a 500 mL separatory funnel. Repeat the extraction procedure using dichloromethane (80 mL) and shaking for 20 minutes. Decant the solvent through the filter paper into the separatory funnel. Allow the filter to drain and rinse twice with dichloromethane (15 mL/rinse). Allow to drain. Discard the filter paper.

Wash the extract by adding Seastar water (50 mL) to the separatory funnel and shaking the mixture for one minute. Discard the aqueous layer and repeat the wash. This step removes the methanol. Collect the dichloromethane extract in an Erlenmeyer flask (500 mL) and dry over anhydrous powdered sodium sulphate for 15-20 min.

Transfer the extract to a Kuderna-Danish (KD) flask (250 mL). Rinse the Erlenmeyer flask with dichloromethane (3 X 15 mL). Add the rinses to the KD flask. Add hexane (2 mL) as a "keeper" and a few boiling chips. Place the chimney onto the flask and fill with glass rings. Concentrate in a 60°C water bath, reducing the volume to about 1 mL. Remove the flask from the water bath and rinse the chimney with a small amount of hexane. Allow to cool before removing the chimney (5-10 min.) Add activated copper turnings to the extract to remove sulphur.

Transfer the extract to a centrifuge tube with three hexane rinses (2 - 3 mL each) and concentrate to 1 mL under a gentle stream of nitrogen. The extract is ready for the column cleanup procedure (6.0).

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5.1.2 Shaker Table Extraction Procedure- Gravel

This method is a modification to the shaker table extraction method (Section 5.11). The modification applies to gravel samples and uses a large sample size in order to achieve required detection limits. The quantities of reagents required have been adjusted accordingly.

Matrix	**Sample Size**
Gravel	200 g

Accurately weigh a sample into a round bottom flask and add an aliquot(s) of the appropriate surrogate standard solution(s). Add dichloromethane:methanol (1:1, 250 mL) to the flask, stopper the flask, and secure the stoppers with Teflon tape. Extract the sample by shaking the flask on a shaker table for 20 minutes. Remove the sample from the shaker table and allow the mixture to separate. Decant the solvent through Whatman #1, Whatman 54 (12. 5 cm), or Whatman fluted filter paper into a separatory funnel (1 L). Repeat the extraction procedure using 200 mL of dichloromethane and shaking 20 minutes. Decant the solvent through the filter paper into the separatory funnel. Allow the filter to drain and rinse twice with dichloromethane (15 mL/rinse). Allow to drain. Discard the filter paper.

Wash the extract by adding Seastar water (100 mL) to the separatory funnel and shaking the mixture for one minute. Discard the aqueous layer and repeat the wash. This step removes the methanol. Collect the dichloromethane extract in an Erlenmeyer flask (500 mL) and dry over anhydrous powdered sodium sulphate for 15-20 min.

Transfer half the extract to a Kuderna-Danish (KD) flask (250 mL). Add hexane (2 mL) as a "keeper" and a few boiling chips. Place the chimney onto the flask and fill with glass rings. Concentrate in a 60°C water bath, reducing the volume to about 1 mL. Add the rest of the extract to the KD flask. Rinse the Erlenmeyer flask with dichloromethane (3 X 15 mL). Add the rinses to the KD flask. Concentrate in a 60°C water bath, reducing the volume to about 1 mL. Remove the flask from the water bath and rinse the chimney with a small amount of hexane. Allow to cool before removing the chimney (5-10 min.)

Transfer the extract to a centrifuge tube with three hexane rinses (2 - 3 mL each) and concentrate to 1 mL under a gentle stream of nitrogen. Add activated copper turnings to the extract to remove sulphur. The extract is ready for the column cleanup procedure (6.0).

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5.2 Tissue Extraction by Column Elution

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

Matrix	Sample Size
Tissue	5 - 10 g
Fat	1 g

Accurately weigh a homogenized tissue sample into a beaker containing anhydrous powdered sodium sulphate (100-150 g). Mix the mixture well with a spatula, and allow the mixture to dry for one hour (longer if necessary). Transfer the mixture to a glass mortar and grind with a glass pestle to a free-flowing powder. Add an aliquot(s) of the appropriate surrogate standard solution(s) and mix with a spatula. Quantitatively transfer the ground sample to a glass chromatographic column containing dichloromethane (100 mL). Allow the solvent to migrate to the top of the column. Add additional dichloromethane (100 mL) and elute the column at 3 - 5 mL/min. Collect the eluate in a Kuderna-Danish flask. Add a few boiling chips and hexane (2 mL) as a "keeper" to the K.D. flask. Place the chimney onto the flask and fill with glass rings. Concentrate the extract to 1 mL (5 mL if lipid analysis required) in a water bath at 60°C. Remove the flask from the water bath and rinse the chimney with a small amount of hexane. Allow to cool before removing the chimney (5-10 min.)

If gravimetric lipid analysis is required, subsample the extract and perform the lipid determination at this point. (Refer to SOPs LAB-20 or LAB-21.)

Add dichloromethane (1 mL) to the extract for gel permeation column (Biobead) cleanup.

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 second fraction and evaporate to a small volume by rotary evaporation. Transfer to extract to a centrifuge tube with hexane rinses. Concentrate to 1 mL under a stream of nitrogen. The extract is ready for the column cleanup procedure. (6.0)

If only coplanar PCBs (Analysis Code **N)** are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B). Depending upon the nature of the extract, some extracts may require Florisil cleanup first (6.0).

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5.3 Soxhlet Extraction Procedures

5.3.1 Tissue Soxhlet Extraction

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

Matrix	Sample Size
Tissue	5 - 10 g

Accurately weigh a homogenized tissue sample into a beaker containing anhydrous powdered sodium sulphate (75-100 g). Stir the mixture well with a spatula, and allow the mixture to dry for one hour (longer if necessary). Transfer the mixture to a glass mortar and grind with a glass pestle to a free-flowing powder. Quantitatively transfer the ground sample to a sintered glass soxhlet thimble. Add an aliquot(s) of the appropriate surrogate standard solutions(s) to the sample. The mixture is soxhlet extracted with dichloromethane (300 mL) for three hours. Allow the mixture to cool. Concentrate the extract to 1 mL by rotary evaporation (water bath <40°C) and then quantitatively transfer to a 15 mL centrifuge tube with hexane rinses.

If a lipid analysis is required, concentrate the extract to 5-20 mL by rotary evaporation and carry out the lipid determination according to standard operating procedures.

Add hexane (1 mL) as "keeper" to the remaining extract. Reduce the volume of final extracts to 1 mL in a gentle nitrogen stream. Add dichloromethane (1 mL) to the extract for gel permeation column cleanup.

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 second fraction and evaporate to a small volume by rotary evaporation. The extract must be in hexane. The extract is ready for the column cleanup procedure (6.0).

If only coplanar PCBs (Analysis Code **N)** are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B). Depending upon the nature of the extract, some extracts may require Florisil cleanup first (6.0).

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5.3.2 Pulp Soxhlet Extraction

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

Matrix	Typical Sample Size
Pulp	20 g

Load a homogenized pulp sample into a sintered glass soxhlet thimble containing 4 g silica. Add an aliquot(s) of the appropriate surrogate standard solution(s) to the thimble. The mixture is soxhlet extracted with dichloromethane (300 mL) for three hours. Allow the mixture to cool. Concentrate the extract to 1 mL by rotary evaporation (water bath <40°C). Add activated copper to the extract to remove sulphur.

Note: The following cleanup procedure cannot be used if pesticide analysis is required. If pesticide analysis is required, proceed to Section 6.0

Load the extract onto a layered acid/base silica column (as described in Section 3.5.3) 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 <40°C).

5.3.3 Sludge, Ash and Sediment Soxhlet Extraction

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

Matrix	Typical Sample Size
Sludge	10 g
Ash	5 g
Sediment	5 – 10 g

Accurately weigh a homogenized sample into a beaker containing anhydrous powdered sodium sulphate (100-150 g). Stir the mixture well with a spatula, and allow the mixture to dry for one hour (longer if necessary). Transfer the mixture to a glass mortar and grind with a glass pestle to a free-flowing powder. Quantitatively transfer the ground sample to a sintered glass soxhlet thimble. Add an aliquot(s) of the appropriate surrogate standard solutions(s). The mixture is soxhlet extracted with dichloromethane (300 mL) for 16 to 20 hours. Allow the mixture to cool. Concentrate the extract to 1 mL (5 mL if lipid analysis is required) by rotary evaporation (water bath <40°C). Add activated copper to the extract to remove sulphur. Quantitatively transfer the extract to a 15 mL centrifuge tube with hexane rinses.

If pesticide analysis is required, proceed to Florisil column cleanup procedures (6.0). If pesticide analysis is not required, cleanup ash and sludge extracts on a layered acid/base silica

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column, as described below. Depending on the nature of a sediment extract, it may also require a layered acid/base silica column cleanup. If not, proceed to Section 6.0.

Note: The following cleanup procedure cannot be used if pesticide analysis is required. If pesticide analysis is required, proceed to Section 6.0.

Load the extract onto a layered acid/base silica column (as described in Section 3.5.3) with rinses (2 x 1 mL hexane). Elute the column with hexane (100 mL) and collect all the eluate. Evaporate the solvent to a small volume by rotary evaporation (Water bath <40°C). The extract is ready for the column cleanup procedure (Section 6.0).

5.3.4 XAD-2 Resin Soxhlet Extraction

This extraction procedure is applicable to the matrix listed below.

Matrix	Sample Size
XAD Resin	1 column

Each sample requires a baked and solvent rinsed Millipore filtration apparatus and Millipore thimble.

1. Choose a Millipore thimble appropriate for the column size.
2. Pour the XAD resin from the jar into the Millipore thimble.
3. Rinse the jar well with Seastar water to transfer all the XAD resin into the thimble.
4. Remove the water using the Millipore filtration apparatus. Allow the XAD resin to dry under vacuum for 15 minutes. If necessary, add a foil cap to the top of the thimble to increase suction.
5. Transfer the thimble containing the dry XAD resin to the soxhlet apparatus.
6. Add the appropriate volume of solvents to make up either 300 mL or 600 mL of dichloromethane, as required for the size of soxhlet. If extract is also being analyzed for PCDD/F compounds, substitute 80:20 toluene:acetone for dichloromethane.
7. If a single analysis is required, spike an aliquot of the surrogate solution onto the XAD resin in the thimble prior to starting the soxhlet extraction. If multiple analyses are required, spike the surrogates after the extraction and extract splitting procedures.
8. The sample is soxhlet extracted for 16 - 20 hours using dichloromethane (or 80:20 toluene:acetone if PCDD/F analysis required) as the solvent. Cool the solution. Remove water from the extract (5.3.6).

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5.3.5 Filter Soxhlet Extraction

This extraction procedure is applicable to the matrix listed below.

Matrix	Typical Sample Size
Glass Fibre or Wrapped Filters	1 to several

Allow filters to air-dry for 3-4 days before soxhlet extraction. Use larger soxhlet bodies and condensers to hold the filters; thimbles are not required. Place the filters or filter cartridge directly into the soxhlet body. Use 1000 mL round bottom flasks with 600 mL of dichloromethane for extraction. If extract is also being analyzed for PCDD/F compounds, substitute 80:20 toluene:acetone for dichloromethane. If a single analysis is required, spike the surrogate solution onto the filters in the soxhlet prior to starting the extraction. If multiple analyses are required, add surrogates after the extraction and extract splitting procedures. The sample is soxhlet extracted for 16-20 hours. Cool extract. Remove water from the extract (5.3.6).

5.3.6 Removal of Water from XAD Resin or Filter Extracts

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

The water removal step is dependent on the amount of visible water.

1. If less than 3 mL of water is visible, transfer the entire extract to an Erlenmeyer flask and dry over anhydrous powdered sodium sulphate. Return the extract to the round bottom flask with complete dichloromethane (or toluene if PCDD/F analysis required) rinses. Concentrate by rotary evaporation to 1-2 mL for extract splitting.

2. If greater than 3 mL of water is visible, transfer the extract to a separatory funnel and separate the water from the sample extract. Draw the organic layer (bottom layer if dichloromethane, top layer if toluene) into a 500 mL Erlenmeyer flask. Extract the aqueous portion twice with dichloromethane (25-50 mL). Discard water layer. Combine dichloromethane extracts with the original dichloromethane extract in the 500 mL Erlenmeyer. (NOTE: If processing filters, larger glassware will be required due to increased extract volume.) Dry over anhydrous powdered sodium sulphate. Transfer the extract back to the round bottom flask with dichloromethane rinses. Concentrate by rotary evaporation to 1-2 mL.

If using the 1000 mL round bottom flasks for the soxhlet, transfer with toluene to 500 mL round bottom flasks during the evaporation process.

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3. Split the sample extract gravimetrically as per batch instructions and record all sample weights.

4. If surrogate standard was not added prior to soxhlet extraction, then spike the PCB/pesticide portion with the appropriate surrogate standard(s).

5. If the sample is being prepared for clean-up on a BioBead column, concentrate the extract under a gentle stream of nitrogen to 500 µL, then dilute to 2 mL with 1:1 dichloromethane:hexane.

6. If the sample is being prepared for clean-up on a Florisil column, concentrate the extract under a gentle stream of nitrogen to 50 µL, then dilute to 1 mL with hexane.

The extract is now ready for chromatographic cleanup (6.0).

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5.4 Effluent/Leachate/Water Extraction

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

Matrix	Sample Size
Effluent, Leachate	1 L
Water, Runoff	1 L

5.4.1 Effluent - Solvent Extraction Procedure

Sample Preparation

Pour an accurately measured sample into an Erlenmeyer flask along with Seastar water rinses of the original container. Add an aliquot of the surrogate standard solution(s) to acetone (1 mL), mix well, and add this solution to the effluent sample, with mixing (10 min). The sample is ready for the solvent extraction procedure.

(a) Extraction of Filter

Filter the sample through a Millipore filter system containing a pre-weighed baked glass fibre filter paper and a glass fibre filter bed, followed by rinsing with Seastar water. Carefully remove the filter and filter bed and air dry in a fumehood overnight. Weigh the dried filter paper with the particulate and load it into a soxhlet thimble[1]. Allow the sample to reflux for 16-20 hours using dichloromethane (300 mL) as the solvent. Cool the solution. Concentrate the 300 mL extract to <10 mL by rotary evaporation. Combine this extract with the extract obtained from the filtrate. (5.4.2b). Add hexane (2 mL) as a "keeper" solvent to the combined extract. Concentrate the extract to 2 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to 1 mL. The extract is ready for the Florisil cleanup procedures (Section 6.0).

(b) Extraction of Filtrate

Carefully rinse the filter apparatus and original container with dichloromethane. Transfer the filtrate and rinses to a clean flask. Add dichloromethane (300 mL) to the flask and stir the solution for a minimum of 8 hours. Transfer the solution to a 2 L separatory funnel. Draw off the dichloromethane layer. Extract the remaining aqueous layer by shaking dichloromethane (2 x 100 mL). Combine all the dichloromethane extracts in an Erlenmeyer flask and dry over

[1] Refer to Option 2 in Section 5.4.1b for a modified soxhlet procedure.

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anhydrous powdered sodium sulphate. Transfer the extract to a round bottom flask (500 mL) and concentrate to 5 mL by rotary evaporation.

At this stage there are two options for continuation of the extraction. The choice is made by the analyst and depends upon the most efficient use of the analyst's time.

Option 1:

Combine the extract from the filtrate with the extract obtained from the soxhlet extraction of the filter (step 5.4.2a). Add hexane (2 mL) as a "keeper" solvent to the combined extract. Concentrate the extract to 2 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane rinses and concentrate to 1 mL. The extract is ready for the Florisil cleanup procedures (Section 6.0).

Option 2:

The round bottom flask containing the extract of the filtrate is used as the flask for the soxhlet extraction procedure, step 5.4.1a. Consequently the extracts are combined prior to the soxhlet procedure.

5.4.2 Water Solvent Extraction Procedure

Clean water (e.g. drinking water) does not require filtration.

Add an aliquot(s) of the appropriate surrogate standard solution(s) to acetone (~1 mL). Add the solution to a measured sample (1 L) in a separatory funnel. Allow the solution to equilibrate for 15 minutes. Extract the sample by adding dichloromethane (100 mL) to the separatory funnel 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 powdered sodium sulphate for at least ½ hour. Transfer the extract to a Kuderna-Danish flask (500 mL) with dichloromethane. Add boiling chips and hexane (2 mL) to the KD flask and concentrate the extract to 2 mL (water bath 60°C). Transfer the sample to a centrifuge tube with hexane rinses and concentrate to 1 mL under a gentle stream of nitrogen. The extract is then ready for the column cleanup procedure (6.0).

If only coplanar PCBs (Analysis Code **N)** are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B). Depending upon the nature of the extract, some extracts may first require Florisil cleanup (6.0).

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5.5 Samples Submitted as Extracts

This extraction procedure is applicable to the matrix listed below.

Matrix	Sample Size
Solvent Extract	One Extract

Determine the weight of the extract as received as follows:

- Weigh the extract in the original container.
- Transfer the extract to a centrifuge tube or an Erlenmeyer flask with rinses, using the same solvent as the extract.
- Re-weigh the original container once it has air-dried.
- Record all weights on the worksheet.

If only PCB/pesticide analysis is required, spike the extract with aliquots of the appropriate surrogate standards solution(s). If multiple analyses are required, spike the portion of extract designated for PCB/pesticide analysis with aliquots of the appropriate surrogate standard solution(s) after drying and splitting of the extract.

Inspect the extract for the presence of water. If less than 3 mL water, transfer extract to an Erlenmeyer flask with solvent rinses (the same solvent as sample) and dry with anhydrous powdered sodium sulphate. Transfer the extract to a clean round bottom flask with solvent rinses. Concentrate the extract to 10-20 mL by rotary evaporation in preparation for sample splitting.

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. Extract the water layer in a separatory funnel with two portions of the solvent. Add the solvent extracts to the original sample in the Erlenmeyer flask and discard the water. Dry the solvent with anhydrous powdered sodium sulphate. Transfer the solvent to a clean round bottom flask with solvent rinses and reduce volume to 10-20 mL by rotary evaporation.

Determine from the batch sheet if the sample requires a single or multiple analysis. If multiple analysis is required, gravimetrically split the sample into equal portions to satisfy the analysis requirements and record all sample weights. If only a single analysis is required, save ½ of sample for back-up. When splitting, always save a back-up portion. Check the batch list for detailed splitting and surrogate spiking instructions.

If the sample is not a hexane extract, a solvent exchange to hexane must be done before proceeding to column cleanup procedures (Section 6.0).

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5.6 Milk Extraction – Solvent Extraction Procedure

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

Matrix	Typical Sample Size
Milk	40 - 50 g

1. Accurately weigh a subsample of milk (50 g). Add the sample to 200 mL of 2:1 acetone:hexane in a 500 mL separatory funnel. Add aliquots of surrogate standards which have been dissolved in ~1 mL acetone. Shake the mixture for 2 min and allow the layers to separate.

2. Transfer the aqueous layer to an Erlenmeyer flask and the hexane 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.

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

4. Drain the hexane extract into an Erlenmeyer flask and dry over anhydrous powdered sodium sulphate.

5. Transfer the dried extract to a 500 mL round bottom flask and concentrate to ~5 mL by rotary evaporation.

6. **Lipid Determination** - Transfer the extract to a 50 mL graduated cylinder with ground-glass stopper and dilute to 30 mL mark with hexane. Pipet two 2 mL aliquots into tared weigh boats for gravimetric lipid analysis.

7. Transfer the remaining extract to a 100 mL round bottom flask and concentrate to ~1 mL by rotary evaporation. Add 1 mL dichloromethane.

8. Load the extract onto a Biobead SX-3 column and elute with 325 mL 1:1 dichloromethane:hexane. Discard the first 150 mL fraction. Collect the 150 to 325 mL fraction.

Concentrate the extract to 1 mL by rotary evaporation followed by evaporation under a gentle stream of nitrogen. The extract is ready for chromatographic cleanup procedure (6.0).

If only coplanar PCBs (Analysis Code **N**) are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B). Depending upon the nature of the extract, some extracts may require Florisil cleanup first (6.0).

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5.7 Blood Extraction Procedure

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

Matrix	Typical Sample Size
Blood	5 to 20 g

1. Accurately weigh a sample of serum (5 to 20 g) into a 250 mL round bottom flask. Add aliquots of surrogate standards and allow to equilibrate for 30 min.

2. Add 10 mL ethanol, 50 mL hexane and 10 mL saturated ammonium sulphate to the sample and shake for 30 min on the shaker table.

   ***Note** - The volume of ethanol and ammonium sulphate should be similar to the sample size and no more than one third the volume of hexane used.

3. Decant the hexane layer into a 500 mL separatory funnel.

4. Add 50 mL hexane to the aqueous layer in the round bottom flask and shake the mixture for 30 min on a shaker table. Decant the hexane into the separatory funnel containing the first hexane extraction. Discard the bottom aqueous layer.

5. Backwash the hexane extract by shaking with Seastar water (2x50 mL) to remove any residual ethanol.

6. Drain the hexane extract into a 500 mL erlenmeyer flask and dry with anhydrous sodium sulphate.

7. Filter the dried extract through baked 934/AH filter paper into a 100 mL round bottom flask. Evaporate just to dryness by rotary evaporation.

8. **Lipid Determination** - Add 4 mL 1:1 dichloromethane:hexane to the round bottom flask. Transfer two 400 TL subsamples to tared weigh boats for gravimetric lipid analysis. Refer to SOP LAB-30 (SLA-30) "Micro-Lipid Determination"

9. Load the remainder of the hexane extract onto a Biobeads SX-3 column and elute with 1:1 dichloromethane:hexane. Refer to the most recent Biobead cutpoint determination for the elution volume. Discard the first fraction. Collect the second fraction.

10. Concentrate the extract to a small volume by rotary evaporation. Transfer to a centrifuge tube with hexane rinses. Concentrate to 1 mL under a stream of nitrogen. The extract is ready for the column cleanup procedure. (6.0)

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If only coplanar PCBs (Analysis Code **N)** are required, the extract can proceed directly to coplanar chromatographic cleanup step (Appendix B). Depending upon the nature of the extract, some extracts may require Florisil cleanup first (6.0).

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5.8 Ambient Air (PUF/Filter) Extraction Method

This method is applicable to the analysis of ambient air samples. The PUF and associated filter(s) are extracted together as one sample.

Matrix	Typical Sample Size
Air Sample	PUF + Filter(s)

5.8.1 Sample Preparation

Using clean solvent-rinsed forceps, transfer the PUF and filter(s) to a soxhlet thimble.

5.8.2 Soxhlet Extraction Procedure

Place the soxhlet thimble containing the PUF and filter in a soxhlet apparatus. If a single analysis is required, spike the surrogate solution onto the sample prior to starting the extraction. If multiple analyses are required, spike the surrogates after the extraction and extract splitting procedure. Heat the sample under reflux for 16-20 hours using 80:20 toluene:acetone as the solvent. Allow the extract to cool and concentrate by rotary evaporation to 5-10 mL. Gravimetrically split the extract (described below) 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.

5.8.3 Gravimetric Splitting Procedures

Transfer the extract to a tared centrifuge tube with solvent rinses (1 toluene rinse followed by 2 hexane rinses) Using a balance that weighs to three decimal places, weigh the centrifuge tube with extract to determine the total weight of the extract. Stir the extract with a vortex mixer. Using a clean Pasteur pipet, transfer one-half (by weight) of the extract to a tared, clean glass ampoule. Record the weight of the extract. Seal and label the ampoule. If multiple analyses are required, use the procedures above, splitting the extract into the designated number of portions and sealing the portions in ampoules.

Concentrate the portion of the extract for PCB/pesticide analysis to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane and evaporate to "just dry" under a stream of nitrogen. Add hexane (1 mL). The extract is ready for column cleanup procedures (Section 6.0).

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5.9 Stationary Source Air Samples (Stack Gas)

5.9.1 Sample Preparation

Stack gas samples are usually submitted as sample trains in several containers labelled A to F or 1 to 6. It is essential that the analyst confirm that the labels on the sample container match those on the sample log-in information prior to commencing extraction procedures.

Front Half of Train

a) Container A (1)

The hexane/acetone rinses from the probe liner, nozzle, cyclone and front half of the filter housing are in container A.

Concentrate the rinses to near dryness by rotary evaporation followed by evaporation under a nitrogen stream. Transfer the residue with toluene rinses to a beaker containing anhydrous sodium sulphate (Na_2SO_4, 20 g). Mix well. The mixture is ready to be transferred to the soxhlet thimble, Section 5.9c.

b) Container B (2)

A particulate filter is in container B.

Transfer the filter to a clean, tared, piece of foil. Weigh the filter and record the weight on the work sheet. The filter is ready to be transferred to the soxhlet thimble containing XAD resin and container A residue, Section 5.9c.

Back Half of Train

c) Container C (3)

XAD resin is in container C.

Place a soxhlet thimble into a large glass funnel, which is set in a beaker (400 mL). Handle the thimble with a clean piece of foil. Remove the glass wool from the XAD container and place half into the soxhlet thimble. Rinse the XAD resin from the container into the soxhlet thimble with acetone. Place the other half of the glass wool on top of the resin. Drain for at least one hour into the beaker.

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Transfer the residue/sodium sulphate mixture (Section 5.9a) with toluene rinses from container A and the filter from container B (Section 5.9b) to the top of the soxhlet thimble containing the XAD resin.

Add an aliquot of the surrogate standard to the soxhlet thimble. If multiple analyses of the extract are required, refer to the Batch List for the name(s) and volume(s) of surrogate standard solutions.

The acetone rinses are ready to be combined with the contents of container E (impinger water washes) (Section 5.9e) in a separatory funnel.

d) Container D (4)

Hexane/acetone rinses of the condenser, coil and back half of filter housing are in container D.

Dry the contents of container D with powdered anhydrous sodium sulphate (Na_2SO_4) if any water is visible. Concentrate by rotary evaporation. Transfer the extract with toluene to the 500 mL round bottom flask used for the soxhlet extraction of the XAD and filters (Section 5.9c).

e) Container E (5)

Impinger contents and water rinses are in container E.

Transfer the contents of the container to a separatory funnel. Rinse the container with dichloromethane. Extract by shaking with dichloromethane (3 x 100 mL). Combine the extracts, dry with powdered anhydrous sodium sulphate (Na_2SO_4) and transfer to a Kuderna-Danish flask (500 mL). Add toluene (1 mL) and concentrate (water bath 55°C). Transfer the extract with toluene rinses to the 500 mL round bottom flask used for the soxhlet extraction of the XAD and filters (Section 5.9c).

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f) Container F (6)

Final solvent rinses of the condenser coil, back half of filter holder and impingers are in container F. This container is not always submitted by the client.

Dry the contents of container F with powdered anhydrous sodium sulphate (Na_2SO_4) if any visible water is present. Concentrate by rotary evaporation. Transfer the extract, using toluene, to the 500 mL round bottom flask used for the soxhlet extraction of the XAD and filters (Section 5.9c).

5.9.2 Soxhlet Extraction Procedure

Add 80:20 toluene:acetone (300 mL) to the 500 mL round bottom flask and extract the XAD/filters under reflux for 16-20 hours. Allow the extract to cool and concentrate by rotary evaporation to 5-10 mL. The extract is ready to be gravimetrically split 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.

5.9.3 Gravimetric Splitting Procedures

Transfer the extract to a tared centrifuge tube. Using a balance that weighs to three decimal places, weigh the centrifuge tube with extract to determine the total weight of the extract. Stir the extract with a vortex mixer. Using a clean Pasteur pipet, transfer one-half (by weight) of the extract to a tared, clean glass ampoule. Record the weight of the extract. Seal and label the ampoule. If multiple analyses are required, use the procedures above, splitting the extract into the designated number of portions and sealing the portions in ampoules.

Concentrate the portion of the extract for PCB/pesticide analysis to 1 mL by rotary evaporation. Transfer the extract to a centrifuge tube with hexane and evaporate to "just dry" under a stream of nitrogen. Add hexane (1 mL). The extract is ready for column cleanup procedures (Section 6.0).

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6.0 COLUMN CLEANUP PROCEDURES

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.

Some extracts require additional cleanup procedures to remove interferences. These procedures are described in Section 6.2.

The additional cleanup procedures for the analysis of coplanar PCBs (Analysis Code **N)** (if requested) are described in the Appendix C.

6.1 Florisil Column Cleanup of E1 and E2

Quantitatively transfer the extract and hexane rinses (3 x 1 mL) to a Florisil column (8 grams, 2.1% deactivated) for cleanup and fractionation. The extract is separated into two fractions. Elute the column with dichloromethane:hexane (15:85, 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	15:85 dichloromethane:hexane	45 mL
E2	dichloromethane	50 mL

Concentrate each fraction to a small volume (~ 1 mL). Add activated copper to sediment, soil, particulate filters, water, effluent, and hexane extract E1 extracts to remove sulphur. Transfer each extract to a centrifuge tube with hexane and concentrate the extract to 500 TL under a stream of nitrogen. Transfer each extract to an autosampler vial. Concentrate the extract to a small volume under a gentle stream of nitrogen. Rinse the centrifuge tube with hexane and add the rinse to the autosampler vial. Concentrate the extract to a small volume under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more. Add an aliquot of the appropriate recovery standard to each extract. Adjust the volume to 100 TL with hexane. Cap the autosampler vial. The extract is ready for instrumental analysis (Section 7.1).

E1 is analyzed by GC/MS for some or all of the following: Aroclors, most chlorinated pesticides, PCB congeners, coplanar PCBs (NOS), and chlorobenzenes. An additional GC/MS-ECNI analysis determines the concentration of toxaphene.

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

If coplanar PCBs (Analysis Code **N)** are required, proceed to the separation and cleanup of NOS (Appendix C).

6.2 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. Once instrumental analysis has been carried out, the presence of interferences in the chromatogram may necessitate additional cleanup procedures followed by re-analysis. These are carried out upon request from a Data Interpretation Chemist or QC Chemist.

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 and elute with 1:1 dichloromethane:hexane. Collect the second fraction. Refer to the most recent Biobead cutpoint for the volume of solvent to collect. Concentrate the eluate by rotary evaporation. Concentrate the extract to 1 mL by rotary evaporation and transfer to a centrifuge tube. Concentrate further under a stream of nitrogen and transfer to an autosampler vial. Adjust the volume to that specified for the fraction in Sections 6.1.

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 enough hexane to make the volume to 1 mL. Load the extract in hexane onto an alumina column (6 g, 1% deactivated). Elute the column with hexane (15 mL, discard) followed by 1:1 dichloromethane:hexane (35 mL, retain). Concentrate the eluate to 1 mL by rotary evaporation and transfer to a centrifuge tube. Concentrate further under a stream of nitrogen and transfer to an autosampler vial. Adjust the volume to that specified for the fraction in Sections 6.1.

FINAL

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6.3 Acid Wash

CAUTION: The following cleanup procedure cannot be used if pesticide analysis is required.
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 extract, with rinses to a 125 mL separatory funnel. Add concentrated sulphuric acid (30 mL) to the extract and shake for 2 minutes. Let stand (no more than 13 minutes). If the aqueous layer is strongly coloured repeat the procedure up to 3 more washes with H_2SO_4.)

Wash the extract by shaking with Seastar water. Transfer the organic layer to a clean 250 mL Erlenmeyer flask and dry over anhydrous sodium sulphate (5-10 g, 10-15 minutes). The extract is ready for clean up by column chromatography clean-up procedures. (Section 6.0).

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7.0 INSTRUMENTAL ANALYSIS

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

Sensitivity

The sensitivity of the GC/MS is checked at least weekly and after any instrument maintenance.

- Signal-to-noise ratios must be greater than or equal to 3:1 for 10 pg of pp'-DDT, HCB and PCB 118 and 20 pg of oxychlordane injected.

GC Resolution

The GC resolution is checked daily. The resolution must satisfy the following criteria.

- The maximum peak width at half height for pp'-DDT is five seconds; there must be baseline separation of p,p'-DDD and o,p-DDT.
- The valley height must be ≤80% of the smallest peak height of the PCB 31/28 pair.
- The PCB 209 peak must be symmetrical with negligible tailing and with a peak width not exceeding 20 seconds.
- The percent breakdown of DDT is checked every 12 hours and must be less than 15%. The breakdown is monitored by checking the breakdown of ^{13}C-labelled DDT in the solvent blank. The % breakdown is calculated as follows:

$$\%\,\text{Breakdown} = 100 \times \frac{(\text{sum of degradation peak area (DDD + DDE)})}{(\text{sum of all peak areas (DDT + DDE + DDD)})}$$

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

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 labelled 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 RRF's determined from the initial calibration to calculate the analyte concentrations. Bracketing calibration procedures use mean RRF's from the analysis of the mid-level calibration solution to calculate analyte concentrations.

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- The relative standard deviation of the RRF's must be ≤20% over the calibration range except for $^{13}C_{12}$-p,p'-DDT which must be ≤25%.
- Continuing CAL VER: RRF's from opening and closing calibration standards must be within ±20% of the mean RRF's from the initial calibration, except for $^{13}C_{12}$-p,p'-DDT which must be within ±25%.
- Bracketing Cal: RRF's from the mid-point calibration standard must agree to ±20% over a 12-hour period.

Analysis

High resolution gas chromatography/low resolution mass spectrometry (HRGC/LRMS) analysis of the E1 fraction is done on a Finnigan Incos 50 mass spectrometer (MS) equipped with a Varian 3400 gas chromatograph, a CTC auto-sampler and a Prolab/Enviroquant data system for MS control and data acquisition or on an Agilent 6890N GC equipped with an Agilent 5973 MS, an Agilent 7683 Series Autosampler, and an HP Chemstation. 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 4.

A typical extract volume is 100 µL; 1.0 - 1.2 µL is injected. If necessary, extracts are diluted 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)	1.1	Injector	Splitless, 2 min
Rate 9 (C° min^{-1})	11.5	Carrier Gas	Helium, 200 kPa
Temp (°C)	150	Maximum Temp (°C)	325
Hold time (min)	0		
Rate (C° min^{-1})	3	**MS Conditions**	
Temp (°C)	250	Source Temp (°C)	180 (Finnigan) 230 (Agilent)
Hold time (min)	0	Electron Energy (eV)	35 (Finnigan) 70 (Agilent)
Rate (C° min^{-1})	50	Trap Current (µA)	NA
Temp (°C)	320	Mass Resolution	NA
Hold time (min)	11	Detector Voltage (V)	Variable

The Instrumental Analytical Chemist is referred to Instrument Method CL-QMS-1.3, documented on the QUAD-MS (Prolab) Method Summary Form, for further details of the instrumental analysis.

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Analysis Sequence

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

Calibration Sample
Reference Sample
Toluene Blank (containing $^{13}C_{12}$-DDT)
Procedural Blank
Samples
Calibration Sample

Where greater sensitivity or higher mass resolution (to resolve interferences) is required, the analysis of pesticides and PCB congeners may be conducted by two separate HRGC/HRMS runs. The Instrumental Analytical Chemist is referred to Instrument Methods below, documented on HR-GCMS Method Summary forms, for further details of the instrumental analysis.

Analysis	**Instrument Method Number**
Pesticides	CL-HMS #1.01 (continuing calibration) CL-HMS #2.01 (bracketing calibration)
Pesticides & Chlorobenzenes	CL-HMS #5.01
Congeners	PB-HMS #3.01 (bracketing calibration, DB-5) PB-HMS #4.01 (continuing calibration, DB-5)

7.2 GC/ECD Analysis of E2 for Pesticides

Sensitivity

The sensitivity of the GC/MS is checked at least weekly and after any instrument maintenance. The following sensitivity requirements must be met.

- Signal-to-noise ratios must be greater than or equal to 3:1 for 50 pg of methoxychlor injected.

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GC Resolution

The resolution of the GC is checked daily when samples are being analyzed. The following criteria must be satisfied.

- The valley height between d_4-I-endosulphan and I-endosulphan must be ≤50% of the d_4-I-endosulphan peak height or the separation of d_4-I-endosulphan and I-endosulphan must be ≥0.15 minutes.
- The breakdown of endrin is checked every 12 hours and must be less than 20%. The percent breakdown is determined from the analysis of the solution which contains only endrin and is calculated as follows:

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

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

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 labelled surrogates, recovery standards and target compounds. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution. The RRF of delta-HCH is determined from the analysis of a separate mid-level calibration solution every 12 hours.

- The relative standard deviation of the RRF's must be ≤ 20% over the calibration range.
- The RRF's from the mid-point calibration standard must agree to ±20% over a 12-hour period.

Analysis

The E2 fraction is analyzed by gas chromatography/electron capture detection (GC/ECD) analysis, using a Hewlett Packard (HP) 5890 Series II or 5890 gas chromatograph, an HP 7673A automatic injector, a ^{63}Ni electron capture detector and an HP Chemstation. 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.

The Instrumental Analytical Chemist is referred to Instrument Method CL-ECD #3.02, documented on an ECD Method Summary Form, for further details of the instrumental analysis.

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Analysis Sequence

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

Endrin Breakdown Solution
Calibration Solution
Delta-HCH Calibration Solution (if required)
Reference Sample
Solvent Blank
Procedural Blank
Samples
Calibration Sample
Delta-HCH Calibration Solution (if required)

Where greater sensitivity or higher mass resolution (to resolve interferences) is required, the analysis may be conducted by HRGC/HRMS. The Instrumental Analytical Chemist is referred to Instrument Methods below, documented on HR-GCMS Method Summary forms, for further details of the instrumental analysis.

Analysis	**Instrument Method Number**
E2 Pesticides	CL-HMS #3.01 (continuing calibration) CL-HMS #4.01 (bracketing calibration)

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8.0 ANALYTE IDENTIFICATION

HRGC/LRMS

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.
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.
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.

GC/ECD

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.

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9.0 QUANTIFICATION PROCEDURES

9.1 Concentrations

Target concentrations are determined with respect to either a labelled surrogate or the internal standard as shown in Tables 3 and 4. 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:

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

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

Those compounds quantified against a labelled standard added at the beginning of the analysis procedure are recovery corrected by the method of quantification. Analytes that are quantified against a recovery standard are not recovery corrected. Surrogate recoveries are determined similarly against the recovery (internal) standard and are used as general indicators of overall analytical quality.

9.2 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 1/3/8 concentrations multiplied by 1.9;
Aroclor 1232 = the sum of PCB 1/3/18 concentrations multiplied by 3.4;
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 15;
Aroclor 1260 = the sum of PCB 183/180/170 concentrations multiplied by 7.1;

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

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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 as in Section 9.0 using the summed area responses of 3 – 10 congeners for each Aroclor identified.

9.3 Quantification of Delta-HCH

The target analyte, delta-HCH, is present in both the E1 and E2 fractions. It must be quantified in both fractions and summed to determine the concentration in the extract. To quantify this analyte, the RRF of delta-HCH must be determined from calibration procedures in both analyses. For the E1 analysis, delta-HCH is present in the calibration solutions. In the E2 analysis an additional calibration solution is analyzed to determine the RRF.

9.4 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.

9.5 Detection Limits

Sample specific detection limits (SDL's) 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 labelled surrogate peak.

9.6 Optional High Concentration Sample Procedure

For samples with target concentrations that would exceed the working range of the method an extraction standard (tetra-chloro-m-xylene (TCMX) and PCB 204) is added to the sample prior to analysis instead of the labelled surrogates. The labelled 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.

9.7 Optional Low Concentration Sample Procedure

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

9.8 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 corrections 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.

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Table 4: Analyte Ions Monitored, Surrogates Used, and RRF Determination for E1 Pesticides and PCB Congeners by LR GC/MS

(Target compounds are listed below the labelled 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
$^{13}C_{12}$-PCB 52 –RS[2]	**26.6**	**302**	**304**	**130**	
$^{13}C_6$-1,4-DiCl-Benzene	**8.43**	**154**	**152**	**156**	
1,3-DiCl-Benzene	8.37	146	148	64	
1,4-DiCl-Benzene	8.43	146	148	64	
1,2-DiCl-Benzene	8.72	146	148	64	
$^{13}C_6$-1,2,3-TriCl-Benzene	**11.08**	**190**	**188**	**312**	
1,3,5-TriCl-Benzene	9.93	180	182	96	
1,2,4-TriCl-Benzene	10.55	180	182	96	
1,2,3-TriCl-Benzene	11.08	180	182	96	
$^{13}C_6$-1,2,3,4-TetraCl-Benzene	**13.58**	**224**	**222**	**208**	
1235/1245-TetraCl-Benzene	12.8	216	214	78	
1,2,3,4-TetraCl-Benzene	13.58	216	214	78	
Hexachlorobutadiene	11.08	225	260	38	1,2,3,4-TetraCl-Benzene
$^{13}C_6$-PentaCl-Benzene	**16.2**	**256**	**260**	**20**	
PentaCl-Benzene	16.2	250	252	64	
$^{13}C_6$-HexaCl-Benzene	**20.5**	**292**	**294**	**43**	
HexaCl-Benzene	20.52	284	286	80	
$^{13}C_6$-beta BHC	**21.43**	**225**	**227**	**48**	
beta-HCH	21.45	219	217	78	
$^{13}C_6$-gamma BHC	**21.72**	**225**	**227**	**48**	
alpha-HCH	20.13	219	217	78	
gamma-HCH	21.73	219	217	78	
delta-HCH	22.92	219	217	78	
$^{13}C_{10}$-Heptachlor	**25.93**	**277**	**279**	**80**	
Heptachlor	25.95	272	270	50	
$^{13}C_{12}$-Aldrin	**27.83**	**270**	**272**	**64**	
Aldrin	27.83	263	261	63	
$^{13}C_{10}$-trans-Chlordane	**31.48**	**383**	**385**	**96**	
trans-Chlordane	31.40	373	375	96	
cis-Chlordane	31.97	373	375	96	
$^{13}C_{10}$-trans-Nonachlor	**32.14**	**419**	**421**	**64**	
trans-Nonachlor	32.16	409	411	64	

[2] RS = Recovery Standard, added just prior to instrument analysis.

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
cis-Nonachlor	35.94	409	411	64	
$^{13}C_{12}$-pp'-DDE	**33.33**	**330**	**328**	**78**	
op'-DDE	31.48	246	248	64	
pp'-DDE	33.35	246	248	64	
$^{13}C_{12}$-PCB 138 -RS	**38.22**	**372**	**376**	**34**	
$^{13}C_{12}$-pp'-DDT	**38.02**	**247**	**249**	**64**	
op'-DDD	33.82	235	237	64	
pp'-DDD	35.82	235	237	64	
op'-DDT	36	235	237	65	
pp'-DDT	38.04	235	237	64	
$^{13}C_{12}$-PCB 118	**35.29**	**338**	**340**	**64**	
Octachlorostyrene	29.54	380	378	89	
Oxychlordane	29.79	185	115	empirical from CAL STD	
Mirex	43.61	272	270	52	
$^{13}C_{12}$-PCB 52 -RS	**26.6**	**302**	**304**	**130**	
$^{13}C_{12}$-PCB 3	**17.32**	**200**	**202**	**32**	
PCB 1	15.72	188	190	32	
PCB 2	17.17	188	190	32	PCB 3
PCB 3	17.32	188	190	32	
$^{13}C_{12}$-PCB 15	**22.33**	**234**	**236**	**64**	
PCB 4/10	18.23	222	224	64	
PCB 7/9	19.35	222	224	64	PCB 4/10
PCB 6	19.85	222	224	64	PCB 4/10
PCB 8/5	20.13	222	224	64	PCB 4/10
PCB 14	20.8	222	224	64	PCB 15
PCB 11	21.77	222	224	64	PCB 15
PCB 12/13	22.07	222	224	64	PCB 15
PCB 15	22.35	222	224	64	
TCMX –EX[3]	**18.27**	**244**	**242**	**76**	
$^{13}C_{12}$-PCB 37	**27.97**	**268**	**270**	**96**	
PCB 19	21.1	256	258	96	
PCB 30	21.53	256	258	96	PCB 19
PCB 18	22.23	256	258	96	
PCB 17	22.35	256	258	96	PCB 18
PCB 24/27	22.78	256	258	96	PCB 18

[3] EX = Extraction Standard

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (CF/Qt)	RRF Used
PCB 16/32	23.22	256	258	96	PCB 18
PCB 34/23	23.72	256	258	96	PCB 31
PCB 29	23.93	256	258	96	PCB 31
PCB 26	24.18	256	258	96	PCB 31
PCB 25	24.32	256	258	96	PCB 31
PCB 31	24.62	256	258	96	
PCB 28	24.69	256	258	96	
PCB 33/20/21	25.22	256	258	96	PCB 31
PCB 22	25.64	256	258	96	PCB 31
PCB 36	25.94	256	258	96	PCB 31
PCB 39	26.42	256	258	96	PCB 31
PCB 38	26.97	256	258	96	PCB 37
PCB 35	27.53	256	258	96	PCB 37
PCB 37	27.99	256	258	96	
$^{13}C_{12}$ PCB 31- FS[4]	**24.62**	**270**	**272**	**33**	PCB 31
$^{13}C_{12}$-PCB 54	**23.95**	**302**	**304**	**130**	
PCB 54	23.93	290	292	130	
PCB 50	24.62	290	292	130	PCB 54
PCB 53	25.28	290	292	130	PCB 52/73
PCB 51	25.53	290	292	130	PCB 52/73
PCB 45	25.91	290	292	130	PCB 52/73
PCB 46	26.36	290	292	130	PCB 52/73
PCB 69	26.51	290	292	130	PCB 52/73
PCB 52/73	26.63	290	292	130	
PCB 49/43	26.86	290	292	130	
PCB 47/48/75	27.05	290	292	130	PCB 52/73
PCB 65/62	27.24	290	292	130	PCB 52/73
PCB 44	27.76	290	292	130	
PCB 42/59	27.89	290	292	130	PCB 44
PCB 72	28.25	290	292	130	PCB 44
PCB 41/71/64/68	28.47	290	292	130	PCB 44
PCB 40	28.91	290	292	130	
PCB 57	29.02	290	292	130	PCB 40
PCB 67	29.27	290	292	130	PCB 40
PCB 58	29.44	290	292	130	PCB 40

[4] FS = Field Standard

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 63	29.58	290	292	130	PCB 40
PCB 74/61	29.83	290	292	130	PCB 66/80
PCB 70/76	30.02	290	292	130	PCB 66/80
PCB 66/80	30.22	290	292	130	
PCB 55	30.73	290	292	130	PCB 56/60
PCB 56/60	31.2	290	292	130	
PCB 79	31.99	290	292	130	PCB 56/60
PCB 78	32.59	290	292	130	PCB 56/60
PCB 81	33.16	290	292	130	PCB 56/60
PCB 77	33.73	290	292	130	PCB 56/60
$^{13}C_{12}$ PCB 138 -RS	**38.22**	**372**	**376**	**34**	
$^{13}C_{12}$ PCB 118	**35.29**	**338**	**340**	**64**	
PCB 104	27.51	326	328	65	
PCB 96	28.71	326	328	65	PCB 95/93
PCB 103	28.94	326	328	65	PCB 95/93
PCB 100	29.27	326	328	65	PCB 95/93
PCB 94	29.73	326	328	65	PCB 95/93
PCB 98/102	30.13	326	328	65	PCB 95/93
PCB 95/93	30.26	326	328	65	
PCB 88/121	30.43	326	328	65	PCB 95/93
PCB 91	30.64	326	328	65	PCB 95/93
PCB 92	31.31	326	328	65	PCB 90/101/89
PCB 84	31.48	326	328	65	PCB 90/101/89
PCB 90/101/89	31.63	326	328	65	
PCB 113	31.82	326	328	65	PCB 90/101/89
PCB 99	31.92	326	328	65	
PCB 119	32.31	326	328	65	PCB 87/115/116
PCB 112	32.44	326	328	65	PCB 87/115/116
PCB 83/108	32.57	326	328	65	PCB 87/115/116
PCB 97/86	32.86	326	328	65	PCB 87/115/116
PCB 125	33.01	326	328	65	PCB 87/115/116
PCB 111/117	33.1	326	328	65	PCB 87/115/116
PCB 87/115/116	33.23	326	328	65	
PCB 85/120	33.42	326	328	65	PCB 87/115/116
PCB 110	33.75	326	328	65	
PCB 82	34.4	326	328	65	PCB 110
PCB 124	34.8	326	328	65	PCB 110
PCB 107/109	35.01	326	328	65	PCB 118/106

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 123	35.16	326	328	65	PCB 118/106
PCB 118/106	35.3	326	328	65	
PCB 114	35.94	326	328	65	
PCB 122	36.09	326	328	65	PCB 118/106
PCB 105/127	36.86	326	328	65	
PCB 126	38.79	326	328	65	
$^{13}C_{12}$ PCB 95 FS	**30.24**	**338**	**340**	**64**	**PCB 95/93**
$^{13}C_{12}$-PCB 167	**39.94**	**372**	**376**	**34**	
PCB 155	31.07	360	362	81	
PCB 150	32.33	360	362	81	PCB 151
PCB 152	32.78	360	362	81	PCB 151
PCB 145	33.16	360	362	81	PCB 151
PCB 148	33.39	360	362	81	PCB 151
PCB 136	33.58	360	362	81	PCB 151
PCB 154	33.84	360	362	81	PCB 151
PCB 151	34.5	360	362	81	
PCB 144/135	34.76	360	362	81	PCB 149/139
PCB 147	34.96	360	362	81	PCB 149/139
PCB 149/139	35.2	360	362	81	
PCB 140	35.38	360	362	81	PCB 149/139
PCB 134/143	35.72	360	362	81	PCB 149/139
PCB 133	35.94	360	362	81	PCB 149/139
PCB 131/142	36.09	360	362	81	PCB 149/139
PCB 165	36.2	360	362	81	PCB 153
PCB 146	36.31	360	362	81	PCB 153
PCB 161	36.4	360	362	81	PCB 153
PCB 153	36.66	360	362	81	
PCB 132/168	36.81	360	362	81	PCB 153
PCB 141	37.4	360	362	81	PCB 138/163/164
PCB 137	37.8	360	362	81	PCB 138/163/164
PCB 130	37.95	360	362	81	PCB 138/163/164
PCB 138/163/164	38.24	360	362	81	
PCB 158/160	38.41	360	362	81	PCB 138/163/164
PCB 129	38.74	360	362	81	PCB 138/163/164
PCB 166	39.12	360	362	81	PCB 138/163/164
PCB 159	39.29	360	362	81	PCB 138/163/164
PCB 162	39.6	360	362	81	PCB 138/163/164
PCB 128	39.86	360	362	81	PCB 138/163/164

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
PCB 167	39.96	360	362	81	
PCB 156	41.27	360	362	81	
PCB 157	41.61	360	362	81	
PCB 169	43.46	360	362	81	
$^{13}C_{12}$ PCB 180	**42.28**	**406**	**408**	**95**	
PCB 188	36.26	394	396	95	
PCB 184	36.64	394	396	95	PCB 188
PCB 179	37.49	394	396	95	PCB 188
PCB 176	37.91	394	396	95	PCB 188
PCB 186	38.42	394	396	95	PCB 187/182
PCB 178	38.79	394	396	95	PCB 187/182
PCB 175	39.12	394	396	95	PCB 187/182
PCB 187/182	39.29	394	396	95	
PCB 183	39.58	394	396	95	
PCB 185	40.19	394	396	95	PCB 183
PCB 174/181	40.64	394	396	95	PCB 183
PCB 177	40.95	394	396	95	PCB 183
PCB 171	41.21	394	396	95	PCB 180
PCB 173	41.56	394	396	95	PCB 180
PCB 172/192	41.94	394	396	95	PCB 180
PCB 180	42.28	394	396	95	
PCB 193	42.45	394	396	95	PCB 180
PCB 191	42.68	394	396	95	PCB 180
PCB 170/190	43.9	394	396	95	
PCB 189	44.95	394	396	95	
$^{13}C_{12}$-PCB 153- FS	**36.64**	**372**	**376**	**34**	**PCB 153**
$^{13}C_{12}$-PCB 202	**41.1**	**440**	**442**	**112**	
PCB 202	41.16	428	430	112	
PCB 201	41.63	428	430	112	PCB196/203
PCB 204 –EX	**41.73**	**428**	**430**	**112**	PCB196/203
PCB 197	42.07	428	430	112	PCB196/203
PCB 200	42.97	428	430	112	PCB 196/203
PCB 198	44.09	428	430	112	PCB 196/203
PCB 199	44.24	428	430	112	PCB 196/203
PCB 196/203	44.43	428	430	112	
PCB 195	45.44	428	430	112	PCB 194
PCB 194	46.07	428	430	112	
PCB 205	46.24	428	430	112	

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Table 4 Cont'd./...

Compound Name	Typical Retention Time (minutes)	Quantification Ion Mass (Qt)	Confirmation Ion Mass (Cf)	Expected Ion Ratio % (Cf/Qt)	RRF Used
$^{13}C_{12}$-PCB 206	**47.15**	**474**	**476**	**128**	
PCB 208	45.4	462	464	128	
PCB 207	45.67	462	464	128	PCB 208
PCB 206	47.17	462	464	128	
^{13}C-PCB 209	**47.99**	**512**	**510**	**117**	
PCB 209	47.99	500	498	117	

Table 5: Surrogates Used and RRF Determination for E2 Pesticides by GC/ECD

ANALYTE	Surrogate	RRF Determined From
E2 Fraction		
delta-HCH	$^{13}C_{12}$-PCB 153	delta-HCH
Heptachlor Epoxide	$^{13}C_{12}$-PCB 153	Heptachlor Epoxide
alpha-Endosulphan	d4-alpha-Endosulphan	alpha-Endosulphan
Dieldrin	$^{13}C_{12}$-PCB 153	Dieldrin
Endrin	$^{13}C_{12}$-PCB 153	Endrin
beta-Endosulphan	d4-beta-Endosulphan	beta-Endosulphan
Endrin Aldehyde	$^{13}C_{12}$-PCB 153	Endrin Aldehyde
Endosulphan Sulphate	d4-beta-Endosulphan	Endosulphan Sulphate
Endrin Ketone	$^{13}C_{12}$-PCB 153	Endrin Ketone
Methoxychlor	$^{13}C_{12}$-PCB 153	Methoxychlor
LABELLED SURROGATES	***Recovery Calculated Against***	
d4-alpha-Endosulphan	$^{13}C_{12}$-PCB 153	
d4-beta-Endosulphan	$^{13}C_{12}$-PCB 153	
RECOVERY STANDARD		
$^{13}C_{12}$-PCB 153		

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APPENDIX A

ECNI GC/MS ANALYSIS OF TOXAPHENE

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A ANALYSIS OF TOXAPHENE BY ECNI GC/MS

The concentration of toxaphene in the E1 extract may be determined by an additional analysis of the extract by electron capture negative ion (ECNI) GC/MS. This may be carried out by either low resolution or high resolution GC/MS. No additional surrogate or recovery standards are required to carry out the analysis. The authentic standard solution named below is used to prepare the spiked matrix sample. The calibration standard named below is used for calibration of the GC/MS. Details of the concentration and composition of these standards are documented on Working Standard Data sheets maintained by the Technical Specialist.

Typical Calibration Standard	Typical Authentic Standard
TP019D-CAL (Total)	TP005A-AUT 1 mL

A.1 INSTRUMENTAL ANALYSIS

Sensitivity

The signal-to-noise ratio must be greater than or equal to 2:1 for 3 ng of technical toxaphene injected and a minimum of five marker peaks must be detected.

Calibration

The linearity of the GC/MS is established using a series of solutions that encompass the working concentration range of the instrument. The solutions are prepared from technical toxaphene, the surrogate standard, and the recovery standard. The percent relative standard deviation (%RSD) of the relative response factor (RRF) determined from the calibration solutions must be ≤20%.

A mid-level calibration solution is analyzed every twelve hours, at the beginning and end of each analysis batch. The RRF's from the beginning and end of the batch must agree to within 20%.

LR GC/MS Analysis

The E1 fraction is analyzed for toxaphene using a Finnigan INCOS 50 mass spectrometer (MS) equipped with a Varian 3400 GC, a CTC autosampler and a data system running HP EnviroQuant and Prolab MS-Extend software for targeting and quantification. The instrument is operated in the electron capture negative ion (ECNI) mode using methane as the reagent gas. The MS is operated at unit mass resolution in the MID mode acquiring two characteristic ions for each target analyte and surrogate standard. The ions monitored are presented in the following table.

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Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 i.d., 0.10 µm film thickness). A splitless/split injection sequence is used.

The Instrumental Analytical Chemist is referred to Instrument Method TP-QMS #1.01, documented on a QUAD-MS (Prolab) Method Summary form, for further details of the instrumental analysis.

Ions Monitored for Toxaphene Analysis

Number of Toxaphene Peaks	Number of Chlorine Atoms	Quantification Ion (m/z)	Confirmation Ion (m/z)
6	7	343	341
8	8	377	375
3	9	413	411
2	10	449	447
Surrogate Std			
^{13}C-PCB 180	7	406	408
Recovery Std			
^{13}C-PCB 138	6	372	374

Where greater sensitivity or higher mass resolution (to resolve interferences) is required the analysis may be conducted by HRGC/HRMS. The Instrumental Analytical Chemist is referred to Instrument Method TP-HMS #1.01, documented on a HR-GCMS Method Summary form, for further details of the instrumental analysis.

A.2 ANALYTE IDENTIFICATION

A peak is identified as a toxaphene component only when the following criteria are met:

1. The peak must occur within 2 seconds of the expected retention time observed for the calibration standard.
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.

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A.3 QUANTIFICATION PROCEDURES

The concentration of toxaphene is calculated using the internal standard method of quantification as described in Section 9.0.

Total Toxaphene

Total toxaphene is quantified as the sum of nineteen individual peaks in the chromatogram, where the peak area used in the calculation is the summed area of 19 characteristic marker peaks in the sample chromatogram. The RRF is calculated using the summed peak areas of the nineteen peaks in the calibration standard.

The sample specific detection limit (SDL), based on a single component in the m/z 413 channel, is determined by converting the minimum detectable signal to a concentration using the calculations described in Section 9.0. Typical detection limits are presented in Specification Table CL/04, Section 4.0.

The concentration of toxaphene is recovery corrected by the method of quantification.

Chlorobornanes

Individual chlorobornanes are quantified, as above, using the RRF's for the individual compounds determined from the calibration solution.

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.
- The percent recovery of the surrogate standards must be between 40 – 130%.
- The percent recovery of toxaphene in a spiked matrix sample must be between 60 – 130%.
- The difference between target concentrations for duplicate samples must be less than 40%.

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APPENDIX B

HIGH RESOLUTION GC/MS ANALYSIS OF COPLANAR PCBs

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B ANALYSIS OF COPLANAR PCBS BY HRGC/HRMS (ANALYSIS CODE N)

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 are presented in Table B1. The authentic standard solution named below is used to prepare the spiked matrix sample. The calibration standard named below is used for calibration of the GC/MS. Details of the concentration and composition of these standards are documented on Working Standard Data sheets maintained by the Technical Specialist.

Table B1: Typical Standard Solutions for Analysis of Coplanar PCBs

Surrogate Standard	Recovery Standard	Calibration Standard	Authentic Standard
CO004A-SUR 40 µL	CO002A-REC 20 µL*	CO005A-CAL	CO002A-AUT 200 µL

* If an extract is split, then use only 10 µL of the recovery standard.

B.1 EXTRACTION/CLEANUP PROCEDURES

Carry out the extraction procedure for the matrix as described in Section 5. Add an aliquot of the surrogate standard solution named above as an additional surrogate to the sample. Cleanup the extract on Florisil as described in Section 6.1. Quantitatively split the E1 extract from the Florisil column in half. Prepare one half for GC/MS analysis codes A, P, T, C, and CB. Apply the other half of the extract to the carbon/Celite column for cleanup as described below.

B.2 PREPARATION OF CHROMATOGRAPHIC CLEANUP COLUMNS

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

Prepare a 4.5% carbon/Celite 545 cleanup column using a 9" Pasteur pipette with the tip cut off. Insert a baked 934 A/H filter paper disk into the pipette to the taper. Pack the column with 4.5% (0.22 g) carbon AX-21/celite 545 mixture and top with another 934 A/H filter disk and clean silanized glass wool. Clean the column by elution with toluene (5 mL), ethylacetate:toluene (10:1, 2 mL), cyclohexane:dichloromethane (1:1, 2 mL) and hexane (5 mL).

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B.2.2 Alumina Column for Coplanar PCB Analysis

Dry 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 powdered sodium sulphate.

B.3 CHROMATOGRAPHIC CLEANUP PROCEDURES

Carbon Column

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

Load the 2 mL of extract in hexane 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 a 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'-HCB (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 labelled surrogates, recovery standards and target compounds. The relative standard deviation of the RRF's 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 RRF's from the mid-point calibration standard must agree to ±20% over a 12-hour period.

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Analysis

The cleaned up E1 fraction is analyzed for coplanar PCBs using a VG/Micromass 70SE or VG/Micromass Ultima high resolution mass spectrometer coupled to a Hewlett Packard 5890A GC, a CTC autosampler and a data system running OPUSQuan software. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm id and 0.10 µm film thickness). A splitless/split injection sequence is used.

The Instrumental Analytical Chemist is referred to Instrument Method CO-HMS #1.01, documented on an HR-GCMS Method Summary form, 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 Specification Table CL/08.

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SPECIFICATION TABLE CL/08: Analysis of Coplanar PCBs by GC/HRMS Method

ACCEPTABLE DETECTION LIMITS*										
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level pg	Acceptable Matrix Spike % Recovery	
Analyte:	pg/g	pg/L	pg/L	pg/g	pg/g	pg/g	pg/col		GC/MS	GC/HRM
3,3',4,4'-TCB (#77)	0.2	2	2	0.2	0.2	2	2	<3	70-130	70-130
3,3'4,4',5-PCB (#126)	0.2	2	2	0.2	0.2	2	2	<2	70-130	70-130
3,3'4,4'5,5'-HCB (#169)	0.2	2	2	0.2	0.2	2	2	<3	70-130	70-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 col			
Final Vol, TL	20	20	20	20	20	20	20			
Authentic Spike GCMS, pg	500	500	500	500	500	500	500			
Authentic Spike HRGCMS pg	50	50	50	50	50	50	50			

* Detection Limits by GC/HRMS are approximately 10 times lower.

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

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'-HCB injected.

INSTRUMENT LINEARITY: Linearity determined by at least a 5-point calibration over the range of 10 pg/TL to 1000 pg/TL of all coplanar PCBs with a rel. std. deviation of the RRF's = ±20%.

RELATIVE RESPONSE FACTOR: RRF's from calibration standards must agree to ±20% over a 12-hour period. RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's 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. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

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APPENDIX C

GC/ECD ANALYSIS OF PCBs AND PESTICIDES

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C ANALYSIS OF PESTICIDES AND PCBS BY GC/ECD (ANALYSIS CODES A, P, C, T)

C.1 STANDARD SOLUTIONS

The analysis of chlorinated pesticides and PCBs (as Aroclors and congeners) by GC/ECD requires the use of surrogate, recovery, authentic and calibration standards presented in Table C1. These solutions are used instead of the solutions for the GC/MS analysis. Details of the concentration and composition of these standards are documented on Working Standard Data Sheets, which are maintained by the Technical Specialist.

Table C1: Typical GC/ECD Analysis Standard Solutions

Code A,C,P	Calibration Standard	Surrogate Standard	Recovery Standard	Authentic Standard
F1	**CL024A-CAL**	CL025A-SUR 40 µL	CL009A-REC 20 µL	CL022A-AUT 1 mL
F2	**CL025A-CAL**	CL025A-SUR 40 µL	CL009A-REC 20 µL	CL022A-AUT 1 mL
F3	**CL032D-CAL**	CL025A-SUR 40 µL	CL013A-REC 40 µL	CL022A-AUT 1 mL and CL021A-AUT 50 µL

C.2 EXTRACTION PROCEDURES

Carry out the extraction procedures described in Section 5.0 for the sample matrix, replacing the surrogate solution with those surrogate standard solutions named in Table C1 above. Cleanup the extract according to the procedures below.

C.3 CHROMATOGRAPHIC CLEANUP PROCEDURES

Extracts are cleaned up on a Florisil column (preparation described in Section 3.5.1). The solvents used to elute the column are different that those used for GC/MS analysis of PCBs and pesticides. **Determine the cutpoints for the elution solvents on Florisil prior to commencing cleanup procedures.**

Quantitatively transfer the extract and hexane rinses (3 x 1 mL) to a Florisil column (8 g, 2.1% deactivated). Elute the column with hexane (F1) and collect the eluate in a round bottom flask. Elute the column with dichloromethane:hexane (15:85, F2) and collect the eluate in another round bottom flask. Elute the column with dichloromethane (F3) and collect the eluate in a third round bottom flask.

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Add an aliquot of the surrogate standard to F2 to allow quantification since the surrogate standards added at the beginning of the procedure elute into F1 and F3. Concentrate each fraction to 1 mL by rotary evaporation. Add activated copper to sediment, soil, particulate filter, water, effluent, and hexane extract F1 extracts to remove sulphur. Transfer each extract to a centrifuge tube with hexane and concentrate to 500 µL in a stream of nitrogen. Transfer each extract to an autosampler vial. Concentrate the extract to a small volume under a gentle stream of nitrogen. Rinse the centrifuge tube with hexane and add the rinse to the autosampler vial. Concentrate the extract to a small volume under a gentle stream of nitrogen. Repeat the rinse and concentration sequence twice more. Add an aliquot of the appropriate recovery standard, named in Table C1, to each extract. Adjust the final volume to 100 µL with hexane. Cap the autosampler vial. The sample extract is ready for analysis by GC/ECD.

F1 is analyzed by GC/ECD for PCBs as Aroclors and/or pesticides and/or PCB congeners.

F2 is analyzed by GC/ECD for moderately polar pesticides.

F3 is analyzed by GC/ECD for the most polar pesticides.

If coplanar PCBs (Analysis Code **N)** are required, The extract must be combined and split as follows:

Upon successful completion of the GC/ECD analysis of the F1 and F2 Florisil extracts, quantitatively split the F1 and F2 extracts in half. Archive one half of each fraction. Combine the remaining halves of F1 and F2 for NOS PCB analysis. Proceed to the separation and cleanup of NOS. (Appendix B)

C.4 GC/ECD ANALYSIS OF F1, F2 AND F3

The F1 and F2 fractions are analyzed for chlorinated pesticides and PCB congeners and the F3 fraction for the most polar pesticides by separate gas chromatography/electron capture (GC/ECD) analyses. The analyses are carried out using a Hewlett Packard (HP) 5890A Series II gas chromatograph equipped with an HP 7673A automatic injector, ^{63}Ni electron capture detector and an HP 3396A integrator. 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.

The Instrumental Analytical Chemist is referred to Instrument Methods below, documented on ECD Method Summary forms, for further details of the instrumental analysis.

Analysis	Instrument Method Number
F1,F2 Pesticides	CL-ECD#1.01
F3 Pesticides	CL-ECD#3.01

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C.5 QUANTIFICATION PROCEDURES

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.

Target analytes concentrations are determined by the internal standard method of quantification, as described in Section 9.0. Target concentrations are determined with respect to either a labelled surrogate or the internal standard as shown in Table C2. Mean relative response factors (RRF) are determined from either a multi-level initial calibration series or a mid-level calibration standard run at the beginning and end of the samples. Those compounds quantified against a labelled standard added at the beginning of the analysis procedure are recovery corrected by the method of quantification. Analytes that are quantified against a recovery standard are not recovery corrected.

The following pesticides are present in both the F1 and F2 fraction and the final concentration is determined by quantifying the analytes in both fractions and summing the two.

trans-Nonachlor
p,p'-DDT
o,p-DDE

Quantification of Delta-HCH

The target analyte, delta-HCH, is present in the F2 and F3 fractions, and to a very small extent in the F1 fraction. Thus it must be quantified in all fractions and summed to determine the concentration in the extract.

C.6 QA/QC 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. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data for F1 and F2 must meet the established acceptance criteria presented in Specification Tables CL/01, CL/02, CL/05 and CL/06 and the F3 QC data must meet the established acceptance criteria presented in Specification Table CL/03.

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Table C2: Surrogates Used and RRF Determination for Pesticides by GC/ECD

ANALYTE	Surrogate	RRF Determined From
	F1 Pesticides	
Hexachlorobenzene (HCB)	Tetrachloro-m-xylene	Hexachlorobenzene (HCB)
Heptachlor	^{13}C-PCB 209	Heptachlor
Aldrin	^{13}C-PCB 209	Aldrin
trans-nonachlor	^{13}C-PCB 209	trans-nonachlor
o,p-DDE	^{13}C-PCB 209	o,p-DDE
p,p-DDE	^{13}C-PCB 209	p,p-DDE
p,p-DDT	^{13}C-PCB 209	p,p-DDT
Mirex	^{13}C-PCB 209	Mirex
	F2 Pesticides	
alpha-HCH	Tetrachloro-m-xylene	alpha-HCH
beta-HCH	Tetrachloro-m-xylene	beta-HCH
gamma-HCH	Tetrachloro-m-xylene	gamma-HCH
delta-HCH	Tetrachloro-m-xylene	delta-HCH
Oxychlordane	^{13}C-PCB 209	Oxychlordane
cis-Chlordane	^{13}C-PCB 209	cis-chlordane
trans-chlordane	^{13}C-PCB 209	trans-chlordane
o,p-DDE	^{13}C-PCB 209	o,p-DDE
p,p-DDT	^{13}C-PCB 209	p,p-DDT
trans-nonachlor	^{13}C-PCB 209	trans-nonachlor
p,p'-DDD	^{13}C-PCB 209	p,p'-DDD
o,p-DDD	^{13}C-PCB 209	o,p-DDD
cis-nonachlor	^{13}C-PCB 209	cis-nonachlor
	F3 Pesticides	
Heptachlor Epoxide	^{13}C-PCB 153	Heptachlor Epoxide
delta-HCH	^{13}C-PCB 153	delta-HCH
alpha-Endosulphan	d4-alpha-Endosulphan	alpha-Endosulphan
Dieldrin	^{13}C-PCB 153	Dieldrin
Endrin	^{13}C-PCB 153	Endrin
beta-Endosulphan	d4-alpha-Endosulphan	beta-Endosulphan
Endrin Aldehyde	^{13}C-PCB 153	Endrin Aldehyde
Endosulphan Sulphate	d4-alpha-Endosulphan	Endosulphan Sulphate
Endrin Ketone	^{13}C-PCB 153	Endrin Ketone
Methoxychlor	^{13}C-PCB 153	Methoxychlor

LABELLED SURROGATES	Recovery Calculated Against
2,4,5,6-Tetrachloro-m-xylene	Dibromo-octafluorobiphenyl
^{13}C-PCB 209	PCB 204
d4-alpha-Endosulphan	^{13}C-PCB 153

RECOVERY STANDARDS
Dibromo-octafluorobiphenyl
PCB 204
^{13}C-PCB 153

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SPECIFICATION TABLE CL/01: Analysis of F-1 Chlorinated Pesticides by GC/ECD Method

ACCEPTABLE DETECTION LIMITS									
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/Col		
HCB	0.1	1	1	0.1	0.1	1	1	<1	60-130
Heptachlor	0.1	1	1	0.1	0.1	1	1	<1	60-130
Aldrin	0.1	1	1	0.1	0.1	1	1	<1	60-130
trans-Nonachlor	0.1	1	1	0.1	0.1	1	1	<1	60-130
o,p'-DDE	0.1	1	1	0.1	0.1	1	1	<1	60-130
p,p'-DDE	0.1	1	1	0.1	0.1	1	1	<1	60-130
p,p'-DDT	0.1	1	1	0.1	0.1	1	1	<1	60-130
Mirex	0.1	1	1	0.1	0.1	1	1	<1	60-130
Typical Sample Size:	10g	1L	1L	10g	10g	1g	1 column		
Final Vol, TL	100	100	100	100	100	100	100		
Matrix Spiking Level, ng	50	50	50	50	50	50	50		

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES**
Tetrachloro-m-xylene	40-130
$^{13}C_{12}$-PCB 209	40-130

ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)
PROCEDURAL BLANK:	1 ng or <5% of Analyte Value.
INSTRUMENT SENSITIVITY:	S/N ≥3:1 for 10 pg HCB and 10 pg pp-DDT
INSTRUMENT LINEARITY:	Linearity is determined by at least a 5-point calibration over the following ranges with a relative standard deviation of the RRF's ≤20%. 0.02 ng/TL - 5.0 ng/TL.
RELATIVE RESPONSE FACTOR:	RRF's from bracketing calibration standards must agree to ±25% over a 12-hour period. RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration.
CHROMATOGRAM QUALITY:	
Max Peak Width:	PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds.
Resolution:	Valley height ≤80% of smallest peak height of PCB 31/28 pair.
ANALYTE/SURROGATE RATIOS:	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

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SPECIFICATION TABLE CL/02: Analysis of F-2 Chlorinated Pesticides by GC/ECD Method

ACCEPTABLE DETECTION LIMITS									
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/Col		
alpha-HCH	0.1	1	1	0.1	0.1	1	1	<1	60-130
beta-HCH	0.1	1	1	0.1	0.1	1	1	<1	60-130
gamma-HCH	0.1	1	1	0.1	0.1	1	1	<1	60-130
delta-HCH	0.1	1	1	0.1	0.1	1	1	<1	60-130
Oxychlordane	0.1	1	1	0.1	0.1	1	1	<1	60-130
trans-Chlordane	0.1	1	1	0.1	0.1	1	1	<1	60-130
cis-Chlordane	0.1	1	1	0.1	0.1	1	1	<1	60-130
o,p'-DDE	0.1	1	1	0.1	0.1	1	1	<1	60-130
trans-Nonachlor	0.1	1	1	0.1	0.1	1	1	<1	60-130
o,p'-DDD	0.1	1	1	0.1	0.1	1	1	<1	60-130
p,p'-DDD	0.1	1	1	0.1	0.1	1	1	<1	60-130
cis-Nonachlor	0.1	1	1	0.1	0.1	1	1	<1	60-130
p,p'-DDT	0.1	1	1	0.1	0.1	1	1	<1	60-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 column		
Final Vol, TL	100	100	100	100	100	100	100		
Matrix Spiking Level ng	50	50	50	50	50	50	50		

SURROGATE STANDARD RECOVERIES:	**%RECOVERY RANGES**
Tetrachloro-m-xylene	40-120
$^{13}C_{12}$-PCB 209	40-120

ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean (applicable to concentrations >10 tin the DL)
PROCEDURAL BLANK:	<1 ng or <5% of analyte value
INSTRUMENT SENSITIVITY:	S/N ≥3:1 For 10 pg pp-DDT
INSTRUMENT LINEARITY:	Linearity is determined by at least a 5-point calibration over the following ranges with a relative standard deviation of the RRF's ≤20%. 0.02 ng/TL - 5.0 ng/TL.
RELATIVE RESPONSE FACTOR:	RRF's from calibration standards must agree to ±25% over a 12-hour period. RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration.
CHROMATOGRAM QUALITY: **Max Peak Width:** **Resolution**:	PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds. Baseline separation of p,p'-DDD, cis-Nonachlor.
ANALYTE/SURROGATE RATIOS:	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

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SPECIFICATION TABLE CL/03: Analysis: F-3 Chlorinated Pesticides by GC/ECD Method

ACCEPTABLE DETECTION LIMITS									
MATRIX-->	Ash Sludge Soil Sedmts	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD column	Procedural Blank Level	Acceptable Matrix Spike % Recovery[1]
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	Ng		
Heptachlor Epoxide	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
alpha-Endosulphan	0.1	1	1	0.1	0.1	1	1	<1 ng	70-130
Dieldrin	0.1	1	1	0.1	0.1	1	1	<1 ng	60-130
Endrin	0.1	1	1	0.1	0.1	1	1	<1 ng	70-130
Methoxychlor	0.5	5	5	0.5	0.5	5	5	<5	70-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 column		
Final Vol, TL	100	100	100	100	100	100	100		
Matrix Spiking Level A, ng	20	20	20	20	20	20	20		

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
d4-alpha endosulphan	40-130

ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean + MDL.
PROCEDURAL BLANK:	<1 ng or <5% of analyte value.
INSTRUMENT SENSITIVITY:	S/N ratio ≤3:1 for 50 pg methoxychlor.
INSTRUMENT LINEARITY:	Linearity is determined by at least a 5-point calibration over the following ranges with a relative standard deviation of the RRFs ≤20%. 0.02 ng/TL - 0.4 ng/TL
RELATIVE RESPONSE FACTOR:	RRFs from calibration standards must agree to ±25% over a 12-hour period.
CHROMATOGRAM QUALITY:	
Max Peak Width:	PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds.
Resolution:	Valley height between d_4-alpha endosulphan and alpha endosulphan must be <50% of d_4-alpha endosulphan peak height or the separation of d_4-alpha endosulphan and alpha endosulphan must be >0.15 minutes.
ANALYTE/SURROGATE RATIOS:	Ratios must be within the linear range of the instrument. Coders may use data from more than one chromatogram to get the ratios in linear range.

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

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SPECIFICATION TABLE CL/05: Analysis of F1 Aroclors by GC/ECD Method

ACCEPTABLE DETECTION LIMITS									
MATRIX–>	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Column	Procedural Blank Level	Acceptable Matrix Spike % Recovery[1]
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/col		
Aroclor 1242	2	20	20	2	2	20	20	<20	60-130
Aroclor 1254	1	10	10	1	1	10	10	<10	60-130
Aroclor 1260	1	10	10	1	1	10	10	<10	60-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1 g	1 col		
Final Vol, TL	100	100	100	100	100	100	100		
Authentic Spike Level, ng	500	500	500	500	500	500	500		

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES ALL MATRICES
Tetrachloro-m-xylene	40-120
$^{13}C_{12}$-PCB 209	40-120

ANALYSIS DUPLICATE:	Must agree to within ±20% of the mean (applicable to concentrations ≥10 times the DL)
PROCEDURAL BLANK:	<10 ng or <5% of analyte
INSTRUMENT SENSITIVITY:	S/N ratio ≥3:1 for 10 pg PCB 118.
INSTRUMENT LINEARITY:	Linearity is determined by at least a 5-point calibration over the following ranges with a relative standard deviation of the RRF's ≤ 20%. 0.1 ng/TL - 12 ng/TL
RELATIVE RESPONSE FACTOR:	RRF's from calibration standards must agree to ±20% over a 12-hour period. RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration.
CHROMATOGRAM QUALITY: **Max Peak Width:** **Resolution:**	PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds. Valley height ≤80% of smallest peak height of PCB 31/28 pair.
ANALYTE/SURROGATE RATIOS:	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

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

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SPECIFICATION TABLE CL/06: Analysis of F1 PCB Congeners by GC/ECD Method

ACCEPTABLE DETECTION LIMITS*									
MATRIX-->	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Col	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/col		
PCB 18	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 15/17	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 54	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 31	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 28	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 52	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 49	0.1	1.0	1.0	0.1	0.1	10	1	<1	60-130
PCB 44	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 40/103	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 61/94/74	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 66/80/95	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 121	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 56/60	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 90/101	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 86/97	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 87	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 77/54/110	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 151	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 135/144	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 149	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 118	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 143	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 114	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 132/153	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 105	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 141/179	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 137	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 138/158	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 129/126	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 187/182/159	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 183	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 128	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 185	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 202/171/156	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 173/201	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 180	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130

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Specification Table CL/06 Cont'd./...

ACCEPTABLE DETECTION LIMITS*									
MATRIX–>	Ash Sludge Soil Sediments	Effluent	Water	Tissue Foliage	Pulp	Oil	XAD Col	Procedural Blank Level ng	Acceptable Matrix Spike % Recovery
Analyte:	ng/g	ng/L	ng/L	ng/g	ng/g	ng/g	ng/col		
PCB 191	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 170	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 199	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 203/196	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 189	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 208/195	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 207	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 194	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 205	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
PCB 206	0.1	1.0	1.0	0.1	0.1	10	1	<1	70-130
Typical Sample Size:	10 g	1 L	1 L	10 g	10 g	1.0 g	1 col		
Final Vol, TL	100	100	100	100	100	100	100		
Authentic Spike (ng)	20	20	20	30	20	20	20		

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
tetrachloro-m-xylene	40-130
$^{13}C_{12}$-PCB 209	40-130

ANALYSIS DUPLICATE: Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)

PROCEDURAL BLANK: <1 ng or <5% of analyte value.

INSTRUMENT SENSITIVITY: S/N ratio ≥3:1 for 10 pg PCB 118

INSTRUMENT LINEARITY: Linearity is determined by at least a 5-point calibration over the following ranges with a relative standard deviation of the RRF's ≤20%, 0.02 ng/TL - 5.0 ng/TL.

RELATIVE RESPONSE FACTOR: RRF's from calibration standards must agree to ±20% over a 12-hour period.
RRF's from opening/closing calibration standards must be within ±20% of the mean RRF's from the initial calibration.

CHROMATOGRAM QUALITY:

Max Peak Width: PCB 209 peak must be symmetrical with negligible tailing. Peak width should not exceed approximately 20 seconds.

Resolution: Valley height ≤80% of smallest peak height of PCB 31/28 pair.

ANALYTE/SURROGATE RATIOS: Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.

SAMPLE