02 - ORGANIC ANALYTICAL METHODS

THE ABOVE TEST METHOD IS CURRENT AND VALIDATED.

Revision 013

Revision Date: September 2008

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Lab Supervisor Date

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Lab Head Date

METHOD NUMBER: 02-001

Title: Determination of Chlorobenzenes (CBs), Organochlorine Pesticides (OCs) and PCBs in Biological Tissue Samples (vegetals and animals) by Column-Solid/Liquid Extraction and Gas Chromotography with Electron Capture Detector (GC-ECD).

1. Introduction and Scope

Chlorobenzenes (CB's) and PCB's form a class of compounds which are widely found as intermediates in the heavy and chemical industry. They are discharged into the environment through chemical waste dump leachate, manufacturing effluent and solvent applications [1].

The organochlorine pesticides (OC's) form a class of compounds which includes once widely used insecticides. OC's can be classified as halogen derivatives of alicyclic hydrocarbons (BHC, Chlordane, Heptachlor, Aldrin, Dieldrin, Endrin and DDT), and are epigenic carcinogens [2].

Polychlorinated biphenyls (PCB's) are also carcinogenic and have been used extensively in industry as a flame retardants, high pressure lubricants, waterproofing compounds, casting wax, adhesives, asphalt and as dielectric, hydraulic and grinding fluids [3].

Generally, CB's, OC's, and PCB's are resistant to degradation and oxidation, are relatively insoluble in water, have low volatility, and are hydrophobic.

The possibility of environmental damage by organochlorine pesticides is of particular concern in view of their persistence. The stability of many of these compounds allows them to persist in the environment almost indefinitely, creating a special hazard to wildlife due to the capability of many organisms to bioconcentrate them. This means that low levels (ng/L) of OC's in water can be biomagnified up the food chain so that fish and fish-eating birds may contain mg/kg levels of these compounds. DDT has been implicated in egg shell thinning in wild birds, endangering survival of the species.

Scope: This method provides the analytical and supporting methodology necessary for the extraction, quantitative determination and confirmation of chlorobenzenes, organochlorine pesticides and polychlorinated biphenyls. The parameters analyzed under this method are listed in Tables 02-001-1:a, b, c. Their chemical names, structures, molecular formulae and weights are displayed in Figures 02-001-1:a, b.

2. Principle of the Method

This method includes the qualitative and quantitative determination of the chemical mentioned (see Appendix A - Tables 02-001-1a, b and c) by column solid-liquid extraction with DCM/Hexane (1:1), lipid and moisture content determinations, (occasionally lipid separation by GPC), clean-up and fractionation on florisil column, (occasionally followed by Coplanar PCB's separation on Carbon/Silica column) and instrumental analysis by GC-ECD. The method is applied to the following types of biota samples: plankton, invertebrates, mussels, fish, birds (muscle, egg or liver) animal tissue (muscle, liver or fat), macrophyte.

3. Interferences

3.1 Common Interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing (labware) that may lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free of any interference under the conditions outlined in the Related Procedures and using reagent blanks with each set of 6-10 samples.

3.2 Limitations - Due to numerous extraneous organics present in the matrix, repeated dilutions of sample extract may be needed. PCB's and some of DDT group are not totally separated using this method. Organic Sulphur Compounds often present in macrophyte samples must be removed by activated Copper Powder treatment of extract before GC/ECD analysis.
Phthalate Esters, must be removed in the routine clean-up procedure.

4. Safety and Waste Disposal

4.1 The stock standards and most chemicals used in this method pose a hazard to the analyst. Protective clothing, such as laboratory coats, latex gloves and safety glasses, must be worn when working with this method. All work with solvents must be performed in a fume hood or canopy and not on the open bench. For more information on first aid and preventive measures, handling and safe disposal of the chemicals and solvents used, consult the appropriate Material Safety Data Sheets.

4.2 Waste from the laboratory is divided into 3 types: chlorinated solvent waste, non-chlorinated solvent waste (including all standards and extracts) and solid waste (used Florisil). Solvent wastes are collected in 20 L container stored in the solvent storage room and disposed of by the Chemical Control Centre, University of Windsor.

5. Equipment, Reagents and Consumable Materials

5.1 Equipment

- Gas Chromatograph, split/splitless capillary injector (HP-6890 Series Plus) equipped with micro electron capture detector.
- Muffle furnace up to 1000^{o}C or more
- Oven capable of producing temperatures up to 200^{o}C
- Balances:
  Mettler - PJ 3000
  Mettler - AE 260-S, Delta Range, 0-60 g, readability 0.0001 g
- Homogenizer, variable speed Omni-mixer; Polytron; food blender
- Vortex Stirrer
- Rotary evaporator - Büchi Rotavapor, with water bath temperature 33^{o}C
- Reacti-Therm evaporator

5.2 Reagents and Consumable Materials (See Related Procedures):

5.2.1 Bulk solvents required for this method are:

- Hexane, Isooctane, DCM and Toluene are supplied by FISHER - Optima grade (distilled in glass)
- Acetone - FISHER- ACS grade (for glassware rinsing)

5.2.2 Sodium sulphate anhydrous granular (Na_2SO_4), - ACS grade

5.2.3 Activated florisil, 60-100 mesh supplied by FISHER

5.2.4 Bio-Beads, S-X3 supplied by BioRad

5.2.5 Activated Copper Powder supplied by Fisher Scientific

5.2.6 Analytical Standards are supplied by Sigma-Aldrich, Chromatographic Specialties, (Accustandard Corporation), Supelco or by Wellington Laboratories.

For specific research project, will be implemented the standards of respective co-laboratory research department will be implemented in place of the above mentioned certified standards.

5.2.7 All analytical standards are stored in a freezer/ refrigerator used exclusively for safety storage of chemicals only, at a temperature of -10 ºC to -20 ºC/ -3 ºC to 3 ºC.

5.2.8 Ensure that calibration standards have not passed the expiry date mentioned by suppliers.

5.2.9 Fiber glass wool (Pyrex Brand) supplied by VWR

5.3 Labware:

Glass mortar and pestle (250 mL); Glass column, 35 cm x 2.1 cm I.D. with Teflon stopcock and 300 mL reservoir at the top; Glass column, 25 cm x 1 cm I.D. with Teflon stopcock and 250 mL reservoir at the top; 500 mL, 250 mL, 150 mL round or flat bottom flasks, with 24/40 outer joint; 50, 100, 250 and 500 mL glass beakers; 40 mL graduated centrifuge tubes with ground glass stopper; Graduated cylinders; 2, 5, 10, 25, 50, 100 mL class A volumetric flasks; 50, 100, 500 and 1000 µL syringes for preparation of standard solutions; 0.2, 1, 2, and 5 mL volumetric pipets; Glass wool; Pasteur pipets; Stainless steel spatulas; Aluminum weigh boats; Disposable glass vials (2mL); Glass inserts and teflon/ rubber crimp caps. For glassware cleaning instructions (See the Related Procedures).

6. Sample Collection, Preservation and Handling

6.1 The samples must be collected exclusively in glass, stainless steel containers with teflon lines or aluminum foil packages. All other materials such as rubber or plastic are totally unacceptable because of eventual interferences which can be released during

preparation of samples. The samples are stored in a freezer at (-20^{o}C) for up to 5 years in amber jars or aluminum foil (previously cleaned and rinsed with solvents).

6.2 Sample Pre-treatment

The samples should be homogenized using a food blender, polytron or omni mixer. One hour prior to extraction, samples are allowed to thaw at room temperature, the excess liquid is poured out, homogenized again using a clean spatula and a corresponding aliquot is weighed for analysis. The sample size is 5-10 g (or depending on lipid content).

7. Sample Receiving

Samples are received (from the field and/or from client suppliers) by GLIER where they are checked and logged into GLIER Laboratory Code by the Sample Receptionist in the Organic Analytical Laboratory. This laboratory is responsible for the proper storage of the samples (at -20 ^{o}C in the dark) until they are processed for analysis Occasionally, a pretreatment of sample is needed as detailed in Section 6.2.

8. Quality Assurance/Quality Control (QA/QC)

8.1 Analyst proficiency:

All analysts involved in the analysis of CB's, OC's, and PCB's must be adequately trained using CAEAL requirements.

8.2 ***Instrument Control, Calibration, Stability:***

8.2.1 Calibration of Instrument is performed by running three replicates for each of 7 Levels (1 calibration blank, 6 standards) of concentrations for the Calibration Standard (the lowest level been less than 10x Det.Limit), evaluate the average, calculate the RSQ and determine the Working linear range for each Analyte; Appendix A: Table 02-001-1.
Note: If GC-ECD chromatogram output graph says no peak stored or matched enter zero. (See L0 in table 02-001-01)

8.2.2 Calibration must be performed once per month or after any major Instrument adjustments (or changes) have been done.

8.2.3 The Instrument is considered to perform accurately when the R^2 is above 0.98 (Table 02-001-1)

8.2.4 If the values are out of this range, the Instrument Performance must be tested, the improvements needed and the calibration repeated (see Quality manual Section 2.6.2)

8.2.5 Implement the Control Charts for Standard response.

8.2.6 The instrument calibration Control Charts must be updated once per month.

8.2.7 An external SRM control standard is used as an instrument calibration cross check of calibration standards. It is run in triplicate once per month in routine but is run more frequently when necessary as per requirements; 1) If new in house calibration standards are prepared 2) evidence of deviation of any of the 7 level calibration standards.
Differences higher than 20% of major Analytes (OCs and PCB-Congeners); HCB p,p'-DDE, Oxychlordane, Dieldrin & PCBs#: 52,101,153,138,180,194 are not acceptable.

8.3 ***Method Quality Control*** is based on the following criteria performed on a daily basis: Background, Accuracy, Precision and % Recovery.

8.3.1 Background: A method blank spiked with a specific amount of an appropriate Surrogate Spiking Standard is run with each batch of 6-10 samples. This is a sample without matrix run through all steps of preparation and analysis procedures, to monitor the background contamination and interferences from the labware and reagents, and serve to evaluate method recovery. If the run indicates the data to be out of control, refer to section 8.5 for Corrective Action.

8.3.2 Accuracy: A Referance sample, (i.e. CWS egg pool or "in-house" fish pool) is run for each batch of 10-12 samples). The "in-house" fish pool consists of a subsample from a homogenized contaminated fish meat. The target values for this "in-house" Reference sample are determined by repeated assays (at least 20 replicates). Accuracy of determinations is evaluated by the authorized QC Officer submitting control samples (or, for the "in-house" fish pool by the Laboratory Supervisor confirming with established control limits). When a Reference sample is not available, a Blank spiked with a known amount of analytical standard (or just some specific compounds) can be used. The data should fall within the established control limits, defined as: +or-3SD of reference value for each analyte in the Reference Sample, or in the Spiked Blank. If not, refer to Section 8.5 for Corrective Action

8.3.3 Precision: A SRM/ Reference Material run with each batch of 5-6 samples to monitor within-day precision. The differences in concentrations should fall within the certified values or established Control Limits (+or-3SD). RDS should be <30 %. If not, refer to Section 8.5 for Corrective Actions.

8.3.4 % Recovery: A Surrogate Spiking Standard (Recovery Internal Standard) containing chemicals which are not currently analyzed and don't give interferences with target analytes, but behave similarly during the processes is used to monitor extraction efficiency and overall method performance. For this method, the Surrogate Spiking Standard contains: 1,3,5-tribromobenzene. This surrogate is added by the analyst to method blank, every sample and quality control sample prior to the extraction step. % Recovery for the surrogate is calculated and should fall within the established control limits (±40%) and. If not, refer to Section 8.5 for Corrective Action.

8.3.5 Control Charts

The procedure to create the Control Charts consist in the next:

8.3.5.1 All data files were created in Microsoft Excel and a Database was set-up for:
- Standard Areas
- Reference Materials Results
- % Recovery for spiked surrogate in Blanks, Reference Materials and samples

8.3.5.2 Organize all Data in a calendaristic order

8.3.5.3 Select ten major Analytes (OCs and PCB-Congeners) to be represented graphically in control Charts. These are: HCB, p,p'-DDE, Oxychlordane, Dieldrin & PCBs#: 52,101,153,138,180,194

8.3.5.4 Perform the following calculations:
Average, Standard deviation (SD), Replicate differences

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8.3.5.5 Based on the Average Response and SD are set up the control limits necessary in representation of control charts. The limits are within +or-3SD.

8.3.5.6 Individual control charts (for each of 10 Analytes selected) are created.

8.3.5.7 All Database and the control Charts are saved into Microsoft Excel software (Data Directory C:\MyFiles\Organic Lab.Data\Organic LIMS.ST\uncertainty\ sop02-004\File name and stored in the QC Office: GLIER, RM. 220

8.4 ***Method Performance*** (Method Validation) once evaluated must be updated only when method is changed, by determination of the following criteria as described in Related Procedures.

Accuracy, expressed as % Error (% E) is a measure of the method bias from the target values, and is referred to in Appendix A, Table 02-001-4, 5.

Precision, expressed as coefficient of variation % (% CV) is a measure of the method repeatability and reproducibility; it is referred to in Appendix A, Table 02-001-4, 5.

Recovery, (% R) of added analytes is an indication of the method trueness, and is referred to in Appendix A, Table 02-001-4/5.

Method Detection Limit (MDL) is the lowest concentration of analyte in biota at which an unquantifiable peak is larger than the uncertainty associated with it. The MDL is estimated by spiking a clean matrix (synthetic triolein) with analytes at a target concentration, quantifying the peaks in replicate spiked matrix samples and determining the lowest spiking level where the mean measured concentration of replicate samples is not significantly different from the spiking level. This lowest value is then multiplied by a factor of 3 as a conservative measure. The MDL's for this method are provided in Appendix A, Tables 02-001-4.

Method Quantification Level (MQL): is the point at which a measured value is larger than the uncertainty associated with it and is statistically different from the response obtained from a blank carried through the complete method, including extraction and pretreatment. The MQL's for this method are provided in Appendix A, Tables 02-001-4.

8.5 Non Conformance and Corrective Actions:

When values for reference materials are outside the established Control Limits (+or-3SD), a "Nonconformance Action" form is submitted by the Organic Analytical Supervisor and Corrective Actions are initiated as mentioned below:

- o If one value in an analyst's data set exceeds the Control Limits (+or-3SD), the Laboratory Supervisor must re-examine the chromatograms (for eventual interferences), double check the data recorded, determine the source of the problem and correct it if possible.

- o If more than one value of an analyst's data set exceeds the Control Limit, reject the data, determine if previous data has been affected, and repeat the analysis.

- o For a consecutive failure, recheck the analyst's proficiency (by performing a blind performance audit).

8.6 Internal Performance Audits

Blind performance audits are conducted semi-annually by the GLIER Organic Laboratory. This audit consists of the submission of samples that have been spiked with known concentrations (blind to the analyst) of CB's, OC's, and PCB's. These concentrations are within the working range of the analytical method. The Organic Laboratory Supervisor reviews the completed analytical report and highlights any abnormal deviations from expected results. The Laboratory Manager is responsible for taking appropriate corrective action, documenting the corrective action taken and submitting a full report to the Laboratory Head.

8.7 Accreditation

The Canadian Association for Environmental Analytical Laboratories (CAEAL) is the accrediting organization for the analysis of CB's, OC's and PCB's. GLIER participates in studies that are necessary to assure accreditation for this method [6].

8.8 Balance Calibration Check

Using an appropriate certified calibration weight or a control sample (OWbal) check and document the balance on as used basis. Record in Balance log book.

9. Preparation and Procedure

9.1 An analytical run will normally consist of 1 method blank, 1 Control Sample and 5 unknown samples, (including a duplicate, when available) as mentioned in Appendix A, Table 02-001-3.

9.2 Sample Extraction:

9.2.1 Balance Calibration Check

Before weighing, make sure that the ball (viewed from outside the balance) is at the centre of the circle. Weigh the Certified calibration weight (or a control sample (OWbal) and record in balance log book in the first row for each type of balance used that day of the test method.

9.2.2 Procedure

2-5 g aliquot of animal tissue (respectively 10g of wet macrophyte sample) is homogenized with anhydrous sodium sulphate (5 times more the sample weight) using a glass mortar and pestle. The free flowing powder obtained is poured into a 35 cm x 2 cm glass column (with teflon stopcock and 300 mL reservoir) which had been plugged previously with glass wool, filled with 25 mL DCM/Hexane (50% V/V) and 2 cm of anhydrous sodium sulphate. Another 10 g sodium sulphate are mixed into the mortar (to remove residue of sample) and then added to the top of the column. The mortar and pestle are rinsed three times with a total volume of 25 mL of 50% DCM/Hexane and these rinses are transferred to the column. Add a specific amount of the Surrogate Spiking Standard in each column. After one hour (to soak the sample in the solvent mixture) the stopcock is opened and the sample is eluted with another 250 mL of 50% DCM/Hexane (total amount of solvent used is 300 mL). The elute is collected in a 500 mL round bottom flask with a steady drip of 5-10 mL/min., then rotaevaporated (Büchi-Rotaevaporator) to approximately 5 mL and transferred to a 25 mL grade A Volumetric flask, made up to 25 mL with Hexane, mixed and allowed to stand until the sodium sulphate is settled out.

9.3 Lipid Content Determination:

Lipid determination is made by pipetting 2 mL of the above extract into a pre-weighed 10 mL glass beaker. The solvent is evaporated to dryness in the fume hood and the beaker is then placed into a drying oven at 110^{o}C for one hour. After one hour, the beaker is removed from the oven, kept for one hour in a desiccator and re-weighed. The difference in weight represents the weight of lipid.

Percent of lipid is calculated as follows:

$$\%lipid = (W.L/W.T)*(V.T/V.e)*100$$

Where:

W_L = Weight of lipid
W_T = Total weight of sample
V_T = Total volume of extract (25 mL)
V_e = Volume of extract used for lipid determination (2 mL)

The remaining 23 mL of sample extract (after determination of lipid content) is rotaevaporated to 2 mL and saved either for bulk lipid removal by GPC or for florisil cleanup and separation.

9.4 Lipid Separation by G.P.C. (Occasionally, i.e. for samples with more then 0.15g lipid/sample)

The GPC column consists of a 50 cm x 2.2 cm Pyrex chromatographic column, with a teflon stopcock, packed previously with a 2 cm glass wool plug and then a slurry of 50 g BioBeads, S-X3 (BioRad) in 50% DCM/Hexane (V/V) and fitted at the top with a 250 mL pressure - equalizing separatory funnel. The column is preserved at all times with this solvent mixture and not allowed to dry out at any point after packing. The column is calibrated and tested periodically to confirm the elution volumes required for separation of lipid and contaminant fractions.

2 mL of DCM are added to 2 mL of isooctane-evaporated extract. This mixture is transferred and allowed to sink into the beads, then collection of eluate is initiated in a graduated container. The initial sample container is rinsed 3 x 4 mL of DCM/Hexane (1:1) from a pre-measured 300 mL of solvent mixture. After the final rinsing, the separatory funnel is placed on the top of the column, then the column is filled with remaining solvent, and elution is performed at a steady drip of 5 mL/min. The first 120 mL of eluent, containing the bulk of lipids, is discarded (or can be used for lipid content determination) and the last 180 mL are collected in a 500 mL round bottom flask. This final fraction is rotaevaporated to approximately 2 mL (after 5mL of isooctane was added) and saved for further cleanup and separation prior to GC-ECD analysis.

9.5 Moisture Content Determination

In addition to extraction procedures a 1 g aliquot of sample is weighed in a pre-weighed aluminum weigh boat, kept in a drying oven for 24 hours at 110^{o}C and re-weighed. The difference in weight represents the weight of moisture. The % of moisture is calculated using the following formula:

$$\% \text{ Moisture Content} = (A-B)/A \times 100$$

Where:

A = Aliquot Sample Weight before drying (g)
B = Aliquot Sample Weight after drying (g)

9.6 Florisil Cleanup and Fractionation:

The 2 mL of evaporated sample extract (saved either after lipid content determination or after GPC separation) is transferred to a florisil column prepared as follows:
25 cm x 1 cm glass column (with a teflon stopcock and 250 mL reservoir) is plugged with 2 cm glass wool, filled with hexane and then, 6 g of activated florisil is poured into the column (Florisil 60-100 mesh from BDH or FISCHER (activated overnight at 130oC). A gentle shaking of the column is necessary to settle the florisil inside the column. Two cm of sodium sulphate is then added on top. When the Hexane reaches the top of the filling, the stopcock is closed, a 250 mL round bottom flask is placed under the column and the evaporated sample extract is transferred to the top of the column using a Pasteur pipet. As the sample passes through the column, the sample container is rinsed with 3 x 2 mL portions of Hexane (from a premeasured 50 mL of Hexane) and rinses are also transferred to the column. The remaining Hexane is poured into the reservoir of the column and the elution of Fraction 1 (containing PCBs, some OCs and CBs) is performed at a steady drip of 3 mL/min. When the Hexane reaches the top of the filling, the stopcock is turned off, the flask with Fraction 1 is replaced with another 250 mL flask, the column is eluted with 50 mL of 15% DCM/Hexane (V/V) and Fraction 2 (containing OCs, CBs and non-ortho-substituted PCB congeners) is collected. The procedure is then repeated for Fraction 3 (containing Heptachor Epoxide and Dieldrin) using 150 mL of 60% DCM/Hexane (V/V). Each of the 3 fractions is separately rotaevaporated (after adding 5 mL of isooctane - Optima grade) to 2 mL, then transferred and made up to a suitable volume with isooctane.

The processed sample's fractions are stored in refrigerator (-3 to +3^{o}C) for up to 2 years).

9.7 Carbon/Silica Column Separation of Non-Ortho substituted PCBs

A 5% carbon/silica gel mixture containing AX-21 activated carbon (Anderson Development Company) and Silica gel (100 - 200 Mesh from Supelco), activated overnight at 130^{o}C. This mixture is cooled to room temperature in a dessicator prior to preparing the glass column. The glass column consists of 0.6 cm x 10 cm glass tube with similar ground joints (7/25) at both ends, to match a 50 mL glass reservoir. A 2 cm bed of 5% carbon/silica mixture is filled (dry) between 2 x 1 cm glass wool beds. The column is rinsed concurrently with 15 mL of Toluene, 15 mL of DCM, and 15 mL of Hexane to activate the column. A 0.5 mL concentrated Fraction 2 (from florisil cleanup step) is then added to the top of the column using a Pasteur pipette. The column is eluted with 30 mL Hexane, and the elute is discarded, then eluted with 30 mL of DCM and discarded. The column is then inverted and eluted with 30 mL of Toluene and the Fraction which contains the non-*ortho* substituted PCBs is collected,

then evaporated and transferred to a graduated volumetric flask and diluted with isooctane to an appropriate volume prior to GC-ECD analysis.

9.8 Instrument Control and Calibration: (GC-ECD, Capillary)

9.8.1 Develop a sample sequence using GC-Chemstation Software.

9.8.2 Load the GC method (created before). Make sure that there is an adequate supply of carrier gas and make-up gas. Fill the wash vials in the autosampler tower turret with Isooctane and empty the waste vials. Run Reagent blank (solvent).

9.8.2 Inject Calibration Standard (S1) to quantify target analyte concentrations at the interval shown in the analytical tray pattern (Table 02-001-3), and response factors are updated and used for quantification of succeeding samples. Analyte concentrations are calculated according to equation in Section 10. The response factors for all standards must be within ±20% of the initial calibration standard or the analysis must be repeated from the point of calibration failure.

9.8.3 Instrumental Conditions

The three fractions from the Florisil Column cleanup step are run separately on a Agilent 6890, Series Plus Gas Chromatograph, equipped as follows:

63Ni-μECD
Agilent-7683 Series Autosampler with tray (100 sample vials)
EPC
Column: 60 m x 0.25 mm. I.D. x 0.10 μm DB-5 film thickness (J&W)
1 μl sample is injected using a splitless injection mode.
Inj. Temp: 250°C; Det. Temp: 300°C
Carrier Gas: (UHP) - He at approximately 22 cm/sec - determined at 90°C (1 mL/min); Column Head Pressure is 22.88 psi
Make-up Gas: Ar/CH4 (95%/5%) at 50 mL/min
Oven Temperature Program:
Initial Temp: 90°C
Initial Time: 1.0 min
Rate: 20°C/min to 200°C, hold 2 min., then 3.0°C/ min to 280°C
Final Hold Time: 5 min
Equilibration Time: 3 min

9.9 Measurement:

9.9.1 Start MicroSoft Excel software to run and collect the data in EXCEL format automatically (Macro).

9.9.2 Start online GC ChemStation machine operating software.

9.9.3 Set up the instrument sequence for automated runs of standards, method blanks and the sample extracts, as shown in Table 02-001-3. Set up the data acquisition system to capture the target compounds for CBs, OCs and PCBs, listing peak areas. Load the autosampler tray with the vialed samples and standards as set up in the sequence and start the run.

9.9.4 On completing the sequence, disable the GC and raise the GC oven temperature to 150 ^{o}C until the next run.

9.10 Identification of CB's, OC's and PCB's

9.10.1 Start offline GC ChemStation to view chromatograms and save the Excel generated spreadsheet as special paste. The chromatograms and reports from capillary DB-5 Column are checked very carefully for symmetrical peak shape, for well-resolved and properly integrated peaks and edit the generated spreadsheet as per required. The sensitivity throughout the run, as indicated by the standard responses of the target compounds in the calibration standards, should not vary by more than ±20%. If excessive variation occurs, the instrumental analysis should be repeated, either from the beginning or from the point where the inconsistency occurred, after the problem has been corrected. Document the nonconformance and action taken to correct the problem on the analysis Nonconformance/Corrective Action Form.

9.10.2 A compound is reported as positively identified if the retention time compares with that of the GC Calibration Standard and the compound is detected using a retention time window of ± 0.05 min (see Figure 02-001-2: a, b, c for a typical GC/μECD chromatogram of CBs, OCs and PCBs).

9.10.3 Some OC Pesticides may split between the first two Fractions, (i.e., HCB, trans-Nonachlor, pp'- DDE). Therefore, both Fractions 1 and 2 must be checked for these chemicals.

10. Calculation of Results

1. Start MicroSoft Excel software
2. Open Master file for OCPCB; Directory C:\MyFiles\Sample DATA\file name
3. Copy and paste the Areas for each compound from the excel generated sheet (generated after the sequence was finished).
4. After entry complete review for correct entry at least 2 of each 10 values entered. Initial and date verified entry.
5. Spikes; 1) PCB #138 Area as Arochlors (1242:1254)
   2) PCB #180 Area as Arochlors (1260)
6. Save file as bench name and date.

The quantification of each compound is done by comparing the sample-peak areas from each Fraction against the respective standard-peak areas, from the 3 following

Calibration Standard Solutions:

Std #1 (S1): Aroclors 1242:1254:1260 (1:1:1) in isooctane, to quantitate compounds in Fraction 1 or Certified Quebec PCB Standard (solution D; sum of PCBs conc: 58.2 ng/mL; Level III); as per client request.
Std #2 (S2): 18 Organochlorine compounds in isooctane, to quantitate compounds in Fraction 2.
Std #3 (S3): Heptachlor Epoxide and Dieldrin in isooctane, to quantitate these 2 compounds in Fraction 3.

The spreadsheet file used for calculating concentrations data is maintained on the Lab office PC, Room 220. Using the Excel software [Directory C:\MyFiles\Sample DATA\file name] spreadsheet, the following data arising from sample analysis are implemented:

- sample ID, sample size, final volume (per each fraction)
- name of each analyte, corresponding retention time, corresponding concentration in respective calibration standard

The calculation of results is based on the following formula and in ng/g, reported in µg/kg:

C par. = (A.sam/A.std) * Std C.par* (V/W)

Where:
C_{param} = concentration of specific parameter in sample extract
A_{sam} = peak area of the same parameter in the sample extract
A_{std} = peak area of the same parameter in the Standard solution
Std C_{param} = concentration of the same parameter in the Standard solution (ng/mL)
V = Final volume of sample extract - prior to GC/µECD analysis (mL)
W = Wet weight of sample extracted (g)

11. Reporting of Data

11.1 The following records of the analytical methods are maintained: analyst's daily working log book, internal audit trail sheets, analysis nonconformance forms, and instrument log books.

11.2 Copy and save the analytical data in external hard drive Iomega: F:\uECD data\Data, QA/QC data and report logs are maintained permanently in Organic Laboratory in Excel (2003) in the PC of Lab Supervisor's office by GLIER Administration Office.

11.3 Data are reported to the client in final report form, using up to two significant figures and concentration units of ug/kg.

12. Comments

12.1 Sample carryover may occur from a sample with a very high concentration to a sample with a very low concentration. If this occurs, a Nonconformance Form must be recorded and action taken (i.e. the low concentration sample must be reanalyzed).

12.2 If the calculated value of any of the CBs, OCs, or PCBs is found to be more than 10 times higher than the calibration level, a dilution must be performed. Dilutions that give values close to the Calibration Standard level are the most accurate.

13. References

1. P. H. Howard, *Fate and Exposure Data for Organic Chemicals*. Lewis Publishers, Inc. Chelsea, Maryland, 1989, p. 230.
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APPENDIX A

Tables

Table 02-001-1: List of CBs, OCs and PCB's, providing Instrument Detection Limit (IDL), Calibration (Working) Linear Range and Coefficient of Determination (R^2)

Parameter	Rt (min)	IDL ng/mL	Calibration/Working Range ng/mL (Levels): L0; L1; L2; L3; L4; L5; L6	R^2 (May/2008)
1,2,4,5-Tetrachlorobenzene (1,2,4,5 - TCB)	8.29	0.092	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9970
1,2,3,4- Tetrachlorobenzene (1,2,3,4- TCB)	8.679	0.040	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9995
Pentaclorobenzene (QCB)	9.911	0.025	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9994
Hexachlorobenzene (HCB)	11.922	0.022	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9994
Octachlorostyrene (OCS)	11.698	0.017	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9995
pp'-DDE	19.304	0.019	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9983
Mirex	19.478	0.028	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9978
a-BHC	11.698	0.014	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9985
b-BHC	12.273	0.044	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9996
g-BHC	12.464	0.016	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9987
oxy-Chlordane	17.054	0.020	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9971
trans-Chlordane	17.903	0.018	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9981
cis-Chlordane	18.515	0.019	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9989
pp'-DDD	18.715	0.026	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9984
cis-Nonachlor	21.395	0.018	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9990
pp'-DDT	22.868	0.026	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9977
HC Epoxide	16.996	0.018	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9971
Dieldrin	17.054	0.017	0 ;0.2; 1.0; 2.0; 10.0; 20.0; 60.0	0.9978
PCB #18/17	12.66/12.72	0.131	0.08-249.20	0.9978
PCB #28/31	13.89/13.94	0.065	0.11-351.10	0.9988
PCB #33	14.231	0.077	0.0634-198.2	0.9986
PCB #52	14.977	0.098	0.0634-198.4	0.9986
PCB #49	15.131	0.073	0.0633-198.0	0.9987
PCB #44	15.685	0.060	0.0633-198.0	0.9988
PCB #74	16.964	0.050	0.064-200.0	0.9991
PCB #70	17.084	0.057	0.0634-198.2	0.9994

PCB #95	17.262	0.068	0.0320-100.2	0.9985
PCB #101	18.141	0.061	0.0643-201.0	0.9989
PCB #99	18.36	0.050	0.0634-198.0	0.9989
PCB #87	19.238	0.035	0.0638-199.4	0.9988
PCB #110	19.667	0.039	0.0637-199.0	0.9990
PCB #151/82	20.198	0.043	0.0801-250.5	0.9990
PCB #149	20.713	0.056	0.0633-197.8	0.9981
PCB #118	20.791	0.046	0.0641-200.2	0.9988
PCB #153	21.793	0.046	0.0635-198.4	0.9987
PCB # 105/132	21.949	0.038	0.0478-149.5	0.9979
PCB #138	23.098	0.035	0.0645-201.6	0.9990
PCB #158	23.221	0.029	0.0161-50.20	0.9977
PCB #187	23.906	0.043	0.0635-198.4	0.9988
PCB #183	24.152	0.035	0.0633-198.0	0.9961
PCB #128	24.406	0.027	0.0643-200.8	0.9988
PCB #177	25.298	0.037	0.0640-200.0	0.9988
PCB #171/156	25.532	0.027	0.1271-397.4	0.9987
PCB #180	26.398	0.029	0.0638-199.4	0.9986
PCB #191	26.742	0.025	0.0640-200.0	0.9987
PCB #170	27.871	0.032	0.0634-198.0	0.9989
PCB #201	28.328	0.025	0.0480-150.0	0.9988
PCB #195/208	30.05/30.11	0.042	0.1281-400.4	0.9985
PCB #194	31.135	0.023	0.0641-200.2	0.9974
PCB #205	31.425	0.023	0.0638-199.4	0.9985
PCB #206	33.067	0.025	0.064-200.0	0.9983
PCB #209	34.643	0.029	0.064-200.0	0.9984

Table 02-001-2: Concentration of Analytical Standards*

Parameter	Intermediate Std. ng/mL (isooctane)	Method Spiking Std. ng/mL (isooctane)	Calibration Std.** ng/mL (isooctane)
1,2,4,5-TCB	20.00	20.00	2.00
1,2,3,4-TCB	20.00	20.00	2.00
QCB	20.00	20.00	2.00
HCB	20.00	20.00	2.00
OCS	20.00	20.00	2.00
tr-Nonachlor	20.00	20.00	2.00
pp'-DDE	20.00	20.00	2.00
Mirex	20.00	20.00	2.00
a-BHC	20.00	20.00	2.00
b-BHC	20.00	20.00	2.00
g-BHC	20.00	20.00	2.00
oxy-Chlordane	20.00	20.00	2.00
tr-Chlordane	20.00	20.00	2.00
cis-Chlordane	20.00	20.00	2.00
pp'-DDD	20.00	20.00	2.00
cis-Nonachlor	20.00	20.00	2.00
pp'-DDT	20.00	20.00	2.00
Heptachlor Epoxide	20.00	20.00	2.00
Dieldrin	20.00	20.00	2.00
Total PCBs (Quebec PCBs)	1455.9	1455.9	58.2
1,3,5-TriBromobenzene***	125.00	125.00	2.50

*Refer to Related Procedures for Preparation of Analytical Standards
**This Calibration Standard is the 1X - Calibration Level (for Tables 02-001-4,5,6)
***Surrogates

Table 02-001-3: Analytical Tray Pattern*

Position #	Sample ID/Sample Type
1	Reagent Blank (Solvent)
2	Method Blank / Surrogate - Faction 1
3	Method Blank - Fraction 2
4	Method Blank - Fraction 3
5	Surrogate Standard
6	Calibration Standard (S1)
7	Reference Sample - Fraction 1 (or SRM)** (or Sample Duplicate)
8-12	Unknown Samples - Fraction 1***
13	Calibration Standard (S2)
14	Reference Sample - Fraction 2 (or SRM)**(or Sample Duplicate)
15-19	Unknown Samples - Fraction 2***
20	Calibration Standard (S3)
21	Reference Sample - Fraction 3 (or SRM)** (or Sample Duplicate)
22-26	Unknown Samples - Fraction 3 ***

*Refer to Section 9.8.2
**SRM: Standard Reference Materials (if available)
***Including duplicate (if available)

Table 02-001-4:
PERFORMANCE EVALUATION DATA for SOP02-001(OC s& PCBs in BIOTA) (MARCH /2008)

Compound	Spike Level (ug/kg)	n	AVG. Mean observed conc. (ug/kg)	S.D. Standard Deviation (ug/kg)	RSD Relative Standard Deviation (%)	Mean Accur (percent of t conc.) (%)	Recovery (%)	*MDL (3*S Method Detection l (ug/kg)	MQL Method Quantific Level (ug/kg)
1,2,4,5-TCB	0.2	6	0.195	0.026	13.46	-2.59	97.41	0.079	0.6
1,2,3,4-TCB	0.2	6	0.197	0.013	6.66	-1.27	98.73	0.039	0.6
QCB	0.2	6	0.218	0.010	4.52	9.24	109.24	0.030	0.6
HCB	0.20	6	0.198	0.018	9.27	-0.92	99.08	0.055	0.6
OCS	0.20	6	0.203	0.008	4.08	1.48	101.48	0.025	0.6
Trans-Nonachlor	0.20	6	0.228	0.008	3.71	14.21	114.21	0.025	0.6
p,p'-DDE	0.20	6	0.222	0.024	10.96	10.97	110.97	0.073	0.6
mirex	0.20	6	0.217	0.006	2.74	8.40	108.40	0.018	0.6
a-BHC	0.20	6	0.192	0.011	5.64	-4.15	95.85	0.032	0.6
b-BHC	0.20	6	0.216	0.023	10.57	7.86	107.86	0.068	0.6
g-BHC	0.20	6	0.228	0.016	7.06	14.04	114.04	0.048	0.6
oxy-Chlordane	0.20	6	0.218	0.025	11.55	9.12	109.12	0.076	0.6
Trans-nonachlor	0.20	6	0.184	0.017	9.24	-7.76	92.24	0.051	0.6
cis-Chlordane	0.20	6	0.207	0.013	6.43	3.38	103.38	0.040	0.6
pp'-DDD	0.20	6	0.209	0.004	1.69	4.27	104.27	0.011	0.3
cis-Nonachlor	0.20	6	0.208	0.016	7.91	3.87	103.87	0.049	0.6
pp'-DDT	0.20	6	0.194	0.011	5.53	-2.97	97.03	0.032	0.6
HC Epox	0.20	6	0.228	0.008	3.71	14.21	114.21	0.025	0.3
dieldrin	0.20	6	0.214	0.016	7.33	6.82	106.82	0.047	0.6
PCB #31/28	0.421	6	0.415	0.025	6.11	-1.59	98.41	0.076	0.6
PCB #52	0.238	6	0.240	0.013	5.24	0.90	100.90	0.038	0.6
PCB #49	0.238	6	0.251	0.009	3.56	5.58	105.58	0.027	0.3
PCB #44	0.238	6	0.236	0.020	8.46	-0.64	99.36	0.060	0.6
PCB #70	0.238	6	0.234	0.008	3.38	-1.87	98.13	0.024	0.3
PCB #95	0.120	6	0.131	0.021	15.79	8.67	108.67	0.062	0.6
PCB #101	0.241	6	0.284	0.030	10.54	17.57	117.57	0.090	0.6
PCB #99	0.238	6	0.247	0.015	5.97	3.87	103.87	0.044	0.6
PCB #87	0.239	6	0.252	0.007	2.89	5.20	105.20	0.022	0.3
PCB #110	0.239	6	0.247	0.026	10.32	3.55	103.55	0.076	0.6
PCB #151/82	0.060	6	0.057	0.008	13.45	-3.88	96.12	0.023	0.3
PCB #149	0.237	6	0.262	0.018	6.84	10.39	110.39	0.054	0.6
PCB #118	0.240	6	0.260	0.021	8.23	8.17	108.17	0.064	0.6
PCB153	0.238	6	0.225	0.024	10.48	-5.58	94.42	0.071	0.6
PCB #105/132	0.179	6	0.160	0.025	15.45	-10.68	89.32	0.074	0.6
PCB 138	0.242	6	0.263	0.008	2.94	8.83	108.83	0.023	0.3
PCB #187	0.238	6	0.252	0.021	8.34	5.99	105.99	0.063	0.6
PCB #183	0.238	6	0.242	0.017	6.91	1.96	101.96	0.050	0.6
PCB #128	0.241	6	0.218	0.015	6.67	-9.59	90.41	0.044	0.6
PCB 177	0.240	6	0.220	0.022	9.89	-8.30	91.70	0.065	0.6
PCB #156/171	0.477	6	0.514	0.042	8.08	7.75	107.75	0.124	0.6
PCB 180	0.239	6	0.267	0.016	6.00	11.65	111.65	0.048	0.6
PCB #170	0.240	6	0.251	0.025	10.11	4.66	104.66	0.076	0.6
PCB 201	0.180	6	0.188	0.012	6.47	4.23	104.23	0.036	0.6
PCB #195/208	0.480	6	0.509	0.025	4.82	5.92	105.92	0.074	0.6
PCB #194	0.240	6	0.270	0.013	4.81	12.28	112.28	0.039	0.6
PCB #205	0.239	6	0.245	0.008	3.16	2.49	102.49	0.023	0.3
PCB #206	0.24	6	0.246	0.020	8.33	2.41	102.41	0.061	0.6
PCB #209	0.24	6	0.254	0.013	5.27	5.90	105.90	0.040	0.6
ΣPCBs	8.0144		8.2726						
1,3,5-TriBrBenzene							93.41		

*MDL=SD*99% t-value
Σ PCBs: Sum of PCBs
Authorization OC/PCB's in Biota (02-001) is fit for its intended use _____
Lab Supervisor Date

Table 02-001-5:
PERFORMANCE EVALUATION DATA for SOP02-001(OC s& PCBs in BIOTA) (JULY/ 2008)

Compound	Spike Level (ug/kg)	n	AVG. Mean observed conc. (ug/kg)	S.D. Standard Deviation (ug/kg)	RSD Relative Standard Deviation (%)	Mean Accuracy (percent of true (%)	Recovery (%)
1,2,4,5-TCB	2.00	6	1.975	0.2194	11.11	-1.23	98.77
1,2,3,4-TCB	2.00	6	2.109	0.2117	10.04	5.45	105.45
QCB	2.00	6	1.960	0.1205	6.15	-2.01	97.99
HCB	2.00	6	2.036	0.1733	8.51	1.82	101.82
OCS	2.00	6	2.073	0.0399	1.92	3.63	103.63
Trans-Nonachlor	2.00	6	1.961	0.0586	2.99	-1.97	98.03
pp'-DDE	2.00	6	2.097	0.0462	2.20	4.87	104.87
Mirex	2.00	6	2.057	0.0402	1.95	2.84	102.84
a-BHC	2.00	6	1.808	0.1516	8.39	-9.61	90.39
b-BHC	2.00	6	1.893	0.1564	8.26	-5.37	94.63
g-BHC	2.00	6	1.951	0.1739	8.91	-2.43	97.57
oxy-Chlordane	2.00	6	1.765	0.1446	8.19	-11.76	88.24
trans-Chlordane	2.00	6	1.789	0.1243	6.95	-10.56	89.44
cis-Chlordane	2.00	6	2.092	0.1288	6.16	4.61	104.61
pp'-DDD	2.00	6	1.792	0.1246	6.96	-10.39	89.61
cis-Nonachlor	2.00	6	1.827	0.1144	6.26	-8.64	91.36
pp'-DDT	2.00	6	2.145	0.1235	5.76	7.27	107.27
HC Epox	2.00	6	2.032	0.0380	1.87	1.61	101.61
Dieldrin	2.00	6	2.120	0.1157	5.46	5.99	105.99
PCB #31/28	2.809	8	2.582	0.281	10.90	-8.069	91.931
PCB #52	1.587	8	1.705	0.0675	3.96	7.42	107.42
PCB #49	1.584	8	1.672	0.085	5.09	5.578	105.58
PCB #44	1.584	8	1.740	0.157	9.05	9.818	109.82
PCB #70	1.587	8	1.808	0.053	2.91	13.880	113.88
PCB #95	0.802	8	0.863	0.077	8.88	7.628	107.63
PCB #101	1.608	8	1.773	0.069	3.91	10.267	110.27
PCB #99	1.587	8	1.673	0.089	5.32	5.402	105.40
PCB #87	1.595	8	1.614	0.033	2.074	1.183	101.18
PCB #110	1.592	8	1.604	0.096	5.997	0.757	100.76
PCB #151/82	2.004	8	1.960	0.0642	3.28	-2.21	97.79
PCB #149	1.582	8	1.691	0.055	3.223	6.892	106.89
PCB #118	1.602	8	1.707	0.053	3.084	6.599	106.60
PCB #153	1.587	8	1.741	0.047	2.685	9.690	109.69
PCB #105/132	1.196	8	1.121	0.086	7.645	-6.295	93.71
PCB #138	1.613	8	1.708	0.1257	7.36	5.93	105.93
PCB #158	0.402	8	0.383	0.0299	7.81	-4.74	95.26
PCB #187	1.587	8	1.746	0.106	6.049	10.003	110.00
PCB #183	1.584	8	1.645	0.028	1.725	3.853	103.85
PCB #128	1.606	8	1.476	0.095	6.455	-8.103	91.90
PCB #177	1.600	8	1.654	0.039	2.351	3.393	103.39
PCB #156/171	3.179	8	3.644	0.120	3.291	14.616	114.62
PCB #180	1.595	8	1.650	0.032	1.954	3.457	103.46
PCB #170	1.584	8	1.635	0.042	2.567	3.231	103.23
PCB #201	1.200	8	1.233	0.084	6.837	2.728	102.73
PCB #195/208	3.203	8	3.236	0.101	3.136	1.026	101.03
PCB #194	1.602	8	1.608	0.045	2.802	0.388	100.39
PCB #205	1.595	8	1.564	0.049	3.160	-1.960	98.04
PCB #206	1.600	8	1.572	0.046	2.952	-1.751	98.25
PCB #209	1.600	8	1.592	0.048	3.026	-0.516	99.48
ΣPCBs	55.036		56.9781				
1,3,5-TriBrBenzene							96.37

*MDL=SD*99% t-value
Σ PCBs: Sum of PCBs

Authorization OC/PCB's in Biota (02-001) is fit for its intended use _____
Lab Supervisor Date

Table 02-001-6: Uncertainty of Measurement (C.I. = 99%, K=2)
In-House Control Sample, **DCCO Egg Pool**

Parameter	n (pop'n)	Mean (ug/kg wet)	Uncertainty = 3*Std Dev (ug/kg wet)
(1,2,4,5 - TCB)	4		
(1,2,3,4- TCB)	4	0.240	0.57
Pentaclorobenzene (QCB)	4	1.583	0.55
Hexachlorobenzene (HCB)	4	14.315	8.88
Octachlorostyrene (OCS)	4	3.601	1.24
tr-Nonachlor	4	0.722	1.78
pp'-DDE	4	836.941	132.47
Mirex	4	56.420	9.68
a-BHC	4	0.246	0.17
b-BHC	4	1.153	0.17
g-BHC	4	0.333	0.11
oxy-Chlordane	4	6.781	3.51
trans-Chlordane	4	1.246	1.59
cis-Chlordane	4	0.970	1.07
pp'-DDD	4	1.343	0.33
cis-Nonachlor	4	4.005	1.05
pp'-DDT	4	3.211	1.73
HC Epoxide	4	6.906	4.93
Dieldrin	4	16.554	3.91
PCB #18/17	4		
PCB #28/31	4	12.573	4.19
PCB #33	4		
PCB #52	4	3.498	0.59
PCB #49	4	2.558	0.54
PCB #44	4	2.997	0.25
PCB #64	4	0.868	0.49
PCB #74	4	19.111	7.42
PCB #70/76	4	2.462	0.58
PCB #95/66	4	22.751	30.35

PCB #101	4	7.764	1.18
PCB #99	4	66.112	17.60
PCB #97	4	0.240	0.43
PCB #110	4	4.618	0.00
PCB #151	4	2.207	0.85
PCB #149	4	8.029	2.89
PCB #118	4	91.442	28.52
PCB #146	4	47.808	17.10
PCB #153	4	274.202	156.96
PCB # 105	4	0.315	1.26
PCB #141	4	4.222	1.32
PCB #179	4		
PCB #138	4	186.262	36.11
PCB #158	4	9.045	6.49
PCB #129/178	4		
PCB #182/187	4	93.573	42.62
PCB #183	4	39.795	9.80
PCB #128	4	48.929	20.53
PCB #174	4	3.271	0.72
PCB #177	4	24.949	2.26
PCB #171/156	4	36.813	4.93
PCB #200	4	8.349	3.51
PCB #172	4	17.001	6.21
PCB #180	4	176.989	74.31
PCB #170/190	4	65.349	10.09
PCB #201	4	43.730	12.33
PCB #203/196	4	42.666	12.56
PCB #195	4	14.969	2.75
PCB #194	4	34.468	10.92
PCB #206	4	17.498	5.17

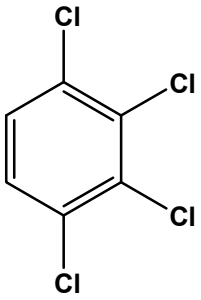
- This method is validated for the intended use.
Analysis Period (mth/yr): August 2007 - March 2008
Data File: OrgLabC:/MyFiles/GLIERManual/SOP_SRMMeanandSD/02-001CWSEggPool.xls
via Log of Data: OrgLabC:/MyFiles/Org.LabData/OrganicLIMS.St./uncertainty/SOP02-001/Bio.Ref.material.xls

APPENDIX B

Figures

Figure 02-001-1a

TETRACHLOROBENZENE

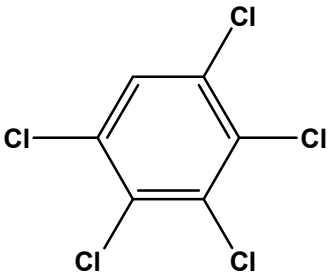


1,2,3,4-Teterachlorobenzene

mol. formula : $C_6H_2Cl_4$

mol. wt. : 216

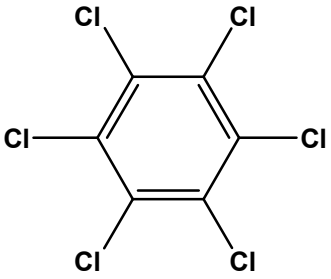
PENTACHLOROBENZENE



mol. formula : $C_6H_1Cl_5$

mol. wt. : 250

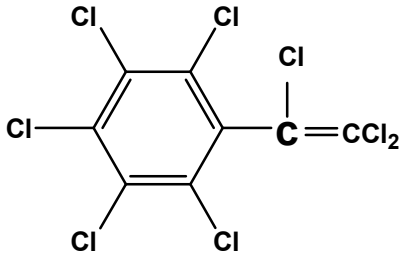
HEXACHLOROBENZENE



mol. formula : C_6Cl_6

mol. wt. : 285

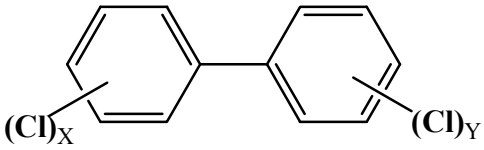
OCTACHLOROSTYRENE



mol. formula : C_8Cl_8

mol. wt. : 284

PCB



Aroclor 1242

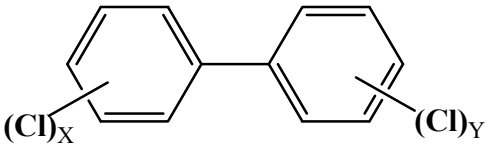
mol. formula: $C_{12}H_{10-x}Cl_x$

% of Chlorine: 42

X+Y = 2

X+Y = 3

X+Y = 4



Aroclor 1254

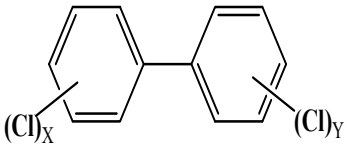
mol. formula: $C_{12}H_{10-x}Cl_x$

% of Chlorine: 54

X+Y = 4

X+Y = 5

X+Y = 6



Aroclor 1260

mol. formula: $C_{12}H_{10-x}Cl_x$

% of Chlorine: 60

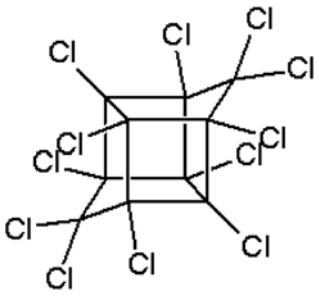
X+Y = 5

X+Y = 6

X+Y = 7

X+Y = 8

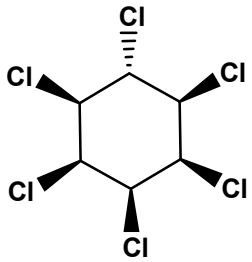
Figure 02-001-1b



mirex

mol. formula : $C_{10}Cl_{12}$

mol. wt. : 546

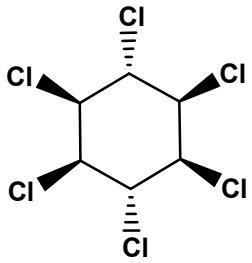


a-BHC isomer & b-BHC isomer;

1,2,3,4,5,6-hexachlorocyclohexane

mol. formula : $C_6H_6Cl_6$

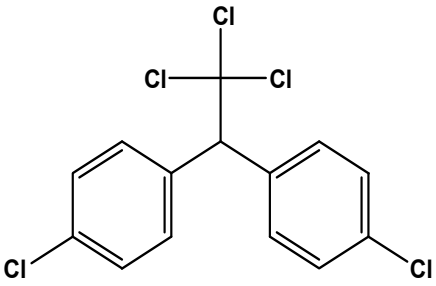
mol. wt. : 291



g -BHC isomer

mol. formula : $C_6H_6Cl_6$

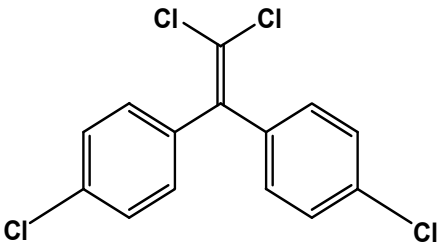
mol. wt. : 291



DDT p, p' isomer

mol. formula : $C_{14}H_9Cl_5$

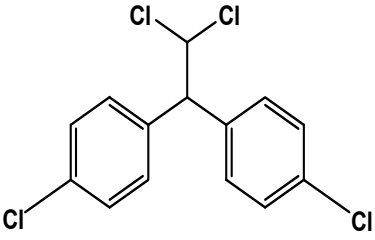
mol. wt. : 354



DDE p, p' isomer

mol. formula : $C_{14}H_8Cl_4$

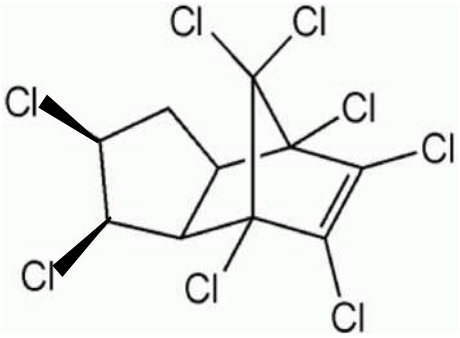
mol. wt. : 318



DDD p, p' isomer

mol. formula : $C_{14}H_{10}Cl_4$

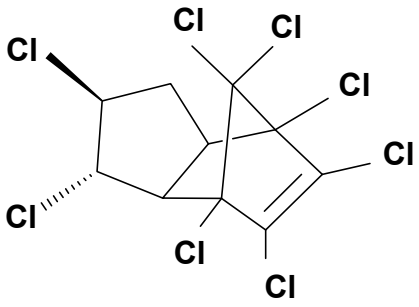
mol. wt. : 320



***cis*-Chlordane**

mol. formula : $C_{10}H_6Cl_8$

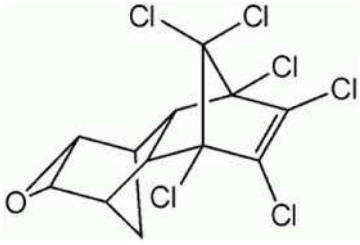
mol. wt. : 410



***trans*-Chlordane**

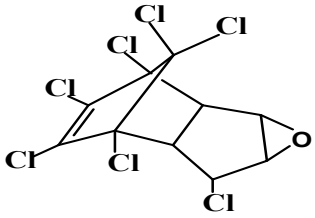
mol. formula : $C_{10}H_6Cl_8$

mol. wt. : 410



Dieldrin

MF: $C_{12}H_8Cl_6O$ MW: 381



Heptachlor epoxide

MF: $C_{10}H_5Cl_7$ MW: 373

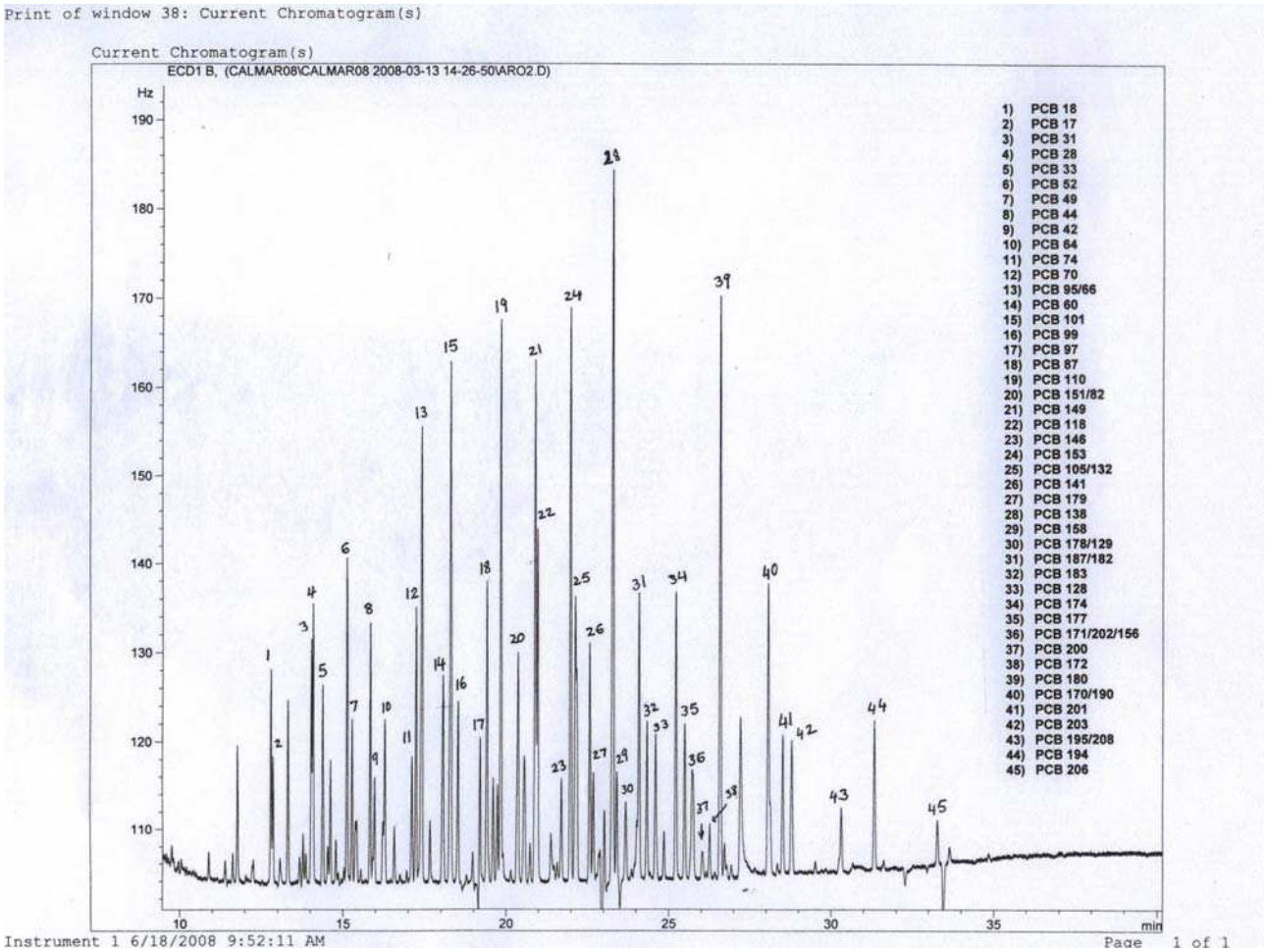


Figure 02-001-2a: Chromatogram of Aroclor 1242:1254:1260 (1:1:1) Standard 1 (S1) recorded on GC/uECD with a 60 m x0.25 x 0.1um DB-5 capillary column.

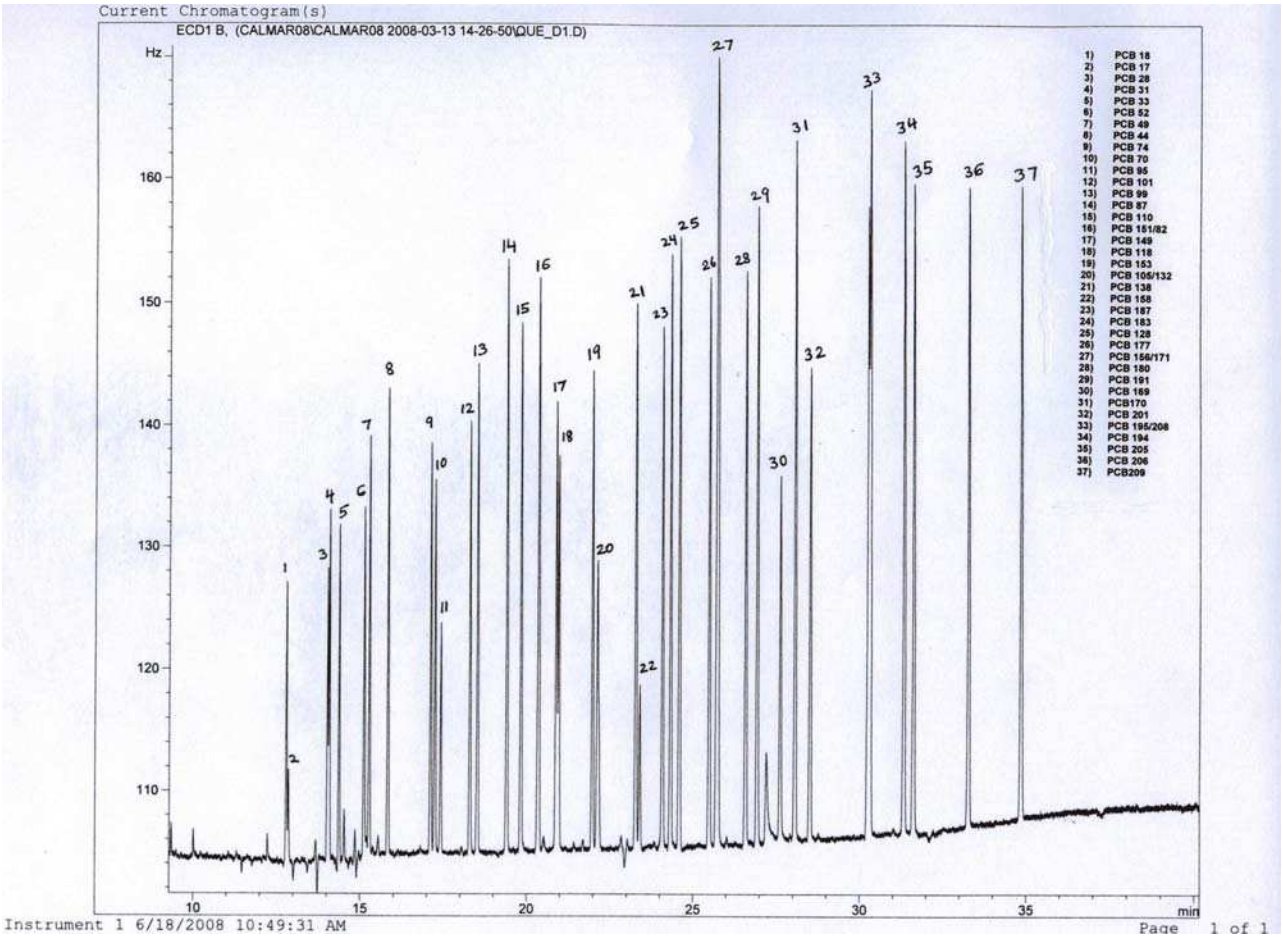


Figure 02-001-2b: Chromatogram of Quebec Ministry of Environment PCB Congener Mix recorded on GC/uECD with a 60 m x0.25 x 0.1um DB-5 capillary column.

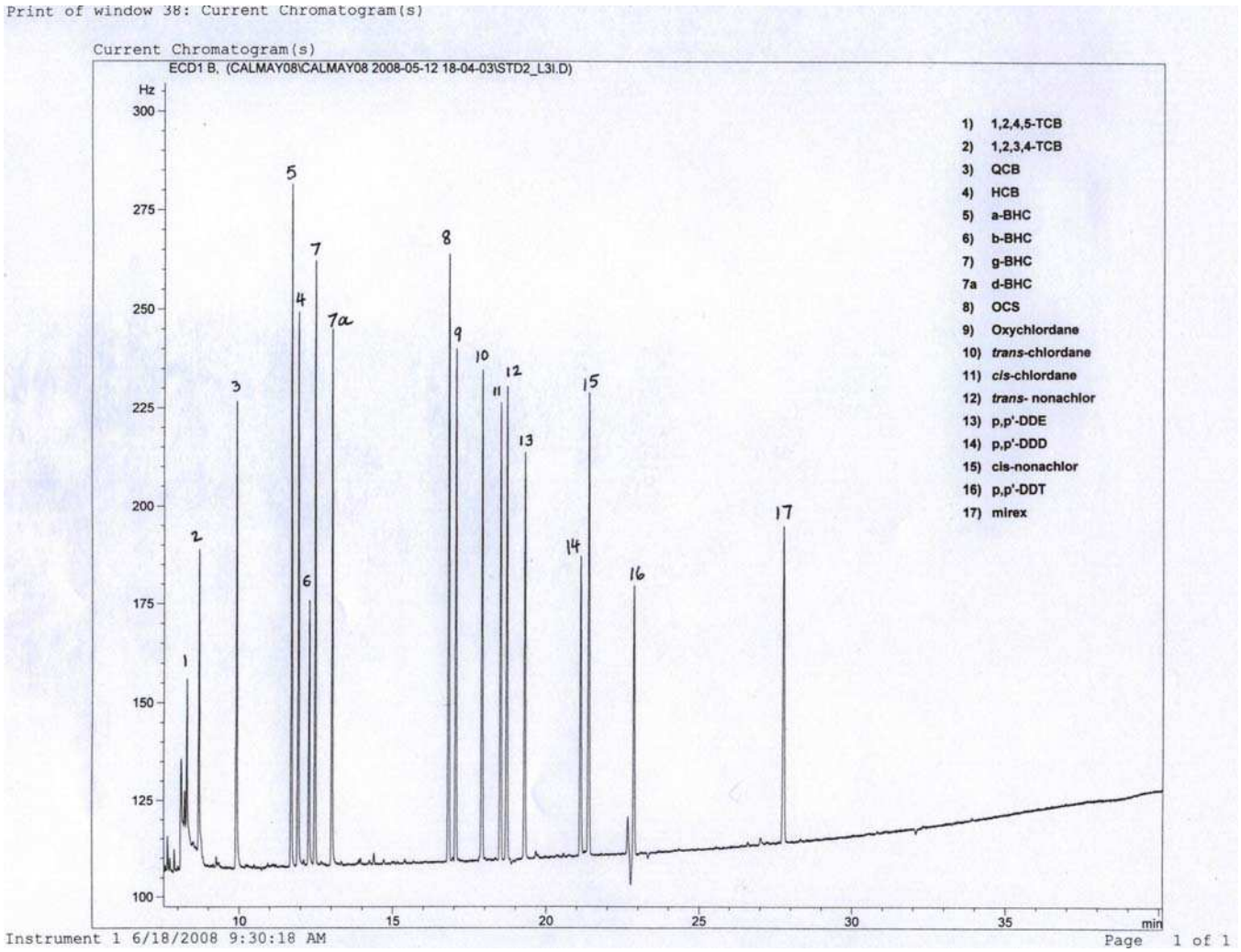


Figure 02-001-2c: Chromatogram of OCs Standard 2 (S2) recorded on GC/uECD with a 60 m x0.25 x 0.1um DB-5 capillary column.

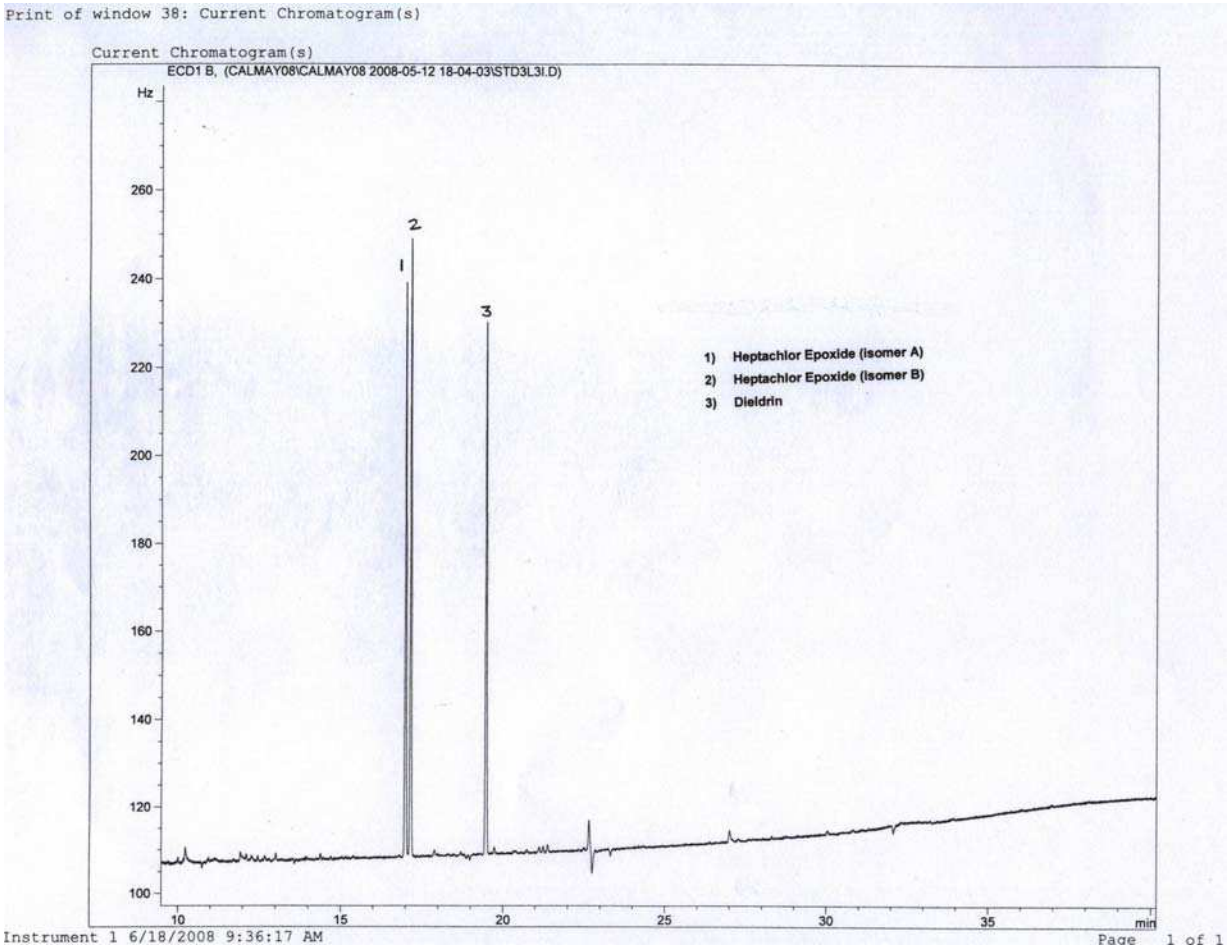


Figure 02-001-2d: Chromatogram of OCs Standard 3 (S3) recorded on GC/uECD with a 60 m x0.25 x 0.1um DB-5 capillary column.