Burlington SOP BRL SOP-00415 / 4 **Burlington**

OC Pesticides by HRMS in Liquid, Solid and Tissue

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Insert Text

BRL SOP-00415 rev 4

Associated Documents

Document Control Documents

- Burlington WI BRL WI-00011 : Review and Validation of Analytical Data
- Burlington SOP BRL SOP-00003 : GPC Cleanup
- Burlington SOP BRL SOP-00010 : Extraction Organochlorine Pesticides from Liquids and Solids
- Burlington FCD BRL FCD-00052 : MDL Template for Multi-Analyte Tests - NELAC
- Campobello WI CAM WI-00001 : Control Charting
- Campobello WI CAM WI-00005 : Chemistry Method Validation
- Campobello WI CAM WI-00025 : Determining Method Detection Limits
- Campobello WI CAM WI-00121 : Maxxam Lab QM Supplement to Meet Requirements of DoD QSM Version 4.1
- Corporate WI Publish to All COR WI-00010 : Corporate Procedure for Nonconformances
- Corporate WI Publish to All COR WI-00011 : Corporate Procedure for Corrective and Preventative Actions
- Corporate WI Publish to All COR WI-00012 : Definitions
- Corporate WI Publish to All COR WI-00018 : Determination of Estimation of Analytical Uncertainty of Measurement

- Corporate WI Publish to All COR WI-00040 : Corporate Procedure for Policy Deviation Forms
- Corporate WI Publish to All COR WI-00044 : Manual Integration of Chromatographic Peaks
- Corporate WI Publish to All COR WI-00049 : Continuing Calibration Verification (CCV) Acceptance Criteria

Reason for Change

Date of Change July 14, 2009 **Version** 2
Section Changed 1.0, 3.5, 4.1.1, 6.2.2, 7.3.4.1, 7.3.4.2, 8.1, 8.3, 9.3, 10.2.2, Table 2, Table 4
Change Made 1.0 updated to current location
3.5 removed the list to eliminate redundancy
4.1.1 referenced to Associated Documents section
6.2.2 added DDT & Endrin Breakdown Solution 100ng/mL
7.3.4.1 + 7.3.4.2 added: DDT/Endrin Breakdown Check
8.1 added: DDT/Endrin Breakdown Check, and renumbered 8.1-8.10
8.3 change limits to +/-35% for ion abundance ratios
9.3 referenced to BRL WI-00011 to reflect current practice
10.2.2 referenced to the QSI Deviation policy Form to freflect current practice
Table 2 added reference to the EPA 1699 current ratios
Table 4 replaced p,p'-DDD-ring d8 with p,p'-DDD-ring 13C12

Date of Change July 15, 2009 **Version** 3
Section Changed Table 1
Change Made Table 1 replaced d8-pp-DDD with 13C12-pp-DDD

Date of Change August 5, 2010 **Version** 4
Section Changed 3.2, 3.3, 4.0, 8.5, 10.2, 10.6.1
Change Made 3.2+3.3 redefined correlation between MDL and detection limit, as well as Maxxam's RDL and LOQ, added performance of quarterly LOD and LOQ
4.0 updated reference section by listing reference methods used in this SOP
8.5 added method blank criteria & corrective actions for DoD related projects
10.2 updated to reflect current practice re: use of COR WI-00040, Corporate Procedure for Policy Deviation Forms
10.6.1. updated storage of records offsite to reflect current practice

Approval Status Table

Approver Source **Role for Approvers** **Parallel-Everybody** Segment

Approver	Action	Date	Comment
Edmond McNeil	Approve	August 5, 2010 3:22 PM GMT-4	
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DETERMINATION OF ORGANOCHLORINE PESTICIDES IN LIQUID, SOLID AND TISSUE SAMPLES BYHIGH-RESOLUTION GC/MS

1.0 LOCATION:

This procedure is performed in the HRMS Prep Lab and the HRMS Instrument Laboratory.

2.0 PURPOSE:

This method is used to measure the concentration of Organochlorine Pesticides, (OCs) in liquid, solid and tissue samples.

2.1 Principle of the Method: OCs are soxhlet extracted from solid samples with dichloromethane (DCM) and from tissue samples with 50:50 DCM/hexane. An optional Dean Stark extraction using toluene may be used for solids. Liquid-liquid extraction with DCM is used for aqueous samples. Following extraction, the samples are cleaned up and passed through a series of columns that remove, by reaction and/or selective adsorption, the bulk of the organic matrix co-extracted with the OCs. The resulting fraction is concentrated to a known volume for analysis. Qualitative/quantitative analysis for OCs is performed using separation by high-resolution capillary gas chromatography, (HRGC), and measurement by high resolution mass spectrometry (HRMS). OCs are identified by comparing gas chromatograph (GC) retention times and the ion abundance ratios of the m/zs with the corresponding values obtained for authentic standards.

The GCMS system is calibrated and the analyte concentration is determined using an isotope dilution technique. Quantitation is based on the use of internal standards and relative response factors (RRFs).

2.2 Nature of Samples: samples can be from many different sources including incinerator ashes, soils, sediments and effluents. The laboratory should be made aware of any past history involving the samples or the presence of potentially interfering substances at elevated levels (e.g. PCB). This can allow the preparation technician to take a smaller aliquot of sample so that internal standards are not diluted out during analysis.

2.3 Safety:

2.3.1 General Considerations: the toxicity or carcinogeneity of each compound or reagent used in this method has not been established precisely. Each compound should be treated as a potential health hazard and exposure should be reduced to the lowest possible level. On the basis of the available toxicological and physical properties of the OCs, only highly trained personnel thoroughly familiar with handling and cautionary procedures should handle these compounds, and who understand the associated risks. All work related to the analysis of OCs should be carried out within a specially designed laboratory.

3.0 SCOPE:

This is a HRGC/HRMS method for the determination of OCs at picogram to nanogram levels in liquid, solid, and tissue samples.

3.0.1 Chemical Abstracts No.: refer to Table 4 in the Appendix for a listing of the CAS No.

3.1 Linear Range: the method is calibrated over the following linear ranges of calibration solutions.

5.0 pg/µL to 2000 pg/µL

3.2 Detection Limit: Detection Limit is also referred to as Method Detection Limits (MDL): an MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. MDLs are determined by the procedure outlined in CAM WI-00005, Method Development, Revision and Validation.

Target MDLs are verified annually by spiking water/soil replicates at the CS2-CS3 levels. These samples are carried through the full extraction and cleanup procedure. MDLs are determined by multiplying the standard deviation of the results by the Student's t-Value for the number of replicates used. If Target Limits are not achieved, the exercise is repeated spiking at a lower level. An MDL study is used to determine if the target limits outlined in test methods have been achieved. These are maintained on file in the QSI MDL Module as well as at the instrument. To meet DOD

requirements, these limits are then used to determine spiking levels for the Limit of Detection (LOD) verification. Each matrix is spiked two to three times the target limit (derived above) for a single analyte test or one to four times the detection limit for a multi-analyte test.

NOTE: As per CAM WI-00121, section D.1.2.1, Maxxam HRMS lab schedules quarterly LOD verifications for each respective matrix to meet DOD requirements.

Estimated Detection Limit (EDL): EDLs are sample specific and are calculated on a case specific basis for all analytes. If a signal produced is not at least 2.5 times the average background level at the expected retention time for specific isomers, the area response for the noise is calculated as follows.

$$2.5 \times \text{peak intensity of background noise} \times \frac{\text{area of internal standard}}{\text{height of internal standard}}$$

This is done for either the quantitation ion or the confirmation ion. Using theoretical ratios for the specific degree of chlorination an area for the second ion is generated. These areas are then totaled and applied to the same calculation scheme as for "positive" hits. (see Section 9). EMPCs (Estimated Maximum Possible Concentrations) are calculated whenever peaks are detected that meet all criteria except for ratios. The area in this case is recalculated (factored up or down) to meet the classical ratio and the result is flagged as an EMPC (NDR, not detected because of ratios, for Canadian Method).

NOTE: Maxxam HRMS lab schedules quarterly LOD verifications. They are analyzed with the next batch of samples for respective matrix.

3.3 Reporting Detection Limit (RDL): Reporting Detection Limit is also referred to as Limit of Quantitation (LOQ), equivalent to the Low Level Standard.

NOTE: As per CAM WI-00121, section D.1.2.1, Maxxam HRMS lab schedules quarterly LOQ verifications for each respective matrix to meet DOD requirements.

3.4 Interferences: interferents co-extracted from the sample material will vary considerably with the matrix and the diversity of the site being sampled. OCs are often associated with other chlorinated organics that may potentially interfere with the analysis. These include polychlorinated biphenyls, polychlorinated dibenzodioxins and furans, polychlorinated hydroxy diphenyl ethers, polychlorinated benzylphenyl ethers, polychlorinated diphenyl ethers, polychlorinated naphthalenes, polychlorinated xanthenes, and polynuclear aromatics.

Often the compounds responsible for interferences may be present at concentration levels several orders of magnitude higher than any OCs that may be present. Cleanup procedures can be used to reduce or eliminate these interferences to the maximum extent practicable in order to ensure reliable quantitation of OCs at trace levels. Despite rigorous cleanup procedures, the possibility of matrix interference will still exist. If detection limits are seriously elevated by the excessive background, the sample extract will have to be re-processed using alternative cleanup techniques.

3.5 Definitions:

Refer to Corporate Definitions procedure: COR WI-00012

4.0 REFERENCES:

In December 2007 EPA released Method 1699, Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS. Method 1699 is performance-based method, which means that any modifications made to it must meet the method's section 9.1.2 requirements for its equivalency.

Maxxam's method meets EPA 1699 method performance and prior to the release of the reference method 1699 it has been developed in conjunction, and with the approval of the NYS Department of Environmental Conservation and is derived from the proposed NYSDEC Method HRMS-2.

4.1 Information Sources:

- EPA 1699, Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, December 2007.

- Draft Analytical Procedure for Organochlorine Pesticides by Isotope Dilution HRGC/HRMS, NYSDEC Method HRMS-2.
- Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples, Section 3A [required for cleaning glass components of the sampling train].
- For further information contact Gary McAlister or Roger Shigehara, Emission Measurement Branch (MD-19), Technical Support Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, USA 27711, telephone (919)-541-1062.
- Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS
- USEPA Method 1699: Pesticides in Water, Soil, Sediment, Biosolids and Tissue by HRGC/HRMS. Brian Englehart, Ph. D., Engineering & Analytical Support Branch, Engineering and Analysis Division (4303T), Office of Science and Technology, Office of Water, U.S.E.P.A, 1200 Pennsylvania Avenue NW, Washington, DC, 20460.
- EPA SW-846 3540C, Soxhlet Extraction, (SW-846 PROPOSED UPDATE III Revision 3, January 1995)
- EPA SW-846 3510C, Separatory Funnel Liquid-Liquid Extraction (SW-846 PROPOSED UPDATE III Revision 3, January 1995),
- EPA SW-846 3620C, Florisil Cleanup, Revision 3, February 2007.
- EPA SW-846 3640A, Gel-Permeation Cleanup, (SW-846 Update II, September 1994)

4.1.1 Other Standard Operating Procedures:
Refer to QSI section Associated Documents for links to related methods

4.3 Analytical Test Codes:

Other test codes may be created as necessary

Waters:	**Analysis:**
PESTOCHR-W	OCs In Water By HRMS

Solids:	**Analysis:**
PESTOCHR-S	OCs In Solids By HRMS

Tissues:	**Analysis:**
PESTOCH-TI	OCs In Tissues By HRMS

5.0 SAMPLE HANDLING AND PRESERVATION:

When feasible, samples should be maintained at 2-6 °C from the time of collection until the point of extraction. Due to the fragile nature of the containers, the samples should be well wrapped with protective packaging during shipment from the field site to the laboratory. Samples that exceed the hold time will be flagged on the Certificate of Analysis. Different hold times may be imposed by other regulating agencies or as determined by client specific projects. Project specific and regulatory requirements for preservation method and reporting criteria, (required detection limit, dry or wet weight basis), must be examined and made clear prior to sample analysis. **Note:** sample containers should have Teflon liners in the caps.

Matrix	Container	Min. Vol.	Time to Extract	Time to Analysis
Waters	4x1 L amber glass	800 mL	30 days	45 days
Soils/Sludge	100 mL amber glass	2x5 gram	30 days	45 days
Pulp	100 mL amber glass	2x15 gram	30 days	45 days
Biological	100 mL amber glass	2x20 gram	30 days	45 days

6.0 APPARATUS AND MATERIALS:

6.1 Reagent Preparation: all reagent preparation information , lot numbers, and supplier are documented in the reagent preparation logbook kept in the sample preparation laboratory. Equivalent quality reagents and standards may be purchased from other suppliers as appropriate

6.1.1 Solvents: hexane, dichloromethane (DCM), toluene, isooctane, , nonane, ethyl acetate, petroleum ether, Distilled in Glass quality from Fisher or Caledon

6.1.2 RODI Water: Reverse Osmosis Deionized Water, (RODI), >16MΩ purity, produced in house

6.1.3 Compressed Gases: UHP Grade nitrogen

6.2 Standard Preparations: all standards preparations , lot numbers, supplier information, pertinent dates are documented by the analyst in the Standard Preparation Log Book located in the instrumentation laboratory. Store stock standards as per manufacturer's directions.

After preparation, use a permanent fine-tip marker to mark a line at the liquid level. This allows monitoring loss due to evaporation. (loss is greatly reduced by using refrigerated storage)

6.2.1 Calibration Standards: refer to Appendix: Table 3 for the volumes of the stock standards to use for preparing calibration standards. Current suppliers and lot numbers are in the Standards Prep Logbook in the Instrument Lab. Stock (neat) standards are stored in the freezer compartment of the standards fridge.

6.2.1.1 Native Intermediate (20 ng/uL): add the volumes of stocks to a 5 mL volumetric flask and dilute to volume with nonane.

6.2.1.2 Native Dilution (0.2 ng/uL): dilute10 uL of the native intermediate to 1 mL with nonane

6.2.1.3 Matrix Spiking Solution (5 ng/uL): add 1250 uL of the native intermediate to a 5 mL volumetric flask and dilute to volume with nonane

6.2.2 Method Spiking or Internal Standard Solution (4 ng/uL): a 10 mL nonane working level internal standard solution, is prepared once every two years or as required containing isotopically labeled OCs stock solutions. The volume of stock standards used is recorded in the Standard Preparation Log Book, located in the Instrument laboratory. Manufacturer Certified Stock standards are purchased from Cambridge Isotope Laboratories, Andover, MA, USA -

Component	Stock Conc. (ng/µL)	Final Conc. (pg/µL)	Vol. Added (µL)
Aldrin $^{13}C_{12}$	100	4000	400
BHC-beta $^{13}C_6$	50	4000	800
Chlordane $^{13}C_{10}$	100	4000	400
cis-Nonachlor $^{13}C_{10}$	100	4000	400
Dieldrin $^{13}C_{12}$	100	4000	400
Endrin $^{13}C_{12}$	100	4000	400
Heptachlor $^{13}C_{10}$	100	4000	400
Heptachlor Epoxide $^{13}C_{10}$	100	4000	400
Lindane $^{13}C_6$ d_6	100	4000	400
Methoxychlor-ring $^{13}C_{12}$	100	4000	400
Mirex $^{13}C_{10}$	100	4000	400
Oxychlordane $^{13}C_{10}$	100	4000	400
p,p'-DDD-ring $^{13}C_{10}$	100	4000	400
p,p'-DDE-ring $^{13}C_{12}$	100	4000	400
p,p'-DDT-ring $^{13}C_{12}$	100	4000	400
trans-Nonachlor $^{13}C_{10}$	100	4000	400
Endosulfan-I-$^{13}C_9$	100	4000	400
Endosulfan-II-$^{13}C_9$	100	4000	400
HXB $^{13}C_6$	100	4000	400
Endrin Ketone $^{13}C_{12}$**	50	4000	800
Endrin Aldehyde $^{13}C_{12}$**	50	4000	800

** Obtained in solid form and reconstituted as per manufacturers instructions. The stability of these compounds in solution is unknown and not certified and they have been included on a trial basis

Recovery or Injection Standard (5 ng/uL) a 5 mL final volume, nonane working level solution, is prepared once every two years or as required and are purchased from Cambridge Isotope Laboratories, Andover, MA, USA , or Wellington Labs, Guelph, Ontario, Canada.
The volumes of stock standards used for the current set of working level standards in use are documented in the Standard Preparation Log Book, located in the Instrument laboratory.

DDT and ENDRIN Breakdown Check Solution (100 ng/mL) a 10 mL final volume, nonane working level solution, is prepared once every two years or as required and are purchased from a reputable supplier such Accustandard or Ultra Scientific. The volumes of stock standards used for the current set of working level standards in use are documented in the Standard Preparation Log Book, located in the Instrument laboratory.

6.2.3 Use the following for 5 ng/µL stocks.

Component	Stock Conc. (ng/µL)	Final Conc. (pg/µL)	Vol. Added (µL)
BHC-delta $^{13}C_6$	100	5000	250
$^{13}C_{12}$-Tetrachlorobiphenyl (BZ 52)	50	5000	500
$^{13}C_{12}$-Pentachlorobiphenyl (BZ 101)	50	5000	500
$^{13}C_{12}$-Heptachlorobiphenyl (BZ 178)	50	5000	500

6.3 Apparatus: autosampler vials; assorted gas-tight micro syringes; assorted screw-cap amber vials with teflon-lined caps. Equivalent labware and equipment may be purchased from other suppliers as available.

6.3.1 Glassware Quality Control: all extraction glassware is engraved with a specific serial number. Glassware used for processing samples is tracked using this number. All glassware is subjected to a proven, rigorous cleaning procedure after which it is acceptable for reuse. Method Blank analyses confirm the acceptability of this procedure.

- Soxhlet apparatus that were exposed to samples resulting in highly coloured extracts are soaked in a soap solution, (Extran), overnight
- after soaking they are rinsed thoroughly with successive rinses of RODI water and acetone
- all Soxhlets are then cycled with toluene overnight
- Rotovap glassware is rinsed thoroughly with appropriate solvents after each use

6.4 Instrumentation: High Resolution GCs coupled to High Resolution Mass Spectrometers

HR Gas Chromatographs:		Associated HR Mass Spectrometers:	
HP6890A	(S/NUS000034029)	Micromass AutoSpec Ultima	(S/N M384)
HP6890	(S/N US00029754)	Micromass Autospec Ultima	(S/N M230)
HP6890D	(S/N US00030341)	Micromass Autospec Ultima	(S/N M449)
HP6890N	(S/N US10131086)	Micromass Autospec Ultima	(S/N M536)
HP5890 Series II	(S/N3203G16149)	VG AutoSpec "S"	(S/N S1800)
HP6890A Plus	(S/NUS00038736)	Micromass AutoSpec Ultima	(S/N M526)

6.4.1 Gas Chromatographs: gas chromatographs consist of the following components

- **Oven:** capable of maintaining a temp. ±1 °C and performing programmed temp. rate increases of at least 3 °C/min
- **Temperature Gauge:** to monitor column oven, detector, & exhaust temperature ±1 °C
- **Gas Flow Metering System:** to measure sample, fuel, combustion gas, & carrier gas flows
- **Capillary Column:** the primary column is a fused silica column, 60 m x 0.25 mm inside diameter (ID) x 0.25 um film thickness, coated with CP Sil 8CB (Varian or equivalent)

6.4.2 Mass Spectrometers: capable of routine operation at 1:10000 resolution with ±5ppm stability with a compatible data system capable of monitoring at least five groups of 25 ions. Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolution throughout the mass range during the function. Therefore, resolution must be ≥8,000 throughout the mass range and must be ≥10,000 in the center of the mass range for each function.

6.5 Miscellaneous Equipment: analytical balance capable of measuring within 0.1 mg

7.0 ANALYTICAL PROCEDURE:

7.1 Sample Extraction: refer to BRL SOP-00010 "for the Extraction and Clean-up of Organochlorine Pesticides from Liquid and Solid Samples.

7.2 Sample Clean Up and Fractionation: refer to BRL SOP-00010 "for the Extraction and Clean-up of Organochlorine Pesticides from Liquid and Solid Samples.

7.3 Analysis: analyze the sample extract by HRGC/HRMS using the instrumental parameters in section 7.3.1 and 7.3.2.

Step 1 add 4 µL of Recovery Standard to the 200 µL sample extract in the 1 mL Reacti-vial
Step 2 vortex for ~30 seconds and allow to equilibrate for at least 3 minutes
Step 3 transfer a portion to an amber autosampler vial using a micropipettor
Step 4 seal with an aluminum teflon lined crimp cap
Step 5 inject 1 or 2 µL of the extract into the GC

Analyze the sample using a GC/MS with the instrument parameters given in Section 7.3.1 and 7.3.2. Some samples, at a 200uL final volume, may contain high levels of the compounds of interest, interfering compounds, or polymeric materials, and may crystallize out of solution at room temperature. A 10-fold dilution is performed to address this issue, as well as the anomalies observed with recoveries of the internal standard. A 1-2 µl aliquot of both extracts is injected into the GC to determine the concentration of each target analyte.

7.3.1 Typical GC Operating Conditions (these are typical conditions and are optimized for each individual column to maximize performance.

- **Injector:** configured for capillary column, splitless, 250 °C
- **Carrier Gas:** helium at 1-2 ml/min
- Initial Temperature 100 °C for 2 minutes
- 20 °C/minute to 150 °C
- 5 °C/min to 285 °C
- hold at 285 °C for 2 minutes

7.3.3 High Resolution Mass Spectrometer

- Resolution of 8,000 minimum
- Ionization Mode: Electron impact, 35-40 eV, (optimize)
- Source Temperature: 290 °C, (optimize)
- Monitoring mode: Selected ion recording - see Table 1 for a list of the ions to be monitored

7.3.4 Run Sequence: at the beginning and the end of a run sequence, a hardcopy of the Resolution check is printed out. The operator manually verifies that a minimum resolution of 1:8,000 has been achieved for each multi group experiment; see the descriptor groups in see Table 1.

7.3.4.1 Initial Calibration: (process takes ~ 4 to 5 hours to perform)

- DDT/ENDRIN Breakdown Check
- CS1_OC
- CS2_OC
- CS3_OC
- CS4_OC
- CS5_OC
- Solvent
- OC_Ref_Std (optional 2nd source standard, if available)
- Solvent

7.3.4.2 Typical Sample Analysis Sequence: (process takes ~12 hours to perform). At the beginning and the end of a run sequence a hardcopy of the Resolution check is printed out. The operator manually verifies that a minimum resolution of 1:8,000 has been achieved for each multi group experiment, see the descriptor groups in Table 1.After this verification is complete the run can proceed as follows:

- DDT/ENDRIN Breakdown Check
- CS3_OC
- Solvent (optional)
- OPR
- Solvent
- Method Blank

- Sample 1
- Sample 2
- Sample 3
- Sample 4
- Sample 5
- Sample 6
- Sample 7
- **…**
- Sample 20
- Solvent
- CS3_OC (post analysis calibration verification)
- Method Spike (the method spike is not used in any calculations but serves only as an aid to determine the source of potential problems for internal and matrix recovery.)

7.4 Identification Criteria: the following preliminary identification criteria shall be used for characterization of OCs.

- the integrated ion-abundance ratio shall be within ± 20 percent of the theoretical value or the value obtained from that days calibration standard
- retention time for the analytes must be within 3 seconds for the corresponding labeled internal, surrogate or alternate standard
- the monitored ions shown in Table 1 for a given analyte shall reach their maximum within 2 seconds of each other
- the identification of specific analytes that do not have corresponding labeled standards is done by comparing the relative retention time, (RRT), of the analyte to the nearest internal standard retention time with reference, (i.e. within 0.005 RRT units), to the comparable RRT's found in the continuing calibration
- the signal to noise for all monitored ions must be >2.5

7.4.1 Quantification: the peak areas for the two ions monitored for each analyte are summed to yield the total response for each analyte.

7.5 Maintenance: Maxxam staff or a manufacturer's Service Engineer will perform emergency maintenance. Preventative maintenance is performed annually through a service contract.

7.6 Troubleshooting and Common Problems: the analyst may perform the following checks if he/she is capable. Otherwise call a Manufacturer's Service Engineer. Check for the following conditions:

- system is fully powered on and is in the Operate mode
- vacuum is in acceptable range, i.e. 10^{-6} mbar or better, if not check for leaks
- filament is "ON" and the trap is regulating
- source and transfer line heaters are at the proper operating temperature, the source temperature affects the fragmentation of the sample
- GC in good working order, adequate supply of carrier gas with respect to oven heater and temperature regulation
- Injector system is clean with a properly installed column, liner and septum. Leaks and contamination may cause performance problems
- capillary column Helium head pressure is adjusted to proper setting, i.e. ~25 psi for a 60 m x 0.25 mm ID column
- autosampler syringe is in good condition, not leaking nor plugged
- data system is running and is controlling and communicating with the instrument

7.6.1 Loss of Beam: may be caused by any of the following

- a defective source, collector or alpha slit may be obstructing the beam
- the isolation valve may not be completely open, check the pneumatic valve
- check the compressed air tank line pressure
- broken or blocked PFK jet or capillary line, replace if necessary

7.6.2 Loss of Filament Current, Trap Current and Electron Energy: may be caused by any of the following

- remove the source, check for short circuits, dirty or defective parts. Clean or replace if needed
- check the filament alignment in the source when replacing it
- if the filament comes on and there is no Trap current, this could indicate that a trap contact problem has occurred
- check the electrical contacts and the alignment of the source magnets

7.6.3 Loss of Magnet: may be caused by any of the following

- high voltage breaker switch tripped due to a power interruption or surge
- high voltage breaker switch tripped due to a loss of the cooling water supply. When cooling water supply is lost or is too warm the magnet unit and the diffusion pumps overheat. The heat sinks on the power supply board cause the power to shut down and may trip the breaker on the power distribution panel on the front of the instrument. These must all be reset before the magnet can be turned back on

7.6.4 Loss of High Voltage: may be caused by any of the following

- an electronic fault and may be brought on by a discharge of a dirty lens or ceramics or arcing of the flight tube
- these regions must be cleaned to restore voltages and to re-establish control of the beam
- electronics and S10S interface will also have to be reset to restore communications and control of the beam control and source control PCBs

8.0 QUALITY CONTROL:

8.1 DDT/ENDRIN Breakdown Check: This test is to be run at the beginning of every run before an initial calibration or continuing calibration. Its purpose is assess the activity of the injection port will eventually which cause Endrin and 4,4'-DDT to breakdown into Endrin Aldehyde and Ketone and 4,4'-DDD and 4,4'-DDE, respectively. The percent breakdowns are calculated as follows:

$$\text{4.4'-DDT Breakdown (\%)} = \frac{\text{(Sum of areas for 4,4'-DDD + sum of areas for 4,4'-DDE)}}{\text{Sum of areas for 4,4'-DDT}} \times 100$$

$$\text{Endrin Breakdown (\%)} = \frac{\text{(Sum of areas for Endrin Aldehyde + Endrin Ketone)}}{\text{Sum of areas for Endrin}} \times 100$$

- A percent breakdown of ≥20% indicates Endrin breakdown into Endrin Aldehyde and Ketone and similarly for 4,4'-DDT into 4,4'-DDD and DDE.
- If ≥20% breakdown is observed, the injection port must be cleaned and reconditioned. This means cooling the injector, wiping surfaces clean with Kim Wipe and methanol, replacing the septum, inlet liner and gold seal.
- Pyrex liners dual tapered are recommended for this test, as they are believed to be less active.
- Liners can be made to be inactive through re-silanization then capping with methanol. Consult a senior analyst or supervisor before attempt this as the silanizing compound dichlorodimethyl silane is corrosive.

8.2 Initial Calibration: calibrate the GC/MS system using the set of five standards, (Table 3). These working level standards are used for a period up to 2 years. If available, an "injection ready" standard from a second source supplier is also run as part of the initial calibration. Instrument parameters are adjusted and standards are analyzed until the following QC criteria are achieved.

- Relative standard deviation, (RSD), of the mean response factor for each unlabeled analytes should be +/- 35%.
- RSD of the mean response factor for each of the labeled standards should be +/-35%
- Signal to noise ratio for the GC signal present in every selected ion current profile shall be ≥10 for both labeled and unlabeled analytes
- The ion abundance ratios shall be within the control limits in Table 2

8.3 Continuing Calibration Verification: inject 1 µL of solution CS3, (see Table 3). At the beginning of each 12 hour sequence, calculate the relative response factors, (RRFs), for each compound and compare each of them to the corresponding mean RRF obtained during the initial multi-point calibration. The following requirements should be met before sample analysis can begin.

- the measured %RSD of the RRF for the labeled analytes for the daily run must be +/-50% except for $^{13}C_{12}$-Endrin and the natives that use it as an internal standard (Endosulfan Sulfate). As this compound is extremely susceptible to GC injector activity, its' response will increase as the liner is used. Calculations may use the daily RF for these compounds to correct for this.
- the measured %RSD of the RRF for the unlabeled analytes for the daily run must be +/-50%
- the ion abundance ratios must be within the limits given in Table 2 or within +/-35% of the value obtained from that days calibration standard

The CS3 standard is analyzed at the end of the run sequence. The %RSD of the RRFs are again compared to the initial calibration and should satisfy the above criteria. As another indicator of breakdown, a decrease in the RF of the ^{13}C-4, 4'-DDT of >50 % compared to the initial calibration is an indicator of injector liner degradation and GC inlet maintenance must be performed.

8.4 Internal Standards: are added to all samples and QC samples.

Recoveries must be 10-200% for the labeled compounds

8.4.1 Corrective Actions: if the above criterion is not achieved

- the data will still be acceptable provided that the signal is equal to or greater than ten times the noise level
- this will be flagged in the Case Narrative section of the final report
- the extract may be diluted and rerun. Complex matrices may mask or enhance the response of several compounds (Aldrin, methoxychlor, 4,4'-DDT)
- the sample may be re-extracted if nothing can be found to explain the low or high recoveries and no obvious interference is causing the problem.

8.5 Method Blanks: a method blank is run in every batch, and a minimum of one for every 20 samples.

Method Blank must be <CS1 Standard concentration (RDL)

NOTE: for DoD samples, the method blank will be considered to be contaminated if:

- The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
- The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
- The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives

8.5.1 Corrective Actions: if the above criteria are not met then

- investigate possible source of contamination by checking at a minimum: instrument Spike Standard Solutions, Recovery Standards, proofing of glassware, proofing of solvent and absorbents used in clean-up
- all of the samples must be re-prepared and reanalyzed
- if sufficient sample is not available then any positive sample data must be flagged as possibly contaminated to the level found in the Method Blank

8.6 Blank Matrix Spike: must be prepared and analyzed at a frequency of 1 Blank Spike for every batch of samples up to a maximum batch size of 20 samples. Calculate the recovery as:

$$\text{Spike Recovery (\%)} = \frac{\text{Spiked Blank Result}}{\text{Spike Added}} \times 100$$

Native analyte recoveries should be 40-200%

Internal Standard recoveries should be 10-200%

8.6.1 Corrective Actions:

- check calculations and reanalyse if recoveries are outside of these limits
- if the blank spike is outside of limits but the matrix spike is acceptable then the blank spike may have been spiked incorrectly. Review the data with the Team or Group Leader. All data may be accepted but must be flagged as exceeding acceptance criteria
- if both the blank spike and the matrix spikes exceed their respective limits re-prepare and reanalyze the samples providing sufficient sample is available
- if sufficient sample is not available the data must be flagged in Tester's Comments

8.7 Matrix Duplicate: must be prepared and analyzed using randomly selected samples, at a frequency of 1 matrix duplicate for every batch of samples up to a maximum batch size of 20 samples. The RPD is calculated as:

$$\% \text{ RPD} = \frac{\text{first sample result - duplicate sample result}}{\text{(First sample result + duplicate sample result)/2}} \times 100$$

RPD between duplicates should be ±25%

8.7.1 Corrective Actions:

- check calculations for errors
- check solid samples for homogeneity, if not homogeneous, flag the data in Tester's Comments
- if the sample is homogeneous, re-prepare and reanalyze the sample

8.8 Matrix Spike/Matrix Spike Duplicate Recoveries: if sufficient sample is available, spike two separate aliquots of a sample in each batch of 20 samples or less with an appropriate level, (2-5 times the sample concentration if possible), of native OCs. Calculate the recoveries as follows:

$$\text{Matrix Spike Recovery (\%)} = \frac{\text{spiked sample result - sample result}}{\text{amount spike added}} \times 100$$

Recoveries of matrix spiked internal standards must be between 10-200%

Native analyte recoveries should be 40-200%

RPD between duplicates should be ±25%

8.8.1 Corrective Actions:

- check calculations for errors
- if recovery calculations aren't possible, (due to high sample concs.), flag the data in Tester's Comments
- if the blank spike is acceptable then flag the data as a possible Matrix interference
- check solid samples for homogeneity, if not homogeneous, flag the data in Tester's Comments
- if the sample is homogeneous, re-prepare and reanalyze the sample
- if both the blank spike and the matrix spikes exceed their respective limits re-prepare and reanalyze the sample the sample if sufficient sample is available
- if sufficient sample is not available flag the data in Tester's Comments

8.9 Control Charts: plot the blank spike recovery of 4,4'-DDT, for waters, soils, and tissues on separate control charts. Update the chart immediately after the analysis of the QC point used or in the morning after an overnight run, prior to reporting of data. Monitor the chart for the development of trends on the chart. Take actions, if necessary, as defined in the SOP for Control Charting. If an "Out of Control" point is found, terminate the analysis and resolve the problem. Note the resolution directly on the chart or code the point to a reference in the analyst's workbook.

8.9.1 Possible Causes:

- deterioration of standard, reagents or injector liner activity
- standard, QC sample or reagent preparation error
- standard contamination or evaporation
- improper sample introduction
- poor analyst technique or insufficient training
- deviation from SOP procedure
- imprecise measuring devices, i.e. pipettors, syringes

8.9.2 Corrective Actions: if "out of control points" are encountered

- check the other run QC, if acceptable report the data
- if not acceptable, reanalyze the QC samples and if acceptable, report the data
- if not acceptable, find and resolve the cause, re-extract and reanalyze the samples
- if insufficient sample exists report the data, flagged as "out of statistical control"
- if hold times will be exceeded upon re-extraction and reanalysis, flag the data
- if no reason can be found, report the data as "out of statistical control"

Note the resolution on the Control Chart or reference it to the Analyst's Workbook.

8.10 Additional QC: in addition to the QC samples listed above, blind replicates, audit and performance evaluation samples may be submitted. These samples may have specific instructions that must be followed, however, analysis is to proceed as for any other sample. Any USACE project has an Analytical Data Review Checklist, documented in the QA Manual, completed and submitted with the final data report.

9.0 DATA ANALYSIS:

9.1 Calculations: the internal standard method is used to quantify OCs. It relies upon consistent linearity of MS response over time and over the calibration range represented by the standard solutions defined in Table 3. This internal standard method is easily integrated into an automated routine for data quantification. Internal standard quantification is based on the use of Relative Response Factors, (RRF). For native standards, the RRF is the ratio of analyte response factor to the response factor of the corresponding labeled surrogate, (internal standard). These RRFs remain unchanged over the range of concentration for which MS response is linear. Using these RRFs, along with native and surrogate responses from the sample run, recovery-corrected concentrations of OCs are calculated directly. Internal standard recoveries are calculated separately and reported. These values reflect the overall data quality.

Relative response factors for the native standard (RRFn) and for the Internal standard (RRFs) are calculated using the following equations:

$$RRFn = \frac{Ac * Csc}{Asc * Cc} \quad \text{and} \quad RRFs = \frac{Asc * Crc}{Arc \bullet Csc}$$

Where:

RRFn = relative response factor, Native Standard to Internal Standard
RRFs = relative response factor, Internal Standard to Recovery Standard
Ac = quantification ion (single or both ions) peak area for Native Standard
Asc = quantification ion (single or both ions) peak area for the appropriate Internal Standard
Arc = quantification ion (single or both ions) peak area for the appropriate Recovery Standard
Cc = concentration of the Native Standard (pg/µl)
Csc = concentration of the appropriate Internal Standard (pg/µl)
Crc = concentration of the appropriate Recovery Standard (pg/µl)

Using the RRFs, sample concentrations of OCs (C) and internal standard recoveries (%R) can be calculated as follows:

$$C(X) = \frac{\sum_{k=1}^{n} Ak \bullet Qss}{Ass \bullet RRFn} / Ws \text{ or } Vs \quad \text{and} \quad \%R(X) = \frac{Ass * Qrs * 100}{Ars * Qss * RRFs}$$

Where:

C(X) = recovery-corrected quantity of analyte X (ng/g for solids, ng/L in liquids)
Ak = quantification ion (single or both ions) peak area for the "k^{th}" homologous isomer of analyte X (n=1 for isomer-specific analysis)
Qss = amount of internal standard X added to the sample (ng)
Ass = quantification ion (single or both ions) peak area for internal standard X in extract
%R(X) = percent recovery of internal standard X
Qrs = amount of the appropriate Recovery Standard in sample extract (ng)
Ars = quantification ion (single or both ions) peak area for the appropriate Recovery Std in the sample extract
Ws = weight of dry sample in grams
V_S = volume of sample in litres

9.2 Data Transfer: data is processed using a custom application macro developed internally. An excel spreadsheet is generated which incorporates weights, dilution factors, splits, internal standard amounts added, recovery standard amounts added and estimated detection limits. The result file, once completed and checked by authorized personnel is electronically transferred to the MaxxLIMS and approved.

9.3 Data Validation: Refer to BRL WI-00011 Review and Validation of Analytical Data and corresponding Data Review Checklist (BRL FCD-00002) to perform a primary and a secondary analytical data review and validation prior to issuance of the Certificate of Analysis. If the data and report are acceptable the Certificate of Analysis is signed by the Project Manager and sent to the client.

9.4 Data Storage: sample data is transferred to a separate computer system as required. The back up computer is maintained by the systems group and is also backed up regularly.

10.0 DOCUMENTATION:

10.1 Tracking Sheet: before samples are prepared, a GC-MS sample tracking /work sheet is completed. This sheet includes details of the Job number, lab sample number, client sample ID, QC samples analyzed in the batch, spiking solutions used and their concentrations, preparation date and lot #, dates of analysis and cleanup, analyst's initials. A copy of the tracking /work sheet is maintained with analytical data

10.2.1 Instrument Logbook:

- Troubleshooting records with full descriptions of the actions taken to resolve the problem. This is required for all instances in which there is an acceptance criteria that is not achieved. Alternatively these may also be recorded on a separate tracking sheet designed for the analysis.
- Record of Instrument Service by the Supplier with the associated paperwork

10.2.2 Method Deviation Records: instances may arise in which the standard operating procedure may not be applicable to the nature of the sample and may require modifications to the normal methodology. These deviations must be discussed with the Supervisor or Operation Manager prior to application. Approval must be obtained as per COR WI-00040 and documented using QSI module for Policy Deviation Form. These deviations must also be described in the comment section of MaxxLIMS.

10.2.3 Sample Deviation Records: these deviations must be recorded in MaxxLIMS Comments.

10.4 Maintenance Tracking: analysts maintain logbooks/tracking sheets to include the following minimum information

- record of Instrument Service by the Supplier with the associated paperwork
- records of all maintenance performed indicating the date performed and by who

10.5 Standard and Reagent Preparation Tracking: record all preparations of standards and reagents in the Standard Preparation and the Reagent Preparation Logbooks. All records must contain at a minimum the following information

- Supplier Identification and Lot Number
- Date of Preparation and Expiry
- Concentration, and name of Analyst who prepared the standard

10.6 Certificates of Analysis for Standards and Reagents: certificate of analyses are kept for all inorganic salts and solutions utilized in this SOP. These records will contain at a minimum the following information.

- supplier information and Lot Number
- date received

10.7 Storage of Records:

10.7.1 Paper Records: raw and calculated data, including calibrations and QC results are stored chronologically in file folders in the laboratory. These files are archived on-site for a period of one year and then transferred to an off-site storage for 5 years, unless otherwise instructed.

Electronic Media: data entered into LIMS is backed up daily.

11.0 WASTE MANAGEMENT:

All efforts are taken to prevent or reduce to a minimum the effect of waste disposal on the environment. All solvents are collected for shipment to a recycling facility. All recyclable plastic, glass and paper products are shipped to an appropriate recycling facility. The disposal of waste materials and samples are to carried out in accordance with protocols outlined in the SOP for the Preparation, Storage and Disposal of Reagents and Standards and the SOP for the Receipt, Handling and Disposal of Hazardous Wastes. All waste disposal will comply with the Ontario Ministry of the Environment and Energy's Sewer Guidelines and Regulation 558.

11.1 Specific Disposal Issues: contaminated sample extracts are collected and sent for disposal to an approved hazardous waste subcontractor. Excess contaminated samples are either returned to the client or submitted for disposal as noted.

12.0 APPENDIX:

- Table 1: Exact Masses of the Ions Monitored by Hi-Resolution Mass Spectrometry
- Table 2: Theoretical Ion Abundance Ratios and their Control Limits
- Table 3: Hi Res OC Calibration Standards
- Table 4: Quantitation References For Organochlorine Pesticides

TABLE 1:

EXACT MASSES OF THE IONS MONITORED BY HI-RESOLUTION MASS SPECTROMETRY

Descriptor No:	Accurate Mass		Analyte
1	216.9145	M	a-BHC, b-BHC, d-BHC, g-BHC
	218.9116	M+2	a-BHC, b-BHC, d-BHC, g-BHC
	222.9347	M	13C6-beta BHC,13C6-delta BHC
	224.9317	M+2	13C6-beta BHC,13C6-delta BHC
	227.9662	M	13C6-d6-gamma BHC (Lindane)
	229.9632	M+2	13C6-d6-gamma BHC (Lindane)
	283.8102	M	Hexachlorobenzene
	285.8072	M+2	Hexachlorobenzene
	289.8303	M	13C6-Hexachlorobenzene
	291.8273	M+2	13C6-Hexachlorobenzene
	230.9856	LOCK MASS	PFK
2	262.857	M	Aldrin
	264.854	M+2	Aldrin
	269.8806	M	13C12-Aldrin
	271.8102	M	Heptachlor
	271.8776	M+2	13C12-Aldrin
	273.8072	M+2	Heptachlor
	276.8272	M	13C10-Heptachlor
	278.8242	M+2	13C10-Heptachlor
	301.9626	M	13C-PCB 52
	303.9597	M+2	13C-PCB 52
	280.9824	LOCK MASS	PFK
3	352.8442	M	Heptachlor Epoxide
	354.8413	M+2	Heptachlor Epoxide
	362.8779	M	13C10-Heptachlor Epoxide
	364.8749	M+2	13C10-Heptachlor Epoxide
	377.7482	M	Octachlorostyrene
	379.7452	M+2	Octachlorostyrene
	386.8055	M	oxy-Chlordane
	388.8025	M+2	oxy-Chlordane
	396.839	M	13C10-Oxychlordane
	398.839	M+2	13C10-Oxychlordane
	380.976	LOCK MASS	PFK
4	194.9537	M	a-Endosulfan
	196.9507	M+2	a-Endosulfan
	201.9771	M	13C9-Endosulfan I
	203.9741	M+2	13C9-Endosulfan I
	234.8445	M	g-Chlordane, a-Chlordane
	235.0081	M	2,4'-DDD
	236.8416	M+2	g-Chlordane, a-Chlordane
	237.0052	M+2	2,4'-DDD
	239.8613	M	13C10-Chlordane
	241.8583	M+2	13C10-Chlordane
	246.0003	M	2,4'-DDE,4,4'-DDE
	247.9974	M+2	2,4'-DDE,4,4'-DDE
	258.0406	M	13C-pp-DDE
	260.0376	M+2	13C-pp-DDE
	262.857	M	trans-Nonachlor, Dieldrin
	264.854	M+2	trans-Nonachlor, Dieldrin
	265.986	M	13C-PCB 101
	267.983	M+2	13C-PCB 101
	269.8806	M	13C10-trans Nonachlor,13C12-Dieldrin
	271.8776	M+2	13C10-trans Nonachlor,13C12-Dieldrin
	242.9856	LOCK MASS	PFK

TABLE 1: continued

Descriptor No:	Accurate Mass		Analyte
5	194.9537	M	b-Endosulfan
	196.9507	M+2	b-Endosulfan
	201.9771	M	13C9-Endosulfan II
	203.9741	M+2	13C9-Endosulfan II
	235.0081	M	4,4'-DDD, 2,4'-DDT, 4,4'-DDT
	237.0052	M+2	4,4'-DDD, 2,4'-DDT, 4,4'-DDT
	247.0484	M	13C12-pp-DDD
	249.0454	M+2	13C12-pp-DDD
	247.0484	M	13C-pp-DDT
	247.8523	M	Endrin Aldehyde
	249.0454	M+2	13C-pp-DDT
	249.8493	M+2	Endrin Aldehyde
	262.857	M	Endrin, cis-Nonachlor
	264.854	M+2	Endrin, cis-Nonachlor
	269.8806	M	13C12-Endrin, 13C10-cis Nonachlor
	271.8102	M	Endosulfan Sulfate
	271.8776	M+2	13C12-Endrin, 13C10-cis Nonachlor
	273.8072	M+2	Endosulfan Sulfate
	242.9856	LOCK MASS	PFK
6	405.8428	M	13C-PCB 178
	407.8398	M+2	13C-PCB 178
	392.976	LOCK MASS	PFK
7	227.1072	M	Methoxychlor
	228.1106	M+2	Methoxychlor
	239.1475	M	13C-Methoxychlor
	271.8103	M	Mirex
	273.8073	M+2	Mirex
	276.8272	M	13C10-Mirex
	278.8242	M+2	13C10-Mirex
	316.9039	M	Endrin Ketone
	318.901	M+2	Endrin Ketone
	280.9824	LOCK MASS	PFK

H	=	1.007825
C	=	12.000000
13C12	=	13.003355
F	=	18.9964
O	=	15.994915
35Cl	=	34.968853
37Cl	=	36.965903

Alternate PFK lock masses may be employed in order to remove column bleed interferences should a new column not be available.

TABLE 2:

THEORETICAL ION ABUNDANCE RATIOS AND THEIR CONTROL LIMITS

Number of Cl Atoms	Ion Type	Theoretical Ratio	Control Limits * lower	upper
4	M/M+2	0.77	0.62	0.92
5	M+2/M+4	1.55	1.24	1.86
6	M+2/M+4	1.24	0.99	1.49
6	M/M+2	0.51	0.41	0.61
7	M/M+2	0.44	0.35	0.53
7	M+2/M+4	1.04	0.83	1.25
8	M+2/M+4	0.89	0.71	1.07

* QC limits represent ± 20% window around the theoretical ion abundance ratios. These limits are preliminary. As of 2007, the method USEPA 1699 states that the ratios are in a 25-35% window.

TABLE 3: Hi Res OC Calibration Standards

Component	Stock Conc. (ng/µl)	CS5		CS4		CS3		CS2		CS1	
		Conc. (pg/µl)	Vol. Req'd. (µl)	Conc. (pg/µl)	Vol. Req'd. (µl)	Conc. (pg/µl)	Vol. Req'd. (µl)	Conc. (pg/µl)	Vol. Req'd. (µl)	Conc. (pg/µl)	Vol. Req'd. (µl)
Native Analytes											
OC Native Intermediate	20	2000	400	400	80	100	20	-	-	-	-
OC Native Dilution	0.2	-	-	-	-	-	-	20	400	5	100
Internal Standards											
OC Internal Standard	4	100	100	100	100	100	100	100	100	100	100
Recovery Standards											
OC Recovery Standard Mix	5	100	80	100	80	100	80	100	80	100	80

Final Volume of all standards is 4 mL.

Table 4

QUANTITATION REFERENCES FOR ORGANOCHLORINE PESTICIDES

Compound	CAS Number	Internal Standard Reference	Recovery Std Reference
2,4'-DDD	53-19-0	p,p'-DDD-ring $^{13}C_{12}$	13C-PCB 101
2,4'-DDE	3424-82-6	p,p'-DDE-ring $^{13}C_{12}$	
2,4'-DDT	789-02-6	p,p'-DDT-ring $^{13}C_{12}$	
4,4'-DDD	72-54-8		
4,4'-DDE	72-55-9	p,p'-DDE-ring $^{13}C_{12}$	13C-PCB 101
4,4'-DDT	50-29-3	p,p'-DDT-ring $^{13}C_{12}$	13C-PCB 101
Aldrin	309-00-2	Aldrin $^{13}C_{12}$	13C-PCB 52
BHC-alpha	319-84-6	BHC-beta $^{13}C_6$	
BHC-beta	319-85-7	BHC-beta $^{13}C_6$	13C6-delta BHC
BHC-delta	319-86-8	BHC-beta $^{13}C_6$	
BHC-gamma	58-89-9	Lindane $^{13}C_6$ d_6	13C6-delta BHC
Chlordane (cis)	5103-71-9	Chlordane $^{13}C_{10}$	
Chlordane (oxy)	27304-13-8	Oxychlordane $^{13}C_{10}$	13C-PCB 52
Chlordane (trans)	5103-74-2	Chlordane $^{13}C_{10}$	13C-PCB 101
Dieldrin	60-57-1	Dieldrin $^{13}C_{12}$	13C-PCB 101
Endosulfan I	959-98-8	endosulfan-I-$^{13}C_9$	13C-PCB 101
Endosulfan II	33213-65-9	endosulfan-II-$^{13}C_9$	13C-PCB 101
Endosulfan Sulfate	1031-07-8	Endrin $^{13}C_{12}$	
Endrin	72-20-8	Endrin $^{13}C_{12}$	13C-PCB 101
Endrin Aldehyde	7421-36-3	Endrin $^{13}C_{12}$	
Endrin Ketone	53494-70-5	Mirex $^{13}C_{10}$	
Heptachlor	76-44-8	Heptachlor $^{13}C_{10}$	13C-PCB 52
Heptachlor Epoxide B	1024-57-3	Heptachlor Epoxide $^{13}C_{10}$	13C-PCB 52
Hexachlorobenzene	118-74-1	HXB $^{13}C_6$	13C6-delta BHC
Methoxychlor	72-43-5	Methoxychlor-ring $^{13}C_{12}$	13C-PCB 178
Mirex	2385-85-5	Mirex $^{13}C_{10}$	13C-PCB 178
Nonachlor (cis)	5103-73-1	cis Nonachlor $^{13}C_{10}$	13C-PCB 101
Nonachlor (trans)	39765-80-5	trans Nonachlor $^{13}C_{10}$	13C-PCB 101