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Title: Confirmation of Pesticides by GC/MS/MS		
Revision: Original	Replaces: NA	Effective: 08/30/2010

Contents

A. INTRODUCTION

1. Theory

Sample preparation and homogenization is performed by grinding adipose tissue with dry ice. Pesticide extraction is performed using solvent extraction into cyclopentane before gel permeation chromatography (GPC) cleanup and solvent exchange to isooctane.

Pesticides are confirmed by gas chromatography/ tandem quadrupole mass spectrometry (GC/MS/MS). Confirmation is based on comparison of sample GC retention time and product ion abundance ratios against those obtained for a reference standard or positive control.

2. Applicability

This method is applicable for the confirmation of the analytes listed below in adipose tissue at levels greater than or equal to the levels in Appendix Table 1:

Aldrin

BHC; alpha-, beta-, and delta-

Chlordane; cis- (alpha-) and trans-

DDD (TDE); o,p'- and p,p'-

DDE; o,p'- and p,p'

DDT; o,p'- and p,p'-

Dieldrin

Endrin

HCB (Hexachlorobenzene)

Heptachlor

Heptachlor epoxide A & B

Lindane (gamma-BHC)

Methoxychlor

Mirex

Nonachlor; trans-

B. EQUIPMENT

Note: Equivalent equipment may be substituted.

1. Apparatus
   a. Food processor – Robot Coupe® model RSI6Y-1, Robot Coupe USA Inc.
   b. Sample cups – eValue 4.5 oz specimen containers w/caps, Cat. No. C686550, E&K Scientific.
   c. Polypropylene copolymer centrifuge tubes with sealing caps, Nalgene, 50 mL, Cat. No. 21010-578, VWR Intl.
   d. Shaker – Horizontal flatbed, two speed, Cat. No. 511105, Eberbach Corp.
   e. Freezer capable of -10 ºC.
   f. Centrifuge capable of 5,000 rcf – Avanti 30, Beckman.
   g. Syringe filters – 0.45 µm, Pall/Gelman nylon Acrodisc 25 mm, Cat. No. 28143-266, VWR Intl.
   h. Syringe, glass, luer-lock, 10 mL, Cat. No. 5027, Popper & Sons.
   i. GPC tubes and caps – 16x100 mm threaded glass tubes Cat. No. BV16100T-PK, 16 mm open top caps Cat. No. BV016, TFE septa, Cat. No. BV016T-PK, J2 Scientific.
   j. Glass autosampler vials and caps – 2 mL, Cat. No. E251036, amber, Cat. No. E251011, caps with septa, Cat. No. E416209, E&K Scientific.
   k. Volumetric Flasks – 5 mL, 25 mL, 100 mL, 1000 mL class A.
   l. Graduated Cylinders – 1 and 2 L.
   m. Pipettors and dispensers – Automatic pipettors and/or dispensers capable of accurately delivering volumes specified (10 – 1000 ± 0.1 µL, 5 mL, 8 mL), Eppendorf.
   n. Pasteur Pipettes – Cat. No. 13-678-20D, Fisher.
   o. Analytical Balance – Readable to 0.1 mg, Model AE160, Mettler.
2. Instrumentation
   a. GPC system with auto-evaporation capability – Accuprep MPS with Accuvap Inline Concentrator, J2 Scientific.
   b. GPC column – Optima, Cat. No. 624-123, O.I. Analytical Sample Preparation

Product Division.

c. GC/MS/MS – Varian 450 gas chromatograph equipped with Varian 300 triple quadruple mass spectrometer. Varian MS Workstation Software.

d. Analytical column – Varian VF 5ms 30m x 0.25mm ID.

C. REAGENTS AND SOLUTIONS

Note: Equivalent reagents and solutions may be substituted.

1. Reagents

a. Iso-octane – pesticide grade, Cat. No. 362-4, Burdick & Jackson.

b. Ethyl acetate – pesticide grade, Cat. No. 100-4, Burdick & Jackson.

c. Cyclopentane – pesticide grade, Cat. No. 057-4, Burdick & Jackson.

d. Magnesium Sulfate Anhydrous, Cat. No. M65-500, Fisher Chemical.

e. Sodium Sulfate SPE Cartridges, Cat. No. 6805-8020, Whatman.

2. Solutions

a. GPC Mobile Phase – Ethyl acetate:Cyclopentane 70:30 (v/v):

Using a graduated cylinder, measure 700 mL of ethyl acetate and combine with 300 mL of cyclopentane or use this ratio to make an appropriate volume. Combine in a suitable bottle.

b. GPC Rinse Solvent:

GPC mobile phase, redistilled mobile phase, ethyl acetate, or cyclopentane.

c. GPC Diluent – dibutyl chlorendate (DBC) internal standard (ISTD) at 0.025 μg/mL in isooctane.

D. STANDARDS

Note: Equivalent standards may be substituted.

1. Source of stock standards:

All pesticide standards can be purchased from Ultra Scientific., Chemservice or other companies which can provide certificates of analysis.

a. DBC stock solution 100 μg/mL in iso-octane.

b. Aldrin stock solution 100 μg/mL in iso-octane.

c. Custom mix of pesticide standard or single pesticide solution at 100-1000 μg/mL in iso-octane, methanol or acetone.

The custom mix of pesticide standard can be ordered with the following concentrations as an example:

Analytes	CAS #	Concentration in the Custom Mix (µg/mL in Isooctane)	Fort Std Concentration (µg/mL in Isooctane)
Aldrin	000309-00-2	75	3
BHC, alpha-	000319-84-6	50	2
BHC, beta-	000319-85-7	50	2
BHC, delta-	000319-86-8	50	2
Chlordane, cis- or alpha	005103-71-9	30	1.2
Chlordane, trans-	005103-74-2	30	1.2
DDD, o,p'-	000053-19-0	500	20
DDD, p,p'-	000072-54-8	500	20
DDE, o,p'-	003424-82-6	500	20
DDE, p,p'-	000072-55-9	1250	50
DDT, o,p'-	000789-02-6	500	20
DDT, p,p'-	000050-29-3	500	20
Dieldrin	000060-57-1	75	3
Endrin	000072-20-8	25	1
HCB (Hexachlorobenzene)	000118-74-1	25	1
Heptachlor	000076-44-8	33.5	1.34
Heptachlor Epoxide A	001024-57-3	33.5	1.34
Heptachlor Epoxide B	001024-57-3	33.5	1.34
Lindane (BHC, gamma-)	000058-89-9	25	1
Methoxychlor	000072-43-5	125	5
Mirex	002385-85-5	50	2
Nonachlor, trans-	039765-80-5	30	1.2

2. Preparation

   a. Working Standards:

      i. Mixed Fortification Standard:

      Pipet 1.0 mL of the stock solution (D.1.c) into a class A 25-mL volumetric flask, dilute to volume with iso-octane. Mix well.

      A solution of a single compound of interest can be prepared instead of the solution of mixed fortification standard.

      ii. Aldrin Process Control (PC) Fortification Standard (2 µg/mL):

Pipet 2.0 mL of the 100 μg/mL aldrin stock solution (D.1.b) into a class A 100-mL volumetric flask and dilute to volume with iso-octane. Mix well.

b. GPC Diluent 0.025 μg/mL DBC (di-butyl chlorendate) solution:

Pipet 0.25 mL of the DBC stock solution (D.1.a) into a class A 1000-mL volumetric flask, dilute to volume with iso-octane and mix well.

c. Injection Standard:

Fill a 5-mL class A volumetric flask with GPC diluent (D.2.b). The diluent is evaporated under nitrogen far enough so that solvent based standards may be added. 78.1 µL of the aldrin process control standard (D.2.a.ii) and 78.1 µL of mixed fortification standard solution (D.2.a.i) are added to the 5 mL volumetric flask, dilute to volume with iso-octane, and mix well.

3. Storage and Stability:

a. Store stock standards in tightly sealed glass containers below -10 ºC. Stock standards stored this way are stable for three years.

b. Store solvent-based working standards in tightly sealed glass containers below -10 ºC. Working standards stored this way are stable for 2.5 years. It is advisable to store standards in amber glass to avoid degradation of light-sensitive compounds.

c. Injection standards are stable for 3 months when stored at room temperature. New injection standards should also be prepared whenever new GPC diluent or new fortification standards are prepared.

E. SAMPLE PREPARATION

Chop approximately 1 lb of adipose tissue into small pieces approximately 2.5 cm square and homogenize with an equal amount of dry ice in a large food processor. The resulting sample homogenate will be a frozen powder.

Transfer a portion of the homogenized sample into a loosely capped sample cup and place in a -10ºC or lower freezer such that the dry ice is allowed to sublime.

Remove the sample cups from the -10 ºC freezer and tighten the caps. If not extracting immediately, store frozen.

F. ANALYTICAL PROCEDURE

1. Extraction

Note: Confirmatory sample sets require a tissue blank and a fortified recovery at approximately the same concentration as the sample to be confirmed or at the tolerance level.

a. Weigh 1.0 ± 0.1 g homogenized adipose tissue into a 50 mL centrifuge tube. Prepare one positive and one negative controls at this time by weighing two 1 g blank homogenized samples and fortifying one at the level of interest or at the screening level by adding 50 µL of the mixed fortification standard (D.2.a.i) (see table in D.1.c. for levels of fortifications).

b. Add 50 µL of the aldrin process control fortification standard (D.2.a. ii.) to each sample.

c. Add 8 mL of cyclopentane to each sample.

d. Add anhydrous magnesium sulfate until there is no visible clumping at this point or a sodium sulfate cartridge can be used immediately after step f.

e. Agitate well for 5 minutes.

f. Centrifuge in a high-speed centrifuge for 5 minutes at approximately 5,000 rcf (or enough to pack down solids sufficiently so that at least 5 mL of solvent can be recovered in the next step).

g. Filter the extract through a 0.45 µm disposable PTFE syringe filter and place 5 mL of filtered extract in a GPC tube for analysis. Rinse the syringe with ethyl acetate or cyclopentane between samples.

2. GPC Cleanup

Instrument Parameters:

Note: Initial column calibration may be performed as described in the GPC manual. Parameters may be adjusted to optimize system resolution and stability. Examples of typical operating parameters are listed below:

Injection Volume: 2.5 mL

Mobile Phase Flow Rate: 4.5 – 5 mL/min

Dump Time: 8.5 – 9.5 min

Collect Time: 9 – 10.5 min

Pre-spike (Keeper): 1 mL ethyl acetate

Vacuum Pressure: 250 – 300 torr

Evaporation Temperature: 51 – 58 ºC

Final Reconstituted Volume: 1 mL in GPC diluent

If a problem occurs with the evaporation section of the GPC, then manual evaporation using an N-evap or any other acceptable evaporator can be used.

3. Instrumental Operating Conditions:

Note: The instrument parameters listed here may be optimized.

a. Gas Chromatograph Parameters:

Carrier Gas:	Helium
Column Flow Rate	1.0 mL/min
Injector Temperature	260 °C
Injection Volume	1 µL
Injection Mode	splitless
Temperature Program:	
Initial temp:	75 °C
Initial hold time	2 min
Program rate up to 150 °C	25 °C/min
Program rate up to 225 ºC	3 °C/min
Program rate up to 300 °C	15 °C/min
Final hold time	10 min
Total Run time	45.0 min

b. Mass Spectrometer Parameters:

Ionization	Positive Electron Impact
Detector Voltage	1250 V
Collision Gas	Argon @ 1.5 mTorr
Collision Energy	Optimized for each compound
MS Source temperature	200 °C
Transferline temperature	280 °C
Acquisition delay	6.0 min.

Note: Autotune the instrument as needed.

c. Summary of Multiple Reaction Monitoring (MRM) transitions and parameters selected for each Compound:

Compound	RT (min)	First transition (m/z)	Coll En (V)	Second transition (m/z)	Coll En (V)	Third transition (m/z)	Coll En (V)	Quant Ion***	MW*
Aldrin	21.09	263 >191	40	293 >186	40	293 >257	10	191	362
BHC, alpha-	13.93	219 >183	10	219 >109	30	181 >145	10	183	288
BHC, beta-	15.14	219 >183	10	219 >109	30	181 >145	10	183	288
BHC, delta-	16.99	219 >183	10	219 >109	30	181 >145	10	183	288
Chlordane, cis-	25.41	373 >337	10	375 >266	10	375 >303	10	266	406
Chlordane, trans-	24.66	373 >337	40	375 >266	10	375 >303	10	266	406
DDD, o,p'-	27.27	235 >165	20	235 >200	30	199 >164	15	165	318
DDD, p,p'-	29.31	235 >165	20	235 >200	30	199 >164	15	165	318
DDE, o,p'-	24.97	246 >176	25	318 >246	20	318 >210	20	176	316
DDE, p,p'-	26.86	246 >176	25	318 >246	20	318 >210	20	176	316
DDT, o,p'-	29.31	237 >165	15	235 >199	30	282 >212	20	165	352
DDT, p,p'-	31.17	237 >165	15	235 >199	30	282 >212	20	165	352
Dieldrin	26.97	277 >241	10	263 >193	25	345 >263	15	241	378
Endrin	28.18	279 >243	10	281 >211	30	263 >191	40	243	378
HCB (Hexachlorobenzene)	14.03	284 >142	50	284 >214	40	284 >249	30	214	282
Heptachlor	19.17	272 >237	20	337 >266	20			237	370
Heptachlor Epoxide A **	23.58	183 >119	20	272 >237	20	353 >317	10	119	386
Heptachlor Epoxide B **	23.33	183 >119	20	272 >237	20	353 >317	10	317	386
Lindane (gamma-BHC)	15.54	219 >183	10	219 >109	30	181 >145	10	183	288
Methoxychlor	32.93	227 >141	35	227 >152	20	227 >212	20	141	344
Mirex	33.98	272 >237	20	272 >140	40	272 >167	40	237	540
Nonachlor, trans-	25.64	409 >302	10	409 >263	20	409 >109	30	302	440

* Molecular Weight (MW) is listed above as supplemental reference information only. The MWs are not monitored on the mass spectrometer.

** The retention times of heptachlor epoxide A and B are separated by less than 2%. This method confirms the presence of heptachlor epoxide, but does not distinguish which isomer.

*** The Quant. Ion listed above is not being used for quantification in this method. It is the most

abundant product ion of those listed for each analyte, under the stated instrument conditions.

4. Analyze Sample Set

   Recommended injection sequence for analysis set:

   a. Standard
   b. Solvent blank (if necessary)
   c. Negative tissue control
   d. Positive tissue control
   e. Solvent blank (if necessary)
   f. Sample
   g. Solvent blank (if necessary)
   h. Standard

   Note: Carry-over may be observed when a high concentration of pesticides is injected. It may be necessary to inject solvent after a high concentration of pesticides.

5. Confirmation of any analyte(s) presence in a sample extract requires the following criteria be met:

   a. The retention time of any peak(s) of interest should match that of the comparison standard within ± 2%. Either the injection standard or the fortified control may be chosen as the comparison standard.

      Note: The use of a fortified control made from the same tissue type as the sample is recommended as the matrix may affect the ion ratios.

      Note: The use of the fortified control as a comparison standard is recommended for Endrin confirmation because of strong matrix effects observed with this analyte.

   b. The Quant ion of interest should exceed a signal to noise (S/N) ratio of 10:1.

   c. If two structurally-significant product ions are monitored for the analyte of interest, the relative abundance ratio must match that of the comparison standard within ±10% absolute difference.

   d. If three structurally-significant product ions are monitored for the analyte of interest, the relative abundance ratios must match that of the comparison standard within ±20% absolute difference.

6. Sample Chromatograms

   The GC/MS/MS chromatograms and spectrums of all pesticides listed in this method are in Section K.

G. CALCULATIONS

None

H. SAFETY INFORMATION AND PRECAUTIONS

1. Required Protective Equipment - Safety glasses, laboratory coat, and nitrile gloves.

2. Hazards

Reagent	*Hazard*	*Recommended Safe Procedures*
Isooctane	Highly flammable, harmful and irritating to skin.	Use in a fume hood. Keep container in a well ventilated area. Wear gloves, safety glasses and protective clothing.
Pesticides	Poisonous and harmful. Hazardous to humans and environment.	Keep tightly closed. Use under the fume hood. Wear gloves, eye protection and protective clothing. Keep away from incompatibles.
Cyclopentane	Highly Flammable and irritant.	Use in a fume hood. Store in tightly closed container. Keep away from electrical devices and open flames. Wear gloves, protective clothing and safety glasses.
Ethyl Acetate	Highly Flammable and irritant to eyes.	Use in a fume hood. Keep away from heat, strong oxidizers, acids and bases. Wear gloves, safety glasses and protective clothing.
CO_2	Breathing air containing more than 10% CO_2 may cause loss of consciousness.	Maintain adequate ventilation.
Dry ice	Causes frostbite and/or blisters.	Use insulated gloves, safety glasses and protective clothing.

3. Disposal Procedures

Procedure Step	*Hazard*	*Recommended Safe Procedures*
Isooctane	Flammable and poisonous.	Collect waste in a clearly labeled, tightly sealed container. Store away from non-compatibles in a cool, well-ventilated flammable liquid storage area in accordance with local, state, and federal regulations.

Pesticides	Poisonous and harmful.	Collect waste in a clearly labeled, tightly sealed container. Follow local, state and federal regulations for storage and disposal. Some pesticides are P list compounds, and require special disposal procedures.
Cyclopentane	See section 2 above	Collect waste in a clearly labeled, tightly sealed container and store in well-ventilated storage area. Dispose in accordance with local, state, and federal regulations.
Ethyl Acetate	See section 2 above	Store in a tightly closed container. Store in a cool, well ventilated area away from incompatibles. Dispose in accordance to federal, state and local environmental regulations.

I. QUALITY ASSURANCE PLAN

1. Performance Standard
   a. Positive control is positive for the compound(s) of interest using the criteria in section F.5.
   b. Negative control is negative for the compound(s) of interest.
2. Critical Control Points and Specifications

	Record	*Acceptable Control*
a.	Sample must be finely ground	Visual inspection

3. Readiness to Perform
   a. Familiarization
      i. Phase I: Analyze an injection standard and a solvent blank over 3 days to ensure that instrument response is adequate for confirmation.
      ii. Phase II: Analyze one blank tissue and four fortified tissues over a period of three different days.

      NOTE: Phase I and Phase II may be performed concurrently.

iii. Phase III: Check samples for analyst accreditation.

(a) Analyze at least six blind check samples consisting of one or two unfortified tissues and the remainder fortified at or above the sensitivity of the method. Each check sample should contain at least three compounds: an early eluter, a late eluter and one in between. For example, alpha-BHC (early eluter), Mirex (late eluter) and p,p'-DDE (eluting in between).

(b) Report analytical findings to Quality Assurance Manager (QAM) and supervisor.

(c) Authorization by the QAM and Supervisor is required to commence official analysis.

b. Acceptability criteria.

Refer to I. 1.

4. Intralaboratory Check Samples

a. System, minimum contents.

i. Frequency: Once per week per analyst when analyses are run.

ii. Records are to be maintained

b. Acceptability criteria.

Refer to I. 1.

If unacceptable values are obtained, then:

i. Stop all official analyses by that analyst with this method.

ii. Take corrective action.

5. Sample Acceptability and Stability

a. Matrix: Adipose tissue.

b. Sample storage: Homogenized tissue should be stored frozen.

6. Analyst Capability

Minimum proficiency level (MPL): See Appendix Table 1

J. WORKSHEET

Pesticide Screening Worksheet

Analyst	
Start Date	
End Date	
Peer Review/Date	
Supervisor Review/Date	

Sample Storage Freezer	
Sample Prep Logbook	
Date Ground	
Grinder	
Sample Weight Logbook	
Date Weighed	
Balance	
Centrifuge	
GPC Logbook or GPC	
Date run on GPC	
GC Logbook or GC	
Date run on GC	

Reagents and Standards	ID	Pipet/Disp
Mixed Fortification Standard		
Aldrin Fortification Standard		
Cyclopentane		N/A
GPC Diluent		N/A
Mixed Injection Standard		

Comments:

Sample Analysis Data and Results

	Sample ID	Aldrin Recovery (ppb=%)	Results (ND=) and Comments
1			
2			
3			
4			
5			
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K. APPENDIX

1. References

CLG-HEC1

CLG-CHC3

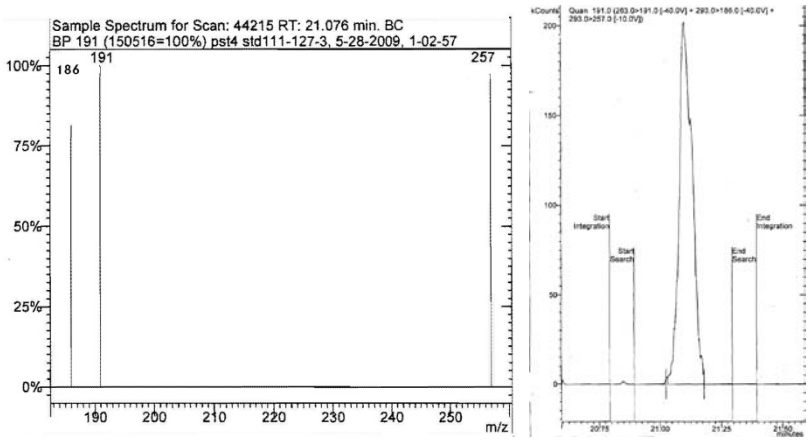
Patel K, Fussell RJ, Hetmanski M, Goodall DM, Keely BJ. Evaluation of gas chromatography-tandem quadrupole mass spectrometry for the determination of organochlorine pesticides in fats and oils. Journal of Chromatography A. Volume 1068, Issue 2, 18 March 2005, 289-296.

2. Table1: Levels and equivalent fortification standard concentrations for the compounds of interest.

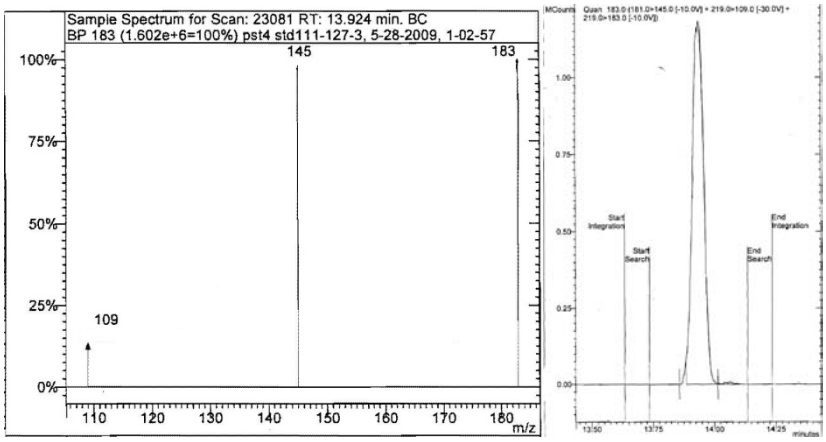
Compound	Level (ppm)	Fort Std Concentration (µg/mL)
Aldrin	0.150	3.0
BHC, alpha-	0.100	2.0
BHC, beta-	0.100	2.0
BHC, delta-	0.100	2.0
BHC, gamma- (Lindane)	0.050	1.0
Chlordane, cis-	0.060	1.2
Chlordane, trans-	0.060	1.2
DDD, o,p'-	1.000	20
DDD, p,p'-	1.000	20
DDE, o,p'-	1.000	20
DDE, p,p'-	2.500	50
DDT, o,p'-	1.000	20
DDT, p,p'-	1.000	20
Dieldrin	0.150	3.0
Endrin	0.050	1.0
HCB (Hexachlorobenzene)	0.050	1.0
Heptachlor	0.067	1.34
Heptachlor Epoxide A	0.067	1.34
Heptachlor Epoxide B	0.067	1.34
Methoxychlor	0.250	5.0
Mirex	0.100	2.0
Nonachlor, trans-	0.060	1.2

3. Ion Chromatograms

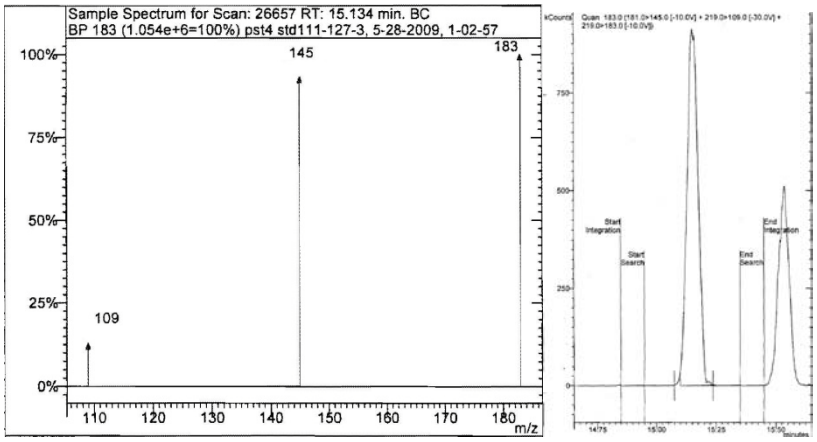
Aldrin



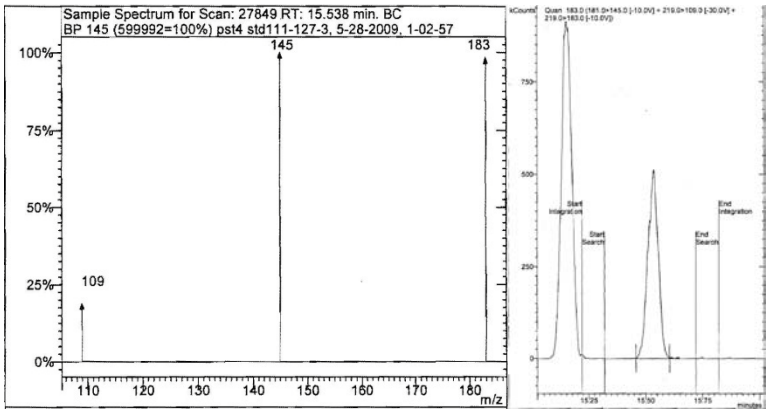
α-BHC



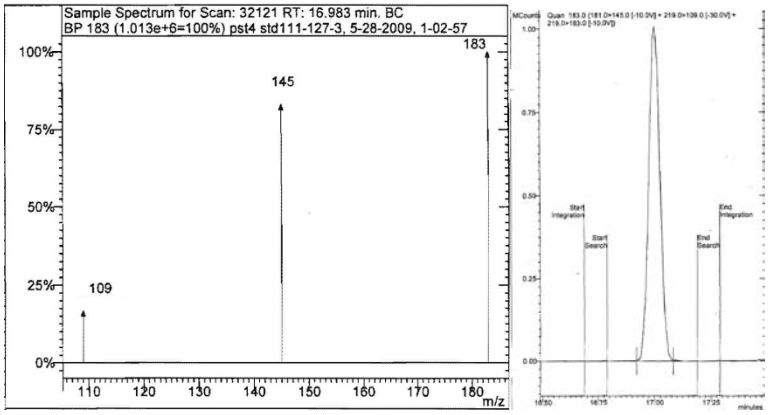
β-BHC



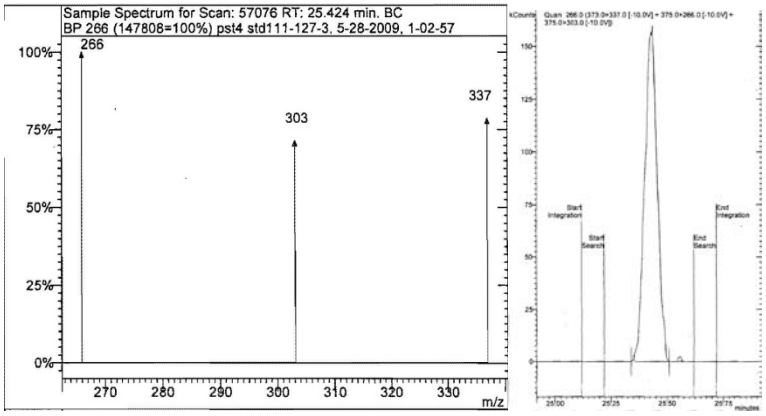
Lindane



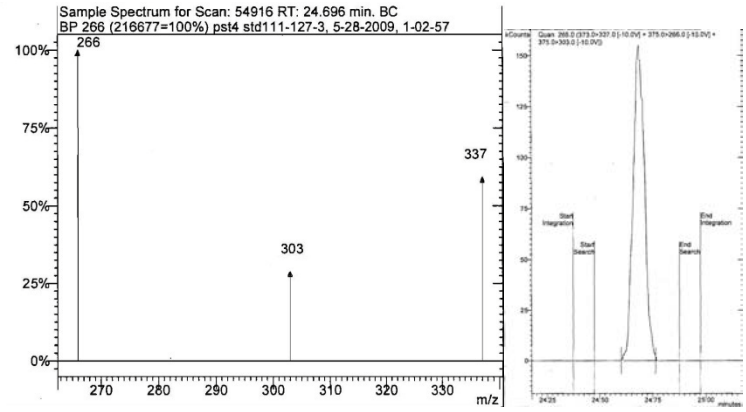
δ-BHC



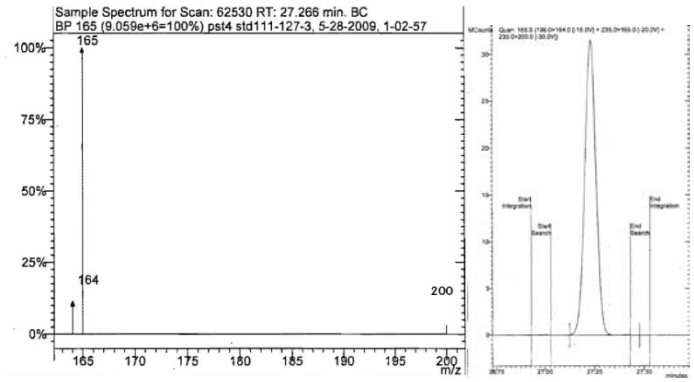
Cis-chlordane



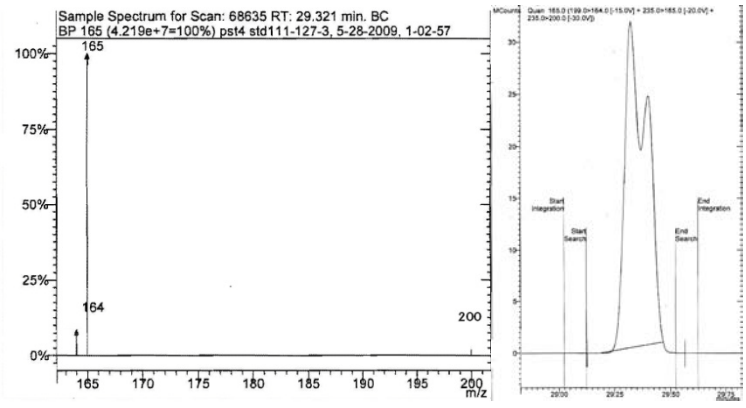
Trans-chlordane



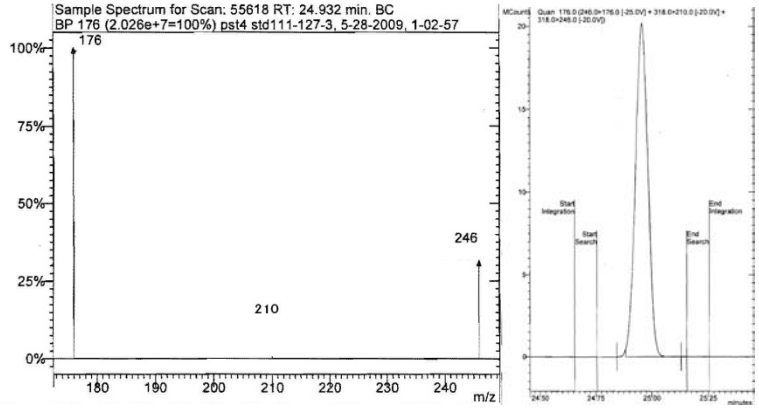
o,p' - DDD



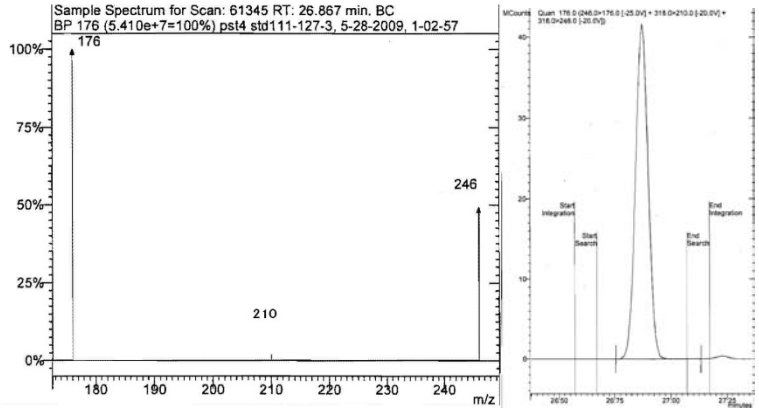
p,p' – DDD



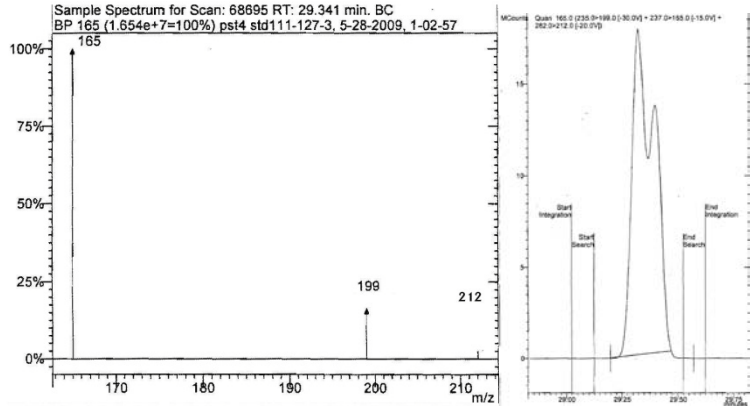
o,p' – DDE



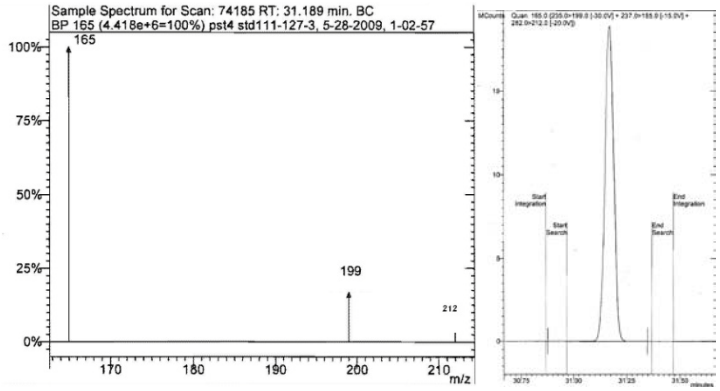
p,p' – DDE



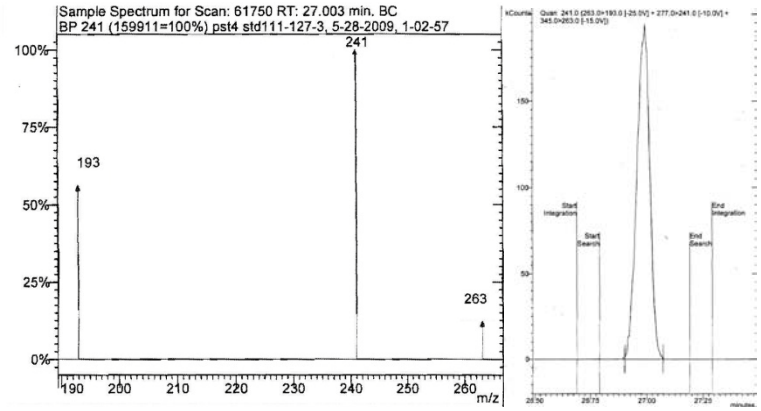
o,p' – DDT



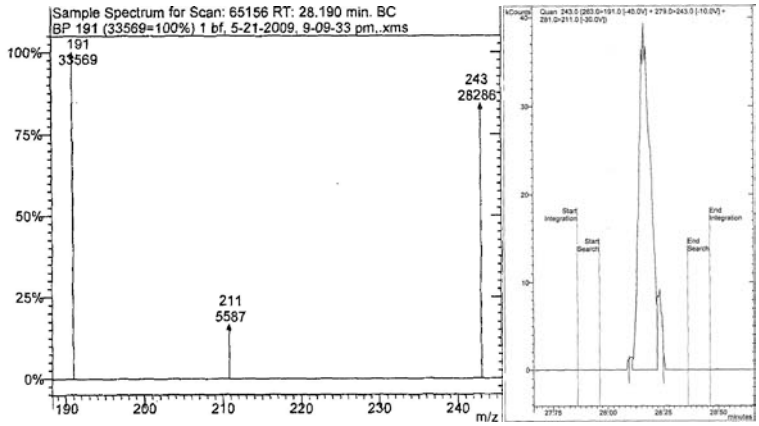
p,p' – DDT



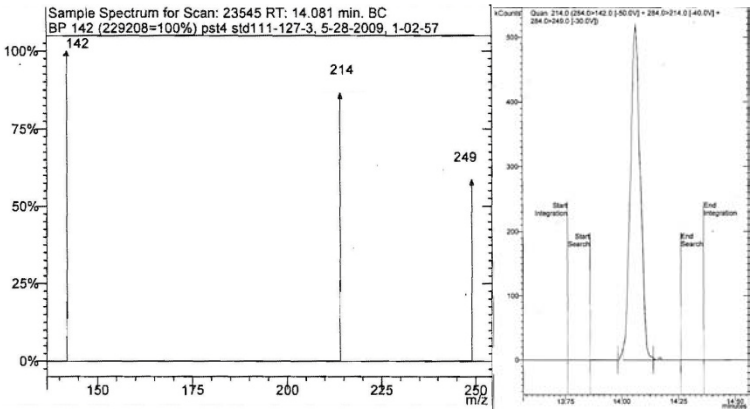
Dieldrin



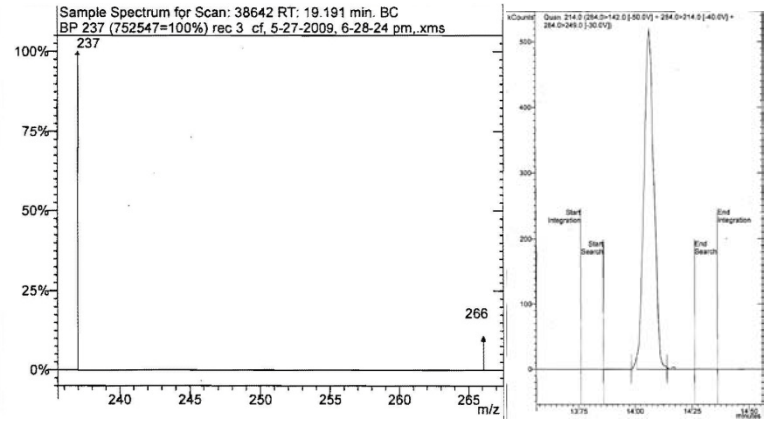
Endrin



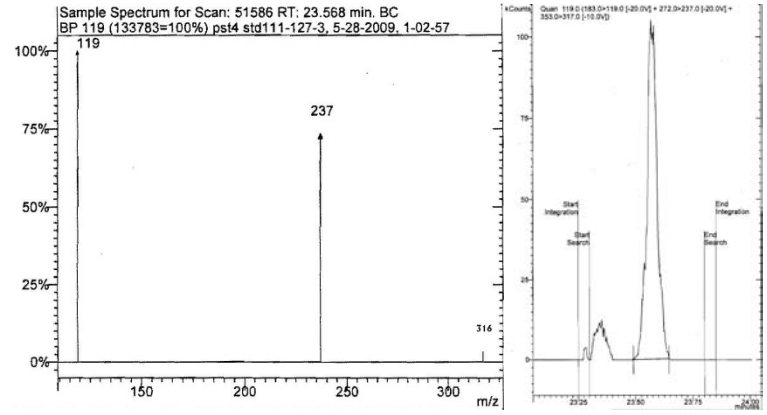
Hexachlorobenzene



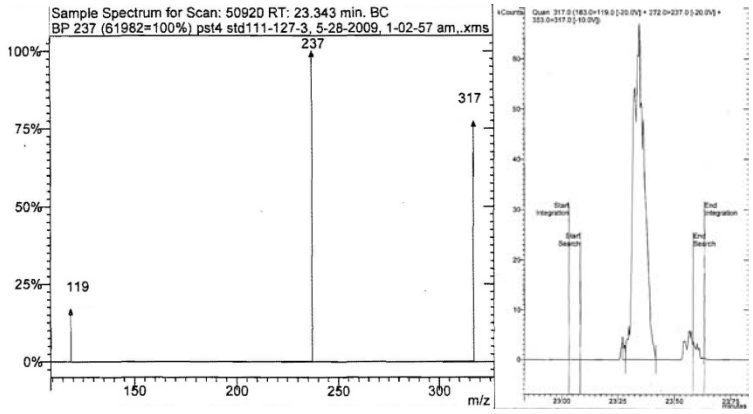
Heptachlor



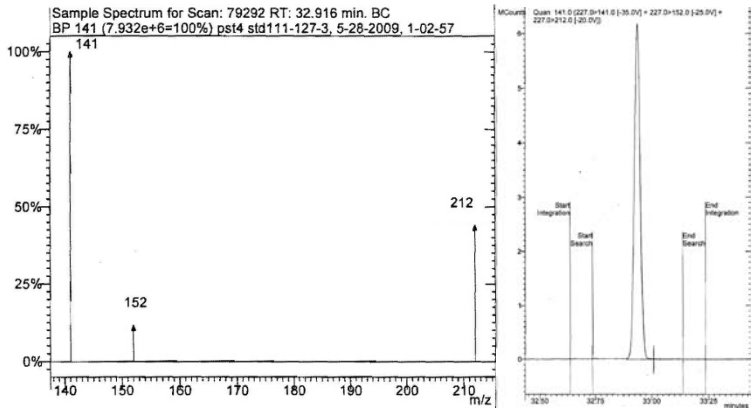
Heptachlor Epoxide A (trans)



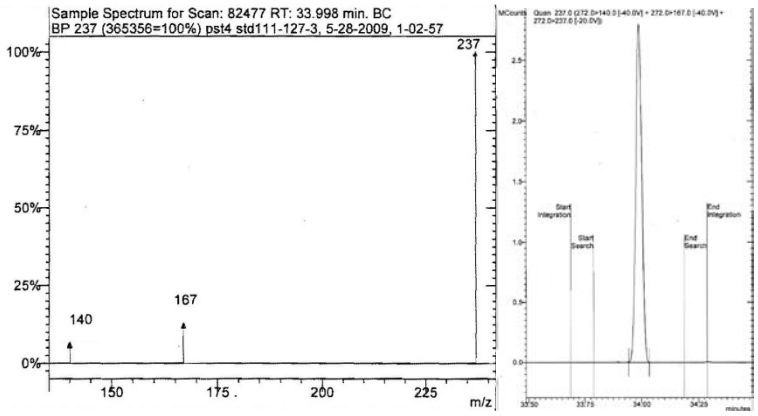
Heptachlor Epoxide B (cis)



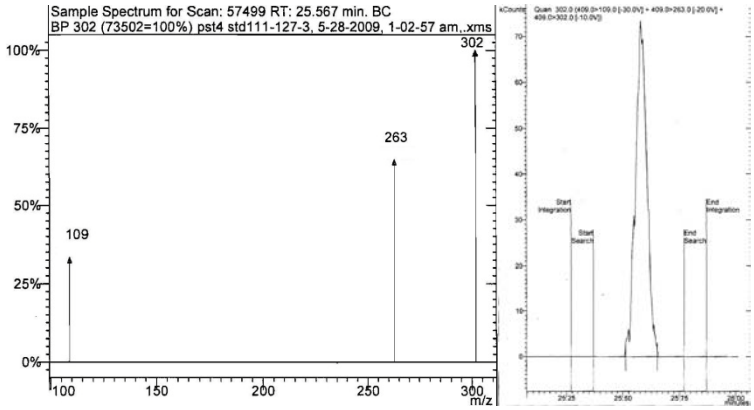
Methoxychlor



Mirex



Trans-nonachlor



4. Possible fragmentation patterns

Aldrin

Ion (m/z)	Fragment
362	[M]+
293	[M + 1 – 2Cl]+
263	[M +2 – C_5H_6 - Cl]+
257	[M + 1 – 3Cl]+
191	[M +2 – C_5H_6 - 3Cl]+
186	[M + 1 – 5Cl]+

BHCs

Ion (m/z)	Fragment
288	$[M]^+$
219	$[M + 1 – 2Cl]^+$
183	$[M + 1 – 3Cl]^+$
181	$[M – 3Cl]^+$
145	$[M – 4Cl]^+$

109	$[M - 5Cl]^+$

Chlordane

Ion (m/z)	Fragment
406	$[M]^+$
375	$[M + 4 - Cl]^+$
373	$[M + 2 - Cl]^+$
337	$[M + 1 - 2Cl]^+$
303	$[M + 2 - 3Cl]^+$
266	$[M + 2 - 4Cl]^+$

DDD

Ion (m/z)	Fragment
318	$[M]^+$
235	$[M - CHCl_2]^+$
200	$[M - CHCl_2 - Cl]^+$
199	$[M - CHCl_2 - Cl]^+$
165	$[M - CHCl_2 - 2Cl]^+$
164	$[M - CHCl_2 - 2Cl]^+$

DDE

Ion (m/z)	Fragment
316	$[M]^+$
318	$[M + 2]^+$
246	$[M - 2Cl]^+$

210	$[M - 3Cl]^+$
176	$[M - 4Cl]^+$

DDT

Ion (m/z)	Fragment
352	$[M]^+$
282	$[M - 2Cl]^+$
237	$[M +2 - CCl_3]^+$
235	$[M - CCl_3]^+$
212	$[M - 2Cl - 2Cl]^+$
199	$[M - CCl_3 - Cl]^+$
165	$[M + 2 - CCl_3 - 2Cl]^+$

Dieldrin

Ion (m/z)	Fragment
378	$[M]^+$
345	$[M + 2 - Cl]^+$
277	$[M - 2Cl - CHO]^+$
263	$[M + 2 - C_5H_6ClO]^+$
241	$[M - 3Cl - CHO]^+$
193	$[M + 2 - C_5H_6ClO - 2Cl]^+$

Endrin

Ion (m/z)	Fragment
378	$[M]^+$
281	$[M + 2 – 2Cl – CHO]^+$
211	$[M + 2 – 4Cl – CHO]^+$
263	$[M – Cl – CHO – C_4H_2]^+$
191	$[M – 3Cl – CHO – C_4H_2]^+$
279	$[M – 2Cl – CHO]^+$
243	$[M + 2 – 3Cl – CHO]^+$

HCB

Ion (m/z)	Fragment
282	$[M]^+$
284	$[M + 2]^+$
249	$[M + 2 – Cl]^+$
214	$[M + 2 – 2Cl]^+$
142	$[M +2 – 4Cl]^+$

Heptachlor

Ion (m/z)	Fragment
370	$[M]^+$
337	$[M + 2 – Cl]^+$
272	$[M + 2 – C_5H_5Cl]^+$
266	$[M + 2 – HCl – 2Cl]^+$
237	$[M + 2 – C_5H_5Cl – Cl]^+$

Heptachlor Epoxides

Ion (m/z)	Fragment
386	$[M]^+$
353	$[M + 2 – Cl]^+$
317	$[M + 2 – 2Cl]^+$
272	$[M + 2 – C_5H_5ClO]^+$
237	$[M + 2 – C_5H_5ClO – Cl]^+$
183	$[M – C_5Cl_4]^+$
119	$[M + 2 – C_5Cl_6]^+$

Methoxychlor

Ion (m/z)	Fragment
345	$[M]^+$
227	$[M -2Cl – OCH_3 – CH_3]^+$
141	$[M +6 - 2Cl – OCH_3 – CH_3 – C_6H_4 - O]^+$
152	$[M – 2Cl - OCH_3 – CH_3 – C_6H_4]^+$
212	$[M – 2Cl - OCH_3 - CH_3 - O]^+$

Mirex

Ion (m/z)	Fragment
545	[M]+
272	$[M - C_5Cl_6]+$
237	$[M - C_5Cl_6-Cl]+$
140	$[M - C_5Cl_6 – C_2Cl_2 - Cl]+$
167	$[M - C_5Cl_6-3Cl]+$

Trans-Nonachlor (m/z)	Fragment
444	[M]+
409	[M – Cl]+
302	[M – 4 Cl]+
263	[M – $C_3H_3Cl_3$ – Cl]+
109	[M - $C_7H_2Cl_6$ – Cl]+

Nonachlor

Ion (m/z)	Fragment
444	[M]+
409	[M – Cl]+
302	[M – 4 Cl]+
263	[M – $C_3H_3Cl_3$ – Cl]+
109	[M - $C_7H_2Cl_6$ – Cl]+

L. APPROVALS AND AUTHORITIES

1. Approvals on file.

2. Issuing Authority: Director, Laboratory Quality Assurance Division.