Analysis of PCBs and Pesticides in

Air and Precipitation Samples

IADN Project

Gas Chromatography Procedure

Prepared by

Karen Arnold and Amina Salamova

O'Neill School of Public and Environmental Affairs
Indiana University
Bloomington, Indiana

Version 2.0 – January 2024

TABLE OF CONTENTS

Chapters Page

I. Introduction……1

II. Routine GC Maintenance .....……3

III. GC Cleaning: ……..5

IV. Routine GC Operation .....……9

V. Mass Hunter Quantitation: ……13

VI. Pesticide Data Reduction in 50% Fraction .....…….……20

VII. PCB and Pesticide Data Reduction in Hexane Fraction.....29

VIII. Appendix: Method Information from GC 6890…….……37

Charts Page

1. A Typical Pesticide Sequence.....11

2. A Typical Batch Sequence for PCB Data Set in Mass Hunter……...……16

3. A Typical Batch Sequence for Pesticide Data Set in Mass Hunter……16

4. A Typical Chart for Manual Integration Using Compounds-at-a Glance in Mass Hunter…….18

5. A Typical Chart for Manual Integration Using Compound Information in Mass Hunter……..19

6. Pesticide Reference Table, DB5 Column.....20

7. Pesticide Reference Table, 1701 Column.....21

8. Pesticide Calibration Table, DB5 Column.....23

Charts Page

9. Pesticide Calibration Table, 1701 Column.....25

10. Pesticide Report, DB5.....27

11. PCB Calibration Table (Common Calibration Standard).....32

12. PCB Report…….....….....35

Chromatograms Page

1. Pesticide Calibration Standard, DB5.....……..……22

2. Pesticide Calibration Standard, 1701.....……..…24

3. Pesticide Sample Chromatogram, DB5.....……..……26

4. Pesticide Sample Chromatogram, 1701.....……..……...28

5. Mike Mullin's 94 Standard with Pesticides..….....30

6. PCB Common Calibration Standard.....31

7. PCBs in Vapor Sample.....……....34

I. INTRODUCTION

This document describes the gas chromatographic (GC) operation and analyses of PCBs and pesticides in air and precipitation samples collected from six sites on the Great Lakes. This research is conducted at the O'Neill School of Public and Environmental Affairs, Indiana University, Bloomington, as a part of the Integrated Atmospheric Deposition Network (IADN). The Great Lakes National Program Office of the U.S Environmental Protection Office supports the research.

There are two GC instruments used for analysis of PCBs and pesticides. These are:

1. **Hewlett Packard GC 6890** Series with an Electronic Pressure Control and Autosampler. It is referred as **GC 1.**
   Installed in by HP Engineer Thomas Kruzil on June 10, 1999 in SPEA 471
   GC Model No. G1530A
   GC Serial No. US00028275

   Autosampler 7683 Series Injector
   Tower Model No. G2613A
   Tower Serial No. US91907156
   Tray Model No. G2614 A
   Tray Serial No. US91605038

   Micro ECD with Ni^{63}, U33126 Date 03/18

2. **Agilent GC 6890N** with an Electronic Pressure Control and Autosampler. It is referred as **GC 2.** Installed by HP Engineer Mike Hartz on March 14, 2005 in SPEA 471
   GC Model No. G1530N
   GC Serial No. CN10505016

   Autosampler 7683 B Series Injector
   Tower Model No. 2913A
   Tower Serial No. CN50423215
   Tray Model No. G2614 A
   Tray No. CN50331934

   Micro ECD with Ni^{63}, U36843 Date 04/20

A 60m, DB-5 column and a 60m, DB-1701 column with 0.25mm id and 0.1µ film thickness are used for good resolution of PCBs and pesticides in 6890 GC1 and GC2. Hydrogen and Nitrogen, ultrapure grade, are used as carrier gas and detector make-up gas. Data acquisition is done with OpenLab CDS A.02.02, a GC Chemstation upgrade C.01.07. Data quantitation is done with Mass Hunter version B.09.00 build 9.0.647.0.

Hexane fraction of a sample after silica gel cleanup is used for the analysis of PCBs, HCB, *p,p'*-DDE, *p,p'*-DDT, *trans*-nonachlor, aldrin, *o,p'*-DDT, and octachlorostyrene in DB 5 column. The 50% dichloromethane/hexane fraction is used for analyses of the other pesticides on both DB-5 and DB-1701. After GC work, the mass of the analytes are calculated by internal standard (ISTD) quantitation procedure. The ISTDs for PCB analysis are PCB congeners 30 and 204. The ISTDs for the pesticides are PCB congeners 65 and 155.

For every GC run one hexane blank and a calibration standard are run for checking the instrument background and for calibrating the instrument. A common reference standard is also run to check the performance of the instrument. Another calibration standard is run at the end to check the shift of response factor of the instrument during the run. Another hexane blank is run at the end to check the cleanliness of the instrument after the samples are run.

Relative response factors (RRFs) for each analyte are determined from the calibration standard's peak areas using equation,

$$\mathrm{RRF_{std}} = \left(\frac{\mathrm{mass_a}}{\mathrm{area_a}}\right)_{\mathrm{std}} \div \left(\frac{\mathrm{mass_{istd}}}{\mathrm{area_{istd}}}\right)_{\mathrm{std}}$$

Where $mass_a$ is the analyte's known mass in the injected amount of calibration standard, $area_a$ is the analyte's peak area, $mass_{istd}$ is the known mass of the appropriate internal standard, and $area_{istd}$ is that internal standard's peak area.

An analyte's mass in a sample ($mass_a$) is calculated from the RRF_{std} above and the internal standard response in the sample by the following equation:

$$\left(\mathrm{mass_a}\right)_{\mathrm{sample}} = \left(\mathrm{area_a}\right)_{\mathrm{sample}} \times \mathrm{RRF_{std}} \times \left(\frac{\mathrm{mass_{istd}}}{\mathrm{area_{istd}}}\right)_{\mathrm{sample}}$$

where $area_a$ is the analyte's peak area in the sample, $mass_{istd}$ is the mass of internal standard spiked into the sample, and $area_{istd}$ is the internal standard's peak area in the sample.

The routine GC maintenance, daily operation, instrument calibration, and the quantitation are described in the following sections.

II. ROUTINE GC MAINTENANCE

1. Gas Tanks

Check the gas tanks. Tanks should not go dry. While changing the tank, lower the temperature of the GC oven down to 40^0C. Leave it at 40^0C for about 15 minutes after changing the tank to get rid of air or oxygen that was drawn in.

2. Head Pressure

It is electronically controlled in 6890. It should be at 22-24 psi.

3. GC oven baking

Before every GC1 run bake the oven at 280^0C, the injector at 280^0C, and the detector at 380^0C for 1 hour. For GC2 bake the oven temperature to 260^0C, the injector at 280^0C, and the detector at 380^0C for 1 hour.

4. Septum

a) After every 50- 60 samples or so change the septum.
b) Cool the oven down to 40 ^{0}C.
c) Remove autosampler tower.
d) Remove septum nut and take the old septum out. Discard.
e) Using clean Q-tips dipped in hexane, wipe off the septum holder.
f) Put a new clean septum and replace the nut. Nut should be snug but not too tight.

5. Background

For 6890 the output is 190-300 mz. Hexane is analyzed at the start of every GC run to monitor the baseline stability. If the signal goes up or hexane run produces noisy chromatogram GC should be cleaned.

6. Standard

A Common Calibration Standard for PCBs and a Mixed Pesticide Standard should be monitored to check the peak detection and the peak broadening or tailing. If the peak shapes are not satisfactory, column should be clipped. Altogether 97 peaks (including PCBs, pesticides, surrogate, and internal standards) should be detected in PCB standard. There should be 56 resolved PCB peaks, 29 unresolved peaks, 3 surrogate standards peaks, 2 internal standards peaks, and 7 pesticides. Congener 17, 18, and 77 should be separated. If not, install a new column. In mixed pesticide standard there should be 22 pesticide peaks, 2 internal standards peaks, and 3 surrogate standard peaks.

7. Checking Leaks and Gas Flow in 6890

Check leaks once in two weeks with a leak detector. Check around the septum, at the injector end, and at the detector end of the column.

Approximate gas flows are as follows:

Split vent	61.4 mL/min
Total flow	70 mL/min
Initial column flow	2 mL/min
Detector gas flow	40 mL/min

The gas flows are set electronically. Sometimes it is advisable to monitor the gas flow with a flow meter to check if the electronic set up match with the actual flow.
The detailed GC 6890 conditions and method information for DB-5 and DB-1701 columns are printed out and added in the appendix.

III. GC CLEANING

CLIPPING OLD COLUMN OR INSTALLING A NEW COLUMN

1. Taking Apart

a) Turn oven, injector, and detector off.

b) Turn hydrogen and nitrogen off manually or electronically. Wait until everything cools down.

c) Take the autosampler tower off.

d) Undo the small nut covering the septum and the large nut underneath it to expose the injection liner. Take the liner out.

e) Open the oven. Take the column out (by detaching from injector and detector ends).

f) Unscrew the nuts from both injector and detector ends of columns and plug the column ends with a septum. **Open end of the column should not be exposed to air.**

g) Place the column on the workbench.

h) Unscrew the holder nut underneath the injection liner. There is a dual vespel ring inlet gold seal in it. The gold seal needs to be replaced each time they are taken apart. Clean these parts by ultrasonication with dichloromethane and hexane and air dry. Clean inlet as specified in step i) below. **These steps are done when there is a problem with signal or base line.**

i) Agilent's preferred method of injection port cleaning is as follows:

Materials: Q-tips, Hexane, Methanol, Acetone, and Methylene Chloride solvents in this order. Proceed with the following steps after completing steps a-h.

1. Place a beaker under the inlet to catch any solvent that should drip from the inlet.

2. Using multiple new Q-tips at once (picture shown below), dip the Q-tips in the hexane and then push Q-tip through the entire body of the inlet weldment. Rotate the Q-tip so that all surfaces are cleaned.

3. Make sure the Q-tips push tightly against the walls of the weldment, like running a "gun brush" through a gun barrel.

4. Run through at least 4 times then inspect the Q-tip.

5. If Q-tip is heavily soiled, get new Q-tips and repeat until Q-tips show no dirt.

6. Repeat steps 2-5 with methanol.

7. Repeat steps 2-5 with acetone.

8. Repeat steps 2-5 with methylene chloride.

9. Install new liner and inert gold seal.

10. Put inlet back together (as mentioned below in next step Assembling Injection Port and Liner) and run pressure decay test.

11. If no leaks found, let hydrogen run through inlet for 30 minutes before heating.

2. Assembling Injection Port and Liner

a) **If steps h) and i) are performed**, assemble the holder nut. The tapered opening of the seal will face downward (the tapered end will hold the end of the ferrule from the column). Screw the nut in before placing the injection liner.

b) Insert a new liner.

c) In 6890 GC1 put a viton O-ring (Agilent part # 5188-5385) on the liner. In 6890 GC2 put a flip top viton O-ring (Agilent part # 5188-5366). Put the big nut on and tighten it. Put in a clean septum. Cover the septum with septum nut (Agilent part #18740-69835). Tighten with a wrench.

3. Clipping Column

a) Take the nut off the injector end of the column. Carefully scrape out all the ferrules from the column nuts, if the ferrule does not come out replace with a new column nut (Agilent part #5181-8830) that has been cleaned. Clean all the different parts with a Q-tip dipped in dichloromethane (DCM) and ultrasonicate these parts with DCM and hexane for 10 minutes with each solvent. After column nuts are cleaned, insert the nut first and then a new ferrule with conical end pointing towards the open end of the column.

b) Clip the column. Make a clean cut with diamond tip score or ceramic square. Examine the cut with a magnifying glass. It should be a clean cut without any jagged edges. **Always clip the column after putting septum, nut, and the ferrule on.**

c) Place the septum so that **25 mm** is measure from the tip of the column to the top of the septum.

d) Carefully insert the column with nut and ferrule through the holder nut and screw it in. Hand tighten the screw more and make it tight with wrench 1/4 turn after hand tight. **Do not over tighten**.

e) Take the nut off the detector end of the column. Remove old ferrule. Turn hydrogen on and check

the flow of gas through the column by inserting the end in a beaker of hexane. Turn hydrogen off. If this is a new column, allow gas to flow through column for 15 minutes to allow the column to bleed before inserting into the detector. Put the septum, nut and the new ferrule on the column in the same way as in the injector end. Clip the column and check for the nice clean cut.

f) Place the septum so that **71 mm** is measure from the tip of the column to the top of the septum. Tighten the screw.

4 Checking Leaks and Gas Flow

a) Turn H_2 and N_2 on. Check leaks with a leak detector. Check around the septum, at the injector, and at the detector ends of the column inside the oven. Check that the head pressure for H_2 is 22-24 psi.

b) Gas flow in 6890 should be back to electronic initial set up.

	GC1	**GC2**
Split vent	61.4 mL/min	61.4 mL/min
Total flow	70 mL/min	70 mL/min
Initial column flow	2 mL/min	2 mL/min
Detector gas flow	40 mL/min	30 mL/min

5. Assembling

a) Reinstall the autosampler tower.

b) Turn the heated zones on.

c) Turn oven on and set the temperature to 40^0C for an hour. Change oven temperature to 70^0C and leave another hour. Change oven temperature to 100^0C.

d) If it is an old column, bake the column, injector, and detector for an hour.

Baking temperature:

Oven:	280^0C (GC1), 260^0C (GC2)
Front Inlet:	280^0C
Front Detector:	380^0C

e) If it is a new column, bake injector, and detector only. Column should be conditioned by ramping it 1 degree per minute to 280^0C (GC1) or 260^0C (GC2). Hold there for 1 hour.

f) If blank run looks satisfactory, check a standard.

5 Changing micro ECD liner:

a) Turn off oven, injector, and detector temperatures.

b) Turn H_2 and N_2 gases off manually or electronically.

c) Open oven door. Unscrew the column nut from the detector end of column. Plug column end.

d) Unscrew the capillary column adapter and remove from the detector. Plug detector end.

e) Remove cap from the capillary column adapter. Take out micro-ECD liner (Agilent Part #G2397-20540).

f) Check bottom of the adapter to see if the graphite ferrule from the column nut did not block the opening.

g) Using care do not touch the micro-ECD liner with bare fingers. Insert new micro-ECD liner and replace the cap.

h) Remove plug and insert the capillary column adapter back into the detector end and tighten the nut.

i) Remove the plug from the column and clip the detector end of the column as is mentioned on clipping the column.

IV. ROUTINE GC OPERATION

1. GC condition and oven temperature program:

PCBs, hexachlorobenzene, *p,p'*-DDE, aldrin, *o,p'*-DDT, octachlorostyrene, about 50% of *trans*-nonachlor, and *p,p'*-DDT are eluted in the hexane fraction, whereas the other chlorinated pesticides and PAHs are eluted in the 50% dichloromethane in hexane fraction after the silica gel column chromatography. The procedure for nitrogen blow down, spiking with internal standard, and making microvials for the autosampler are described in IADN Project Sample Preparation Procedure, Version 1.9, October 2020.

GC 6890	**GC-1**	**GC-2**
Carrier gas:	Hydrogen	Hydrogen
Make up gas	Nitrogen	Nitrogen
Split vent	61.4 mL/min	61.4 mL/min
Total flow	70 mL/min	70 mL/min
Initial column flow	2 mL/min	2 mL/min
Detector gas flow	40 mL/min	30 mL/min

The detailed GC conditions for the GCs are attached in appendix.

2. Temperature Program for 6890

GC1 6890, DB-5	
Initial temp.	100^0 C
Initial time	1 min.
Rate	1^0 C/min
Final temp.	240^0 C
Rate A	10^0 C/min
Final temp A	280^0 C
Final time	20 min.
Purge time	0.5 min.
Run time	165 min

GC2 6890, DB-1701	
Initial temp.	100^0 C
Initial time	1 min.
Rate	10^0 C/min
Final temp.	160^0 C
Rate A	0.6 C/min
Final temp A	222^0 C
Rate B	10^0 C/min
Final temp B	260^0 C
Final time	20 min.
Purge time	0.5 min
Run time	134.13 min.

Mike Mullin specified the gas types, GC condition, column type, and the oven temperature program. The method name is Mullin.m. The method was modified for GC2 and named 1701(3).m.

3. GC Pre-run

a) Check if there is sufficient H_2 and N_2 for operation. If not, change the tank(s). If necessary, change the septum.

b) Bake oven at 280 ^{0}C (DB-5) or at 260 ^{0}C (DB-1701), injector and detector at 280 ^{0}C, and 380 ^{0}C respectively for about an hour.

c) Cool oven to 100 ^{0}C, injector to 250 ^{0}C, and detector to 350 ^{0}C.

Make the samples ready in microvials and load the autosampler tray.

4. Logging into the computer

a) Username Karen
b) Password ********
c) Domain BL-SPEA-Lab GC (non-ADS)

5. Preparing Sequence in ChemStation

Open OpenLab. Open South GC or GCSPEA and then Instrument 1 or 2 (GC1 or GC2)

Method and Run	Sequence	Sequence parameter

a) Type in the subdirectory name (Batch ID). Type the information about calibration standard, dates, and spikes in the comment section.

b) Set the prefix/counter, signal 1: Type in analysis date as prefix. Example J2704 (data acquired on January 27, 2004). Counter should be 001.

c) Prepare a sample table with hexane blank, calibration standard, calibration reference standard, and actual samples with proper ID's. At the end of each sample ID indicate whether the sample is a hexane fraction or 50% fraction with H or F1. Repeat a fresh hexane blank and a fresh standard at the end of the sequence.

d) Save the sequence in c:\chem32\1\Data\ (created batch ID) \ batch ID.S file for GC1 (DB-5) and c:\chem32\2\Data\(created batch ID)\batch ID.S file for GC2 (DB-1701).

Chart 1

A Typical Pesticide Sequence for a GC run

```
Sequence Parameters:

  Operator:

  Data File Naming:               Prefix/Counter
  Signal 1  Prefix:                s0809
  Counter:                         001
  Signal 2  Prefix:                SIG2
  Counter:                         0001
  Data Directory:                  C:\HPCHEM\2\DATA\

  Data Subdirectory:              A09CF1

  Part of Methods to run:         According to Runtime Checklist

  Barcode Reader:                 not used

  Shutdown Cmd/Macro:             none

  Sequence Comment:
  9/8/09 6890 gc1 db-5.pestcalst (b23) bottle 2 4/13/09. Pesticide Common
reference Standard (pestcrs) (b5) 5/5/09


Sequence Table (Front Injector):

  Method and Injection Info Part:
```

Line	Vial	SampleName	Method	Inj	SampleType	InjVolume	DataFile
1	1	hexane blank	MULLIN	1	Sample	2.0	
2	2	pestcalst 090908	MULLIN	1	Sample	2.0	
3	3	pestcalst 090908	MULLIN	1	Sample	2.0	
4	4	pestcrs 090908	MULLIN	1	Sample	2.0	
5	5	lbc 090824,f1	MULLIN	1	Sample	2.0	
6	6	msc 090824,f1	MULLIN	1	Sample	2.0	
7	7	eh 01c 090408,f1	MULLIN	1	Sample	2.0	
8	8	sh 02c 090408,f1	MULLIN	1	Sample	2.0	
9	9	th 01c 090408,f1	MULLIN	1	Sample	2.0	
10	10	pestcalst 090908	MULLIN	1	Sample	2.0	
11	11	ch02c1 090408,f1	MULLIN	1	Sample	2.0	
12	12	ch02c2 090408,f1	MULLIN	1	Sample	2.0	
13	13	ph 01c 090408,f1	MULLIN	1	Sample	2.0	
14	14	lh 01c 090408,f1	MULLIN	1	Sample	2.0	
15	15	pestcalst 090908	MULLIN	1	Sample	2.0	
16	16	hexane blank	MULLIN	1	Sample	2.0	

```
Sequence Table (Back Injector):
  No entries - empty table!
```

6. GC run

a) Starting a 6890 GC run

HPChemStation controls this instrument. After saving the sequence start the instrument with the following steps in the computer.

OpenLab	Method and run control	Run	Run Sequence

d) Post GC run

The data files (*.d folders) will be saved on C:\chem32\1 or 2 data\. Copy the data on flash drive and transfer to L:/HitesR/personnel/name of person analyzing data. This will be copied and worked on with the final file placed in the L;/HitesR/completed?name of person that did the analysis.

V . Mass Hunter Quantitation
GENERAL INTEGRATION AND REPORTING

1. Translate data.

When using Open Lab for data acquisition, the data will need to be translated for Mass Hunter. If Mass Hunter is used to acquire data, the data should not need translation. To translate data, use a GCMS translator software package in Mass Hunter.

a) Open GC/MS Translator.

b) Under MSD Chemstation click on Translate Data Files.

c) Browse to the folder containing the data files to import.

d) Check the box for In-place translation.

e) Browse to the output folder where translated files will go.

f) Click Start Translation. Click close once completed.

2. Create a new batch.

a) Select **File>New Batch**, and navigate to, or create the directory where you want to store the batch and the results of your analysis (the batch directory).

b) In the **File Name** field, type the name you wish to assign to the batch directory.

c) Press **Open.** The Quantitative Analysis program will:

1) Create a folder named QuantResults in the batch directory.

2) Create a batch file with the name you specified and the **extension.batch.bin**.

3) Save the **filename.batch.bin** file to the QuantResults directory.

4) Put the batch filename in the title bar of the current batch.

d) To add samples to this batch, select **File>Add Samples.** The Quantitative Analysis program will display a list of all the data files in the batch directory.

e) Select the files you want to include in the batch.
Press the **Select All** button to select all the data files in the directory, or select files using the shift and control keys to select groups and skip groups, then press **OK**.

f) You are returned to the Batch Table window. Each sample selected for the batch is included as a single line in a batch.

g) Select **File>Save Batch** to save work up to this point.

h) The batch directory now contains all the data file folders selected, plus the folder created by Quantitative Analysis program named QuantResults, with the **filename.batch.bin** file.

i) **Create New Method** or load a current method for the batch.

j) Analyze the data after method applied to batch by clicking **Analyze**.

3. Creating a new method.

From the main window, main menu, click **Method>New>New Method from Acquired Scan Data**. The **New Method from Acquired Data** dialog box opens.

a) Navigate to and select the folder that contains the scan data saved by your MassHunter Data Acquisition program. Agilent recommends that you select data with a high signal so that a representative chromatogram and spectrum can be easily viewed and click **Open**.

b) Click **OK**. The **Method Edit** view opens, the deconvolution algorithm is applied, and the method is created from your data acquisition file. The method table is displayed.

c) If the **Method Setup Tasks** menu is not open on the left side of the screen, go to the main menu, and select **View>Method Development Tasks**.

d) Go to the **Method Setup Tasks** menu, select **Compound Setup**. Under name in the table select the retention time of each compound and rename to the compound of interest. Verify the compounds and their retention times. Compounds can be added or removed manually.

e) From the **Method Setup Tasks** menu, select **ISTD Setup**. Locate the Internals and input their concentration and assign the ISTD to each target compound.

g) From the **Method Setup Tasks** menu, select **Concentration Setup.** This creates the calibration levels and concentrations for each compound in the new method.

h) From **Method Setup Tasks** menu, select **Calibration Curve Setup**. Change the calibration curve parameters. The Curve Fit (CF) should be linear, the CF origin should be included and the CF weight should have none.

i) From the **Method Setup Tasks** menu, Select **Globals Setup.** These boxes should be checked: **Apply Multiplier to Matrix Spike, Apply Multiplier to Surrogate,** and **Apply Multiplier to Target.** The **Correlation Window** should be **1,** the **Non-Reference Window** should be **0.25** with the **Non Reference Window Type** being in **%**, **the Reference Window** should be **0.25** and the **Reference Window Type** being in **%,** and verify these global parameters.

j) From the **Outlier Setup Tasks** menu, limits can be set for various parameters. The **Signal-to-Noise Ratio** should be **3**. **Limit of Detection (LOD)** will be determined one to two times per year after running an Instrument Detection Limits (IDL) study, these values will be adjusted accordingly.

k) From the **Advanced Tasks** menu, the integration parameters can be set. Mass Hunter sets the default to Agile2, which is a more robust integration, however, we are sticking with the type of integration we use with Open Lab. Under the tab **INT.,** select the box and chose **Universal** under integration and then select **Apply to All**. Next select the **Parameters** box and input under the Universal tab the inital parameters:

Threshold	10
Peak Width	0.04
Area Reject	5

Next by selecting **Peak Filter** you can change the following:

Peak Area Counts
Peak Height
Peak Area (%)
Peak Height (%)
Signal to Noise
Limit to the largest

Only one of these filters can be applied at a time. Open lab had Peak Area % at 5, however, the Signal to Noise ratio will eliminate small peaks that may show as a peak but is background noise. However, if there is a specific peak that is a problem you can apply any on these filters to that specific peak in the method. For example, there is a peak near 135+144 that can be problematic to calculations for 135+144 in smaller concentrations. A signal to noise filter can be applied to that congener and if the peak is below the signal to noise it would not allow for quantitation.

l) From **Advanced Tasks** menu, a smoothing parameter can be set. Check calibration peaks to check if some peaks need smoothing, these are usually the smaller peaks. Go to Smoothing tab and select **Gaussian**. **Smoothing Function Width** can be increased, as this is increased the **Smoothing Gaussian Width** must also increase and maintain difference of 10 between the two. For example, Cong.11 needs smoothing and the Smoothing Function width is increase from 15 to 20, therefore, the smoothing Gaussian Width must increase and will increase from 5 to 10 to keep the difference of 10. This may not be necessary to include in the parameters but can be used to help with peaks that are small and may not appear smooth in the chromatograph.

m) From the **Method Tasks** menu find **Validate** and validate the method. Correct any errors. Errors will show up by double clicking on the listed error and will go straight to the error in the method. Once all errors are fixed, validate again until no errors found. Once completed **Save Method** and **Exit**.

Chart 2

A Typical Batch Sequence for PCB Data Set in Mass Hunter

Sample										Compound name		
!	Flag	Name	Data File	Type	Vial	Level	Sample Group	AcqDate_Time	Acq Operator	RT	Final Conc	Area
		hexane blank	d3119001.d	Blank	1		1	mm/dd/yyyy				
		ccs(b10) 191231	d3119002.d	Cal	2	1	1	mm/dd/yyyy				
		pcb crs 191231	d3119003.d	QC	3		1	mm/dd/yyyy				
		msc 191224,h	d3119004.d	Sample	4		1	mm/dd/yyyy				
		eh 01c 191101,h	d3119005.d	Sample	5		1	mm/dd/yyyy				
		sh 01c 191101,h	d3119006.d	Sample	6		1	mm/dd/yyyy				
		th 01c 191101,h	d3119007.d	Sample	7		1	mm/dd/yyyy				
		ch 02c 191101,h	d3119008.d	Sample	8		1	mm/dd/yyyy				
		lh 01c 191101,h	d3119009.d	Sample	9		1	mm/dd/yyyy				
		pp 01c 191101,h	d3119010.d	Sample	10		1	mm/dd/yyyy				
		ccs(b10) 1912131	d3119011.d	Cal	11		1	mm/dd/yyyy				
		hexane blank	d3119012.d	Blank	12		1	mm/dd/yyyy				

Chart 3

A Typical Batch Sequence for a Pesticide Data Set in Mass Hunter

Sample										Compound name		
!	Flag	Name	Data File	Type	Vial	Level	Sample Group	AcqDate_Time	Acq Operator	RT	Final Conc	Area
		hexane blank	d3119001.d	Blank	1		1	mm/dd/yyyy				
		pestcalst 191231	d3119002.d	Sample	2		1	mm/dd/yyyy				
		pestcalst 191231	d3119003.d	Cal	3	1	1	mm/dd/yyyy				
		pcb crs 191231	d3119004.d	QC	4		1	mm/dd/yyyy				
		msc 191224,h	d3119005.d	Sample	5		1	mm/dd/yyyy				
		eh 01c 191101,h	d3119006.d	Sample	6		1	mm/dd/yyyy				
		sh 01c 191101,h	d3119007.d	Sample	7		1	mm/dd/yyyy				
		pestcalst 191231	d3119008.d	Cal	8	1	2	mm/dd/yyyy				
		th 01c 191101,h	d3119009.d	Sample	9		2	mm/dd/yyyy				
		ch 02c 191101,h	d3119010.d	Sample	10		2	mm/dd/yyyy				
		lh 01c 191101,h	d3119011.d	Sample	11		2	mm/dd/yyyy				
		pp 01c 191101,h	d3119012.d	Sample	12		2	mm/dd/yyyy				
		pestcalst 191231	d3119013.d	Sample	13		2	mm/dd/yyyy				
		hexane blank	d3119014.d	Blank	14		2	mm/dd/yyyy				

4. Loading an existing method

Click on **Method** tab then click on **Open** tab, a drop-down menu will show. Click on **Open from existing file**. Browse to the proper folder that have the methods setup for each instrument and each fraction, i.e., ccs.m, pest.m, or 1701.m. The method will open, changes can be made for concentration, retention times, internal standard, and compound names which can be updated, as necessary. When these adjustments have been made, save method as the method in the set being quantitated. Exit the method.

5. Integration of a chromatogram

Parameters for Mass Hunter default to Agile2 which is a more robust quantitation. In order to stay as close as data quantitation of past software, we have put in place the following parameters:

a) Starting Parameters should be found in **Edit Method/Integration Parameters Setup. Click box next to compound name under INT.,** a drop-down menu will open. **Click** and find **Universal.** Set this to **Apply to all. Click** on the box below named **parameters.** These are the following initial parameters:

Threshold	10
Peak Width	0.04
Area Reject	5

Once these are verified, **click** on **Peak Filter** tab. Verify the peak threshold and maximum number of peaks.

Peak Area Counts	10000
Peak Height	10000
Peak Area (%)	5
Peak Height (%)	5
Signal to Noise	3
Limit to the largest	100

Only one of these filters can be utilized in the method. See comments on creating new method. **Save method** to set the parameters if changes are needed. **Exit** edit method.

b) Manually integrate using 1 of 2 pathways after updating the retention time in the method.

1) **Compounds-at-a-Glance.** This method allows you to see several of the compounds at one time.

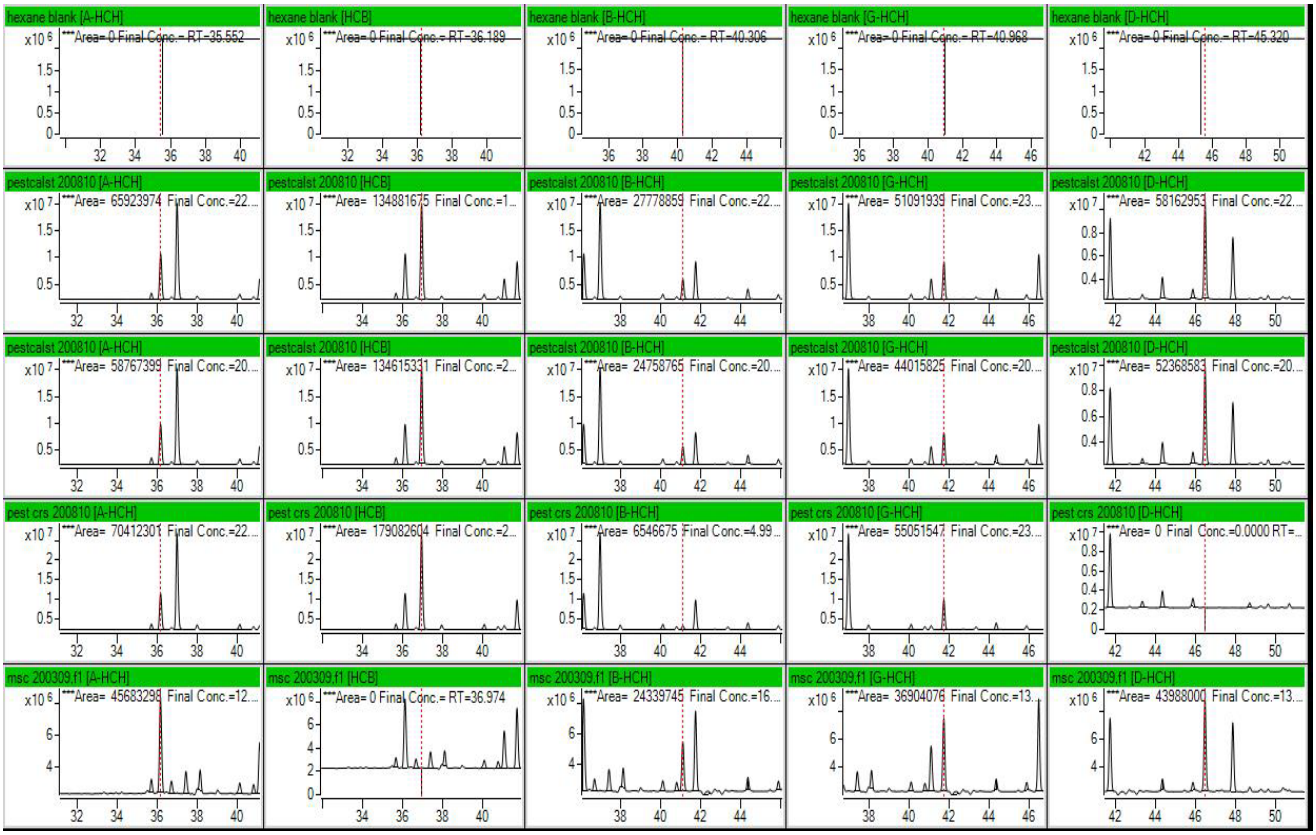
Click View tab then **Click Compounds-at-a-Glance**. This opens to show several samples in a grid format in one location. Click on one of the squares so it is highlighted around the square and right click for menu and click on **properties**. Several items can be custom made to help identify peaks easier. Find **Peak annotations** this will allow showing of retention times, area, final concentration, etc., if so desired for your viewing. Next go to **Reference RT** and click on the box on the right. A menu will pop up. **Check box for Show reference RT.** Adjust the settings to your preference for the color and type of line used for showing the RT for all the compounds. **Click okay, Click apply**. Close properties and setting should now be visible in all the grids.

Each square will show the specified compound within the time set up with the retention time in the method. If the setting has right and left delta at 3 minutes, then the time frame will show 3 minutes prior to the retention time of the compound and 3 minutes after the retention time of the compound.

Manual integration is already operational, a baseline can be set by clicking on the icons for **snapping** and baseline and **dropping** a baseline. Once this has been done the peak will turn a different color to show a manual integration. Do this for each compound.

Chart 4

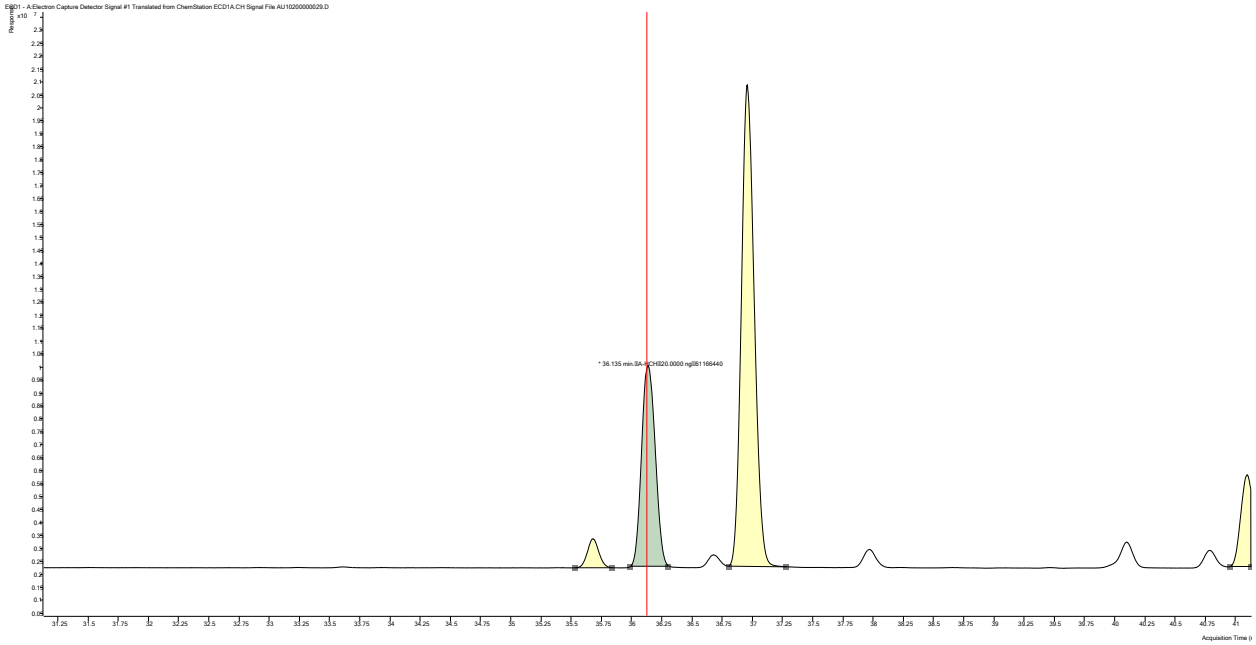
A Typical Chart for Manual Integration Using Compounds-at-a-Glance in Mass Hunter



2) **Compound Information.** This allows for looking at a single compound in a single chromatograph. Using arrow will determine whether you look at the compound to the right of the current compound or looking at the same compound of the next chromatograph. **Click on the icon for manual integration** and the use the**n Click on snapping** and **dropping** baseline. Do this for each compound.

Chart 5

A Typical Chart for Manual Integration Using Compound Information in Mass Hunter



c) Save batch and export table with quantitated results.

1) **Click** on **File** then **Save Batch**.
2) **Click** on **File**, **Export** and then **Export Table.** Place created table into the data set by browsing to the correct set. The program will want to automatically place in the last set where a table was exported. The export table can now be used with a macro to upload data to the reporting data sheets.

VI. PESTICIDE Reference Table, 50% FRACTION

Inject a Mixed Pesticide Standard and load the standard chromatogram in Mass Hunter. Correct baseline, integrate, and identify the pesticide peaks (except HCB, p,p'-DDE, aldrin, o,p'-DDT, and octachlorostyrene) from the following Reference Table. This Reference Table was prepared from individual pesticide injection.

Chart 6

Pesticide Reference Table, DB5 Column

Compounds	GC Retention time Min. (approx.)	concentration ng/ml
α-HCH	36	20
Hexachlorobenzene	37	20
β-HCH	41	20
γ-HCH	42	20
δ-HCH	47	20
e-HCH	48	20
Aldrin	60	20
Congener 65 (ISTD)	61	20
Heptachloroepoxide	67.8	20
Oxychlordane	68.1	20
γ-Chlordane	72	20
Congener 155(RF)	73	20
Endosulfan I	74	20
α-Chlordane	75	20
t-Nonachlor	76	20
Dieldrin	79	20
p,p'-DDE	81	20
o,p'-DDD	81.8	20
Endrin	82	20
Endosulfan II	84	20
p,p'-DDD	88	20
o,p'-DDT	88.2	20
Endosulfan sulfate	92	20
p,p'-DDT	94	20
Methoxychlor	106	20
Dibutylchlorendate	112	20

Chart 7

Pesticide Reference Table, 1701 Column

Compound Name	GC Retention Time Min (approx.)	Concentration (ng/ml)
Hexachlorobenzene	16	20
α-HCH	19	20
γ-HCH	23	20
Aldrin	28	2.5
Congener 65 (ISTD)	29	20
β-HCH	35	20
e-HCH	36	20
Oxychlordane	37	20
Congener 155 (Ref)	37.5	20
δ-HCH	38	20
Heptachlorepoxide	39	20
Endosulfan I	43	20
γ-Chlordane	46	20
α-Chlordane	47	20
T-Nonachlor	48	20
p,p'-DDE	50	20
Dieldrin	51	20
Endrin	54	20
o,p'-DDD	57	20
o,p'-DDT	59	2.5
Endosulfan II	66	20
p,p'-DDD	68	20
p,p'-DDT	71	20
Endosulfan sulfate	85	20
Methoxychlor	89	20
Dibutylchlorendate	92	20

Chromatogram 1

Pesticides Calibration Standard Chromatogram, DB5

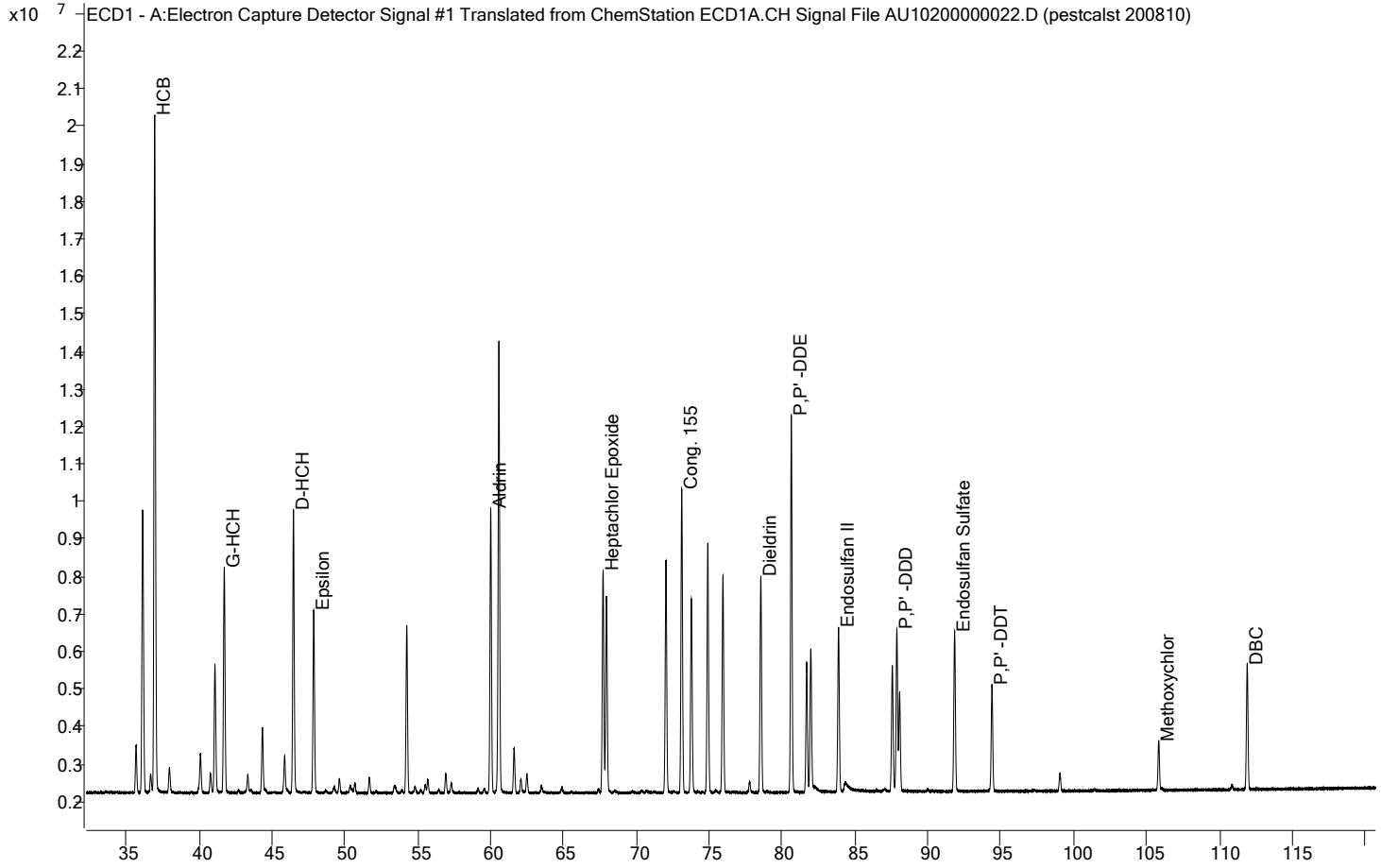


Chart 8
Pesticide Calibration Table, DB5
Method Information

Compound Method	pestcalst 200810			ISTD Method	
Name	RT	Final Conc.	Area	Name	ISTD Conc.
A-HCH	36.133	20	58767399	Cong. 65	20
HCB	36.964	20	134615331	Cong. 65	20
B-HCH	41.094	20	24758765	Cong. 65	20
G-HCH	41.738	20	44015825	Cong. 65	20
D-HCH	46.483	20	52368583	Cong. 65	20
Epsilon	47.872	20	34229273	Cong. 65	20
Aldrin	60.014	20	53914061	Cong. 65	20
Cong. 65	60.576	20	87539709		
Heptachlor Epoxide	67.726	20	43598268	Cong. 65	20
Oxychordane	67.958	20	36975797	Cong. 65	20
G-Chlordane	72.033	20	44933745	Cong. 65	20
Cong. 155	73.118	20	60255120	Cong. 65	20
Endosulfan I	73.781	20	38367732	Cong. 65	20
A-Chlordane	74.911	20	48353040	Cong. 65	20
T-Nona	75.946	20	42362541	Cong. 65	20
Dieldrin	78.551	20	42532986	Cong. 65	20
P,P' -DDE	80.638	20	71610715	Cong. 65	20
O,P' -DDD	81.694	20	24669098	Cong. 65	20
Endrin	81.969	20	26757287	Cong. 65	20
Endosulfan II	83.879	20	33076872	Cong. 65	20
P,P' -DDD	87.881	20	30852154	Cong. 65	20
O,P' DDT	88.067	20	18555786	Cong. 65	20
Endosulfan Sulfate	91.841	20	32535396	Cong. 65	20
P,P' -DDT	94.402	20	20368687	Cong. 65	20
Methoxychlor	105.841	20	9314981	Cong. 65	20
DBC	111.916	20	25229865	Cong. 65	20

Chromatogram 2
Pesticide Calibration Standard, 1701 Column

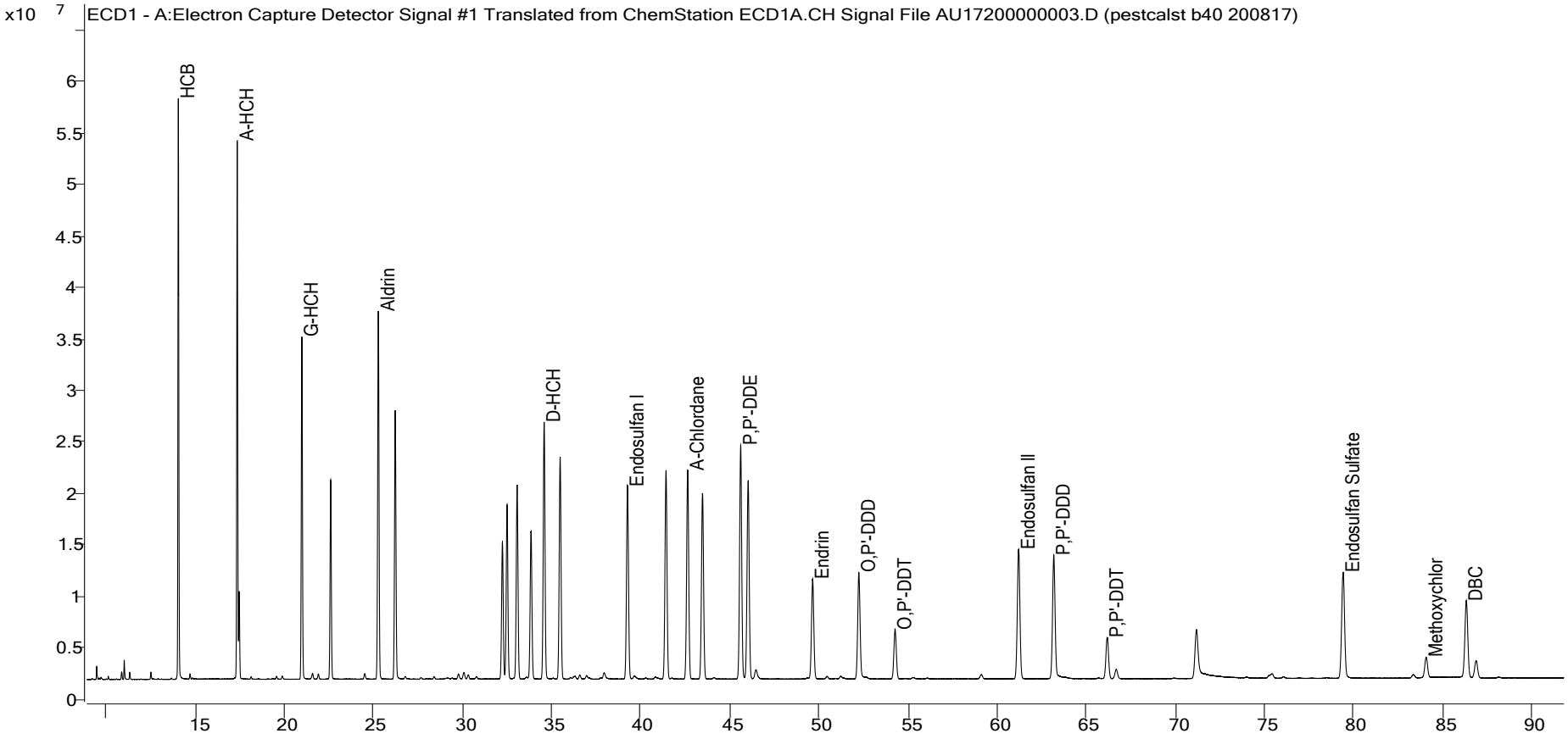


Chart 9
Pesticide Calibration Table, 1701

Compound Method	pestcalst 200810			ISTD Method		ISTD Results	
Name	RT	Final Conc.	Area	Name	ISTD Conc.	RT	Area
HCB	36.964	20	134615331	Cong. 65	20	60.576	87539709
A-HCH	36.133	20	58767399	Cong. 65	20	60.576	87539709
G-HCH	41.738	20	44015825	Cong. 65	20	60.576	87539709
Aldrin	60.014	20	53914061	Cong. 65	20	60.576	87539709
Cong. 65	60.576	20	87539709				
B-HCH	41.094	20	24758765	Cong. 65	20	60.576	87539709
Epsilon	47.872	20	34229273	Cong. 65	20	60.576	87539709
Oxychordane	67.958	20	36975797	Cong. 65	20	60.576	87539709
Cong. 155	73.118	20	60255120	Cong. 65	20	60.576	87539709
D-HCH	46.483	20	52368583	Cong. 65	20	60.576	87539709
Heptachlor Epoxide	67.726	20	43598268	Cong. 65	20	60.576	87539709
Endosulfan I	73.781	20	38367732	Cong. 65	20	60.576	87539709
G-Chlordane	72.033	20	44933745	Cong. 65	20	60.576	87539709
A-Chlordane	74.911	20	48353040	Cong. 65	20	60.576	87539709
T-Nona	75.946	20	42362541	Cong. 65	20	60.576	87539709
P,P' -DDE	80.638	20	71610715	Cong. 65	20	60.576	87539709
Dieldrin	78.551	20	42532986	Cong. 65	20	60.576	87539709
Endrin	81.969	20	26757287	Cong. 65	20	60.576	87539709
O,P' -DDD	81.694	20	24669098	Cong. 65	20	60.576	87539709
O,P' DDT	88.067	20	18555786	Cong. 65	20	60.576	87539709
Endosulfan II	83.879	20	33076872	Cong. 65	20	60.576	87539709
P,P' -DDD	87.881	20	30852154	Cong. 65	20	60.576	87539709
P,P' -DDT	94.402	20	20368687	Cong. 65	20	60.576	87539709
Endosulfan Sulfate	91.841	20	32535396	Cong. 65	20	60.576	87539709
Methoxychlor	105.841	20	9314981	Cong. 65	20	60.576	87539709
DBC	111.916	20	25229865	Cong. 65	20	60.576	87539709

Chromatogram 3
Pesticide Sample Chromatogram
Vapor Phase, DB-5
EH 01C 190802, F1

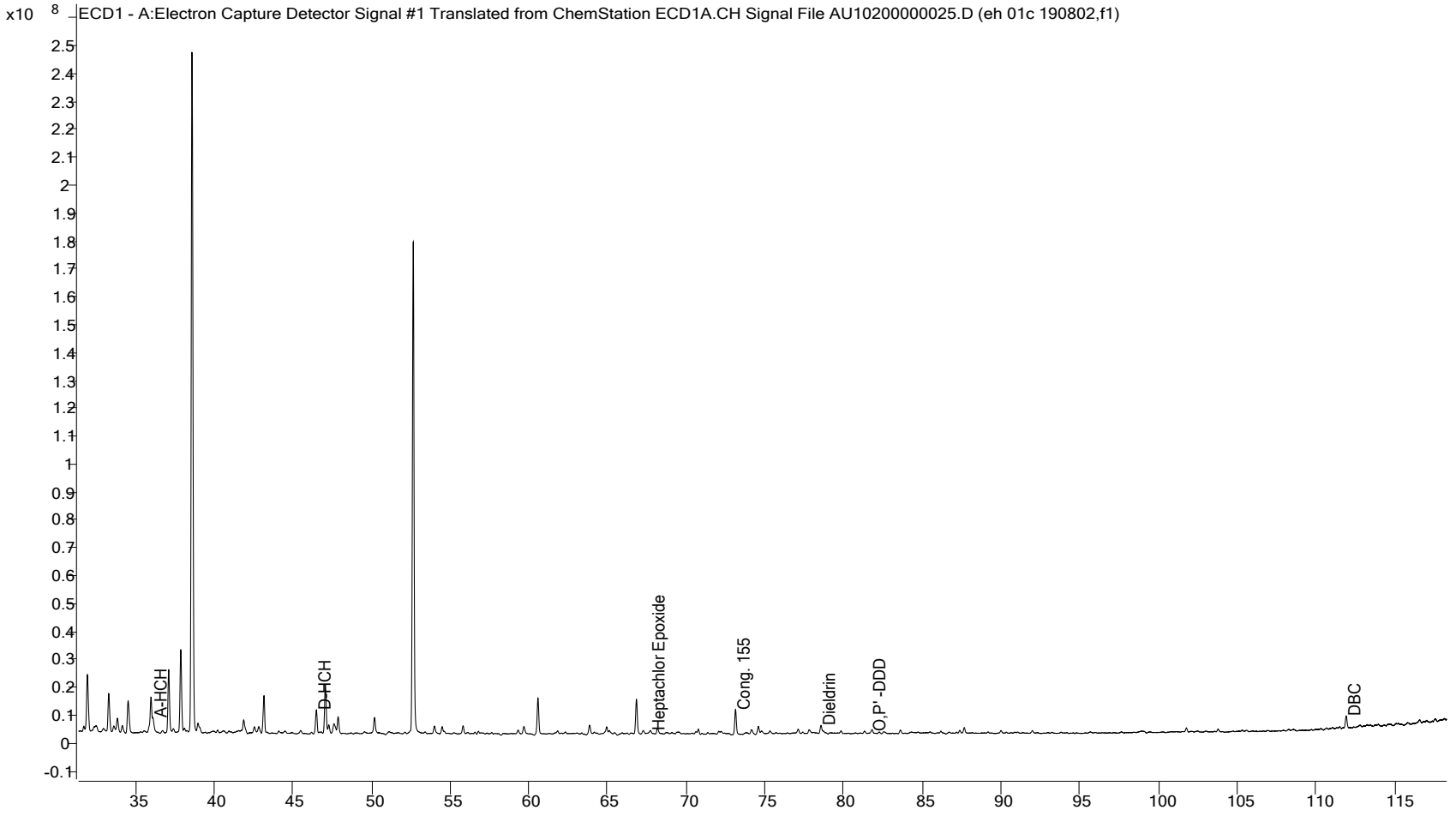


Chart 10

Pesticide Report, DB5

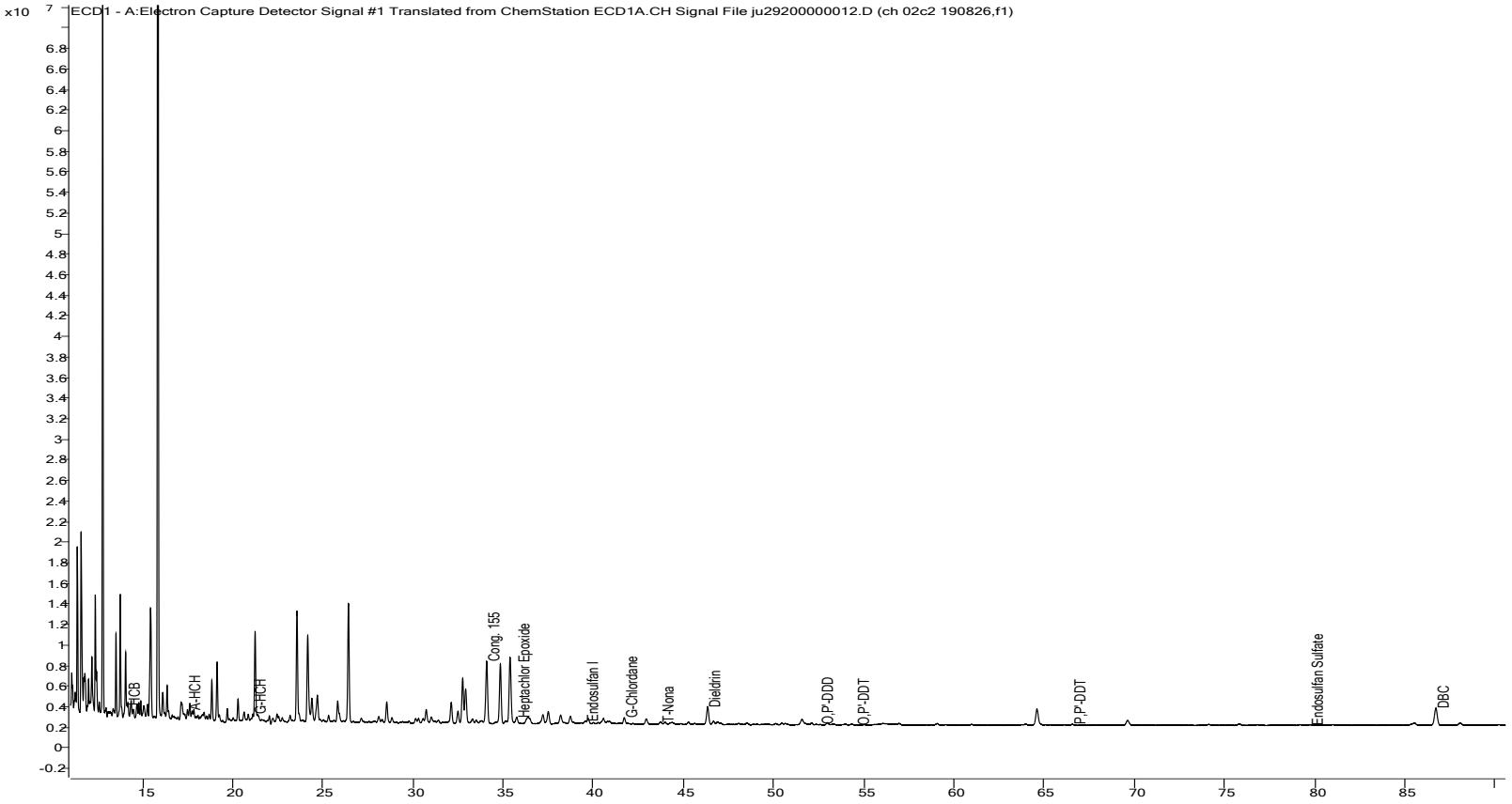
Compound	RT	Area	Final Conc.
A-HCH	36.099	32003216	10.471
HCB	37.014	0	
B-HCH	41.11	0	
G-HCH	41.745	0	
D-HCH	46.514	56077349	20.589
Epsilon	47.895	37224796	20.910
Aldrin	60.004	0	
Heptachlor Epoxide	67.73	7217142	3.183
Oxychlordane	67.955	0	
G-Chlordane	72.072	7509744	3.214
Cong. 155	73.134	64664201	20.634
Endosulfan l	73.792	7130374	3.573
A-Chlordane	74.868	1630201	0.648
T-Nona	75.966	0	
Dieldrin	78.555	22105244	9.993
P,P'-DDE	80.654	0	
P,P'-DDD	81.752	1698480	1.324
Endrin	81.97	0	
Endosulgan ll	83.896	0	
P,P'-DDD	87.9	0	
O,P'-DDT	88.101	0	
Endosulfan Sulfate	91.862	0	
P,P'-DDT	94.418	0	
Methoxychlor	105.91	0	
DBC	111.92	30692045	23.390

A report will be exported from Mass Hunter and a macro will automatically place the final concentration data into the reporting spreadsheets. To export table, make sure cursor is on the table and go to **File>Export>Export Table**. A box will pop up to allow the table to be placed in a specific file.

Chromatogram 4

Pesticide Sample Chromatogram
Vapor Phase, 1701

CH 02C1 190826,F1



VII. PCB AND PESTICIDE DATA REDUCTION IN HEXANE FRACTION

1. Creating a Method File

a) Integration and Peak Identification

Inject Common Calibration Standard which was mixed with (PCB 11, HCB, p,p'-DDE, t-nona, p,p'- DDT and Aldrin, o,p'-DDT, and octachlorostyrene).
Load the standard chromatogram and integrate it following the direction in Chapter V.
Identify PCBs from **S-8074-AR1, S-8074-BR1, S-8074-CR1** (Chromatogram 6) and pesticides from individual pesticide standards in Pesticide Reference Table.

2. Statistical Calculations

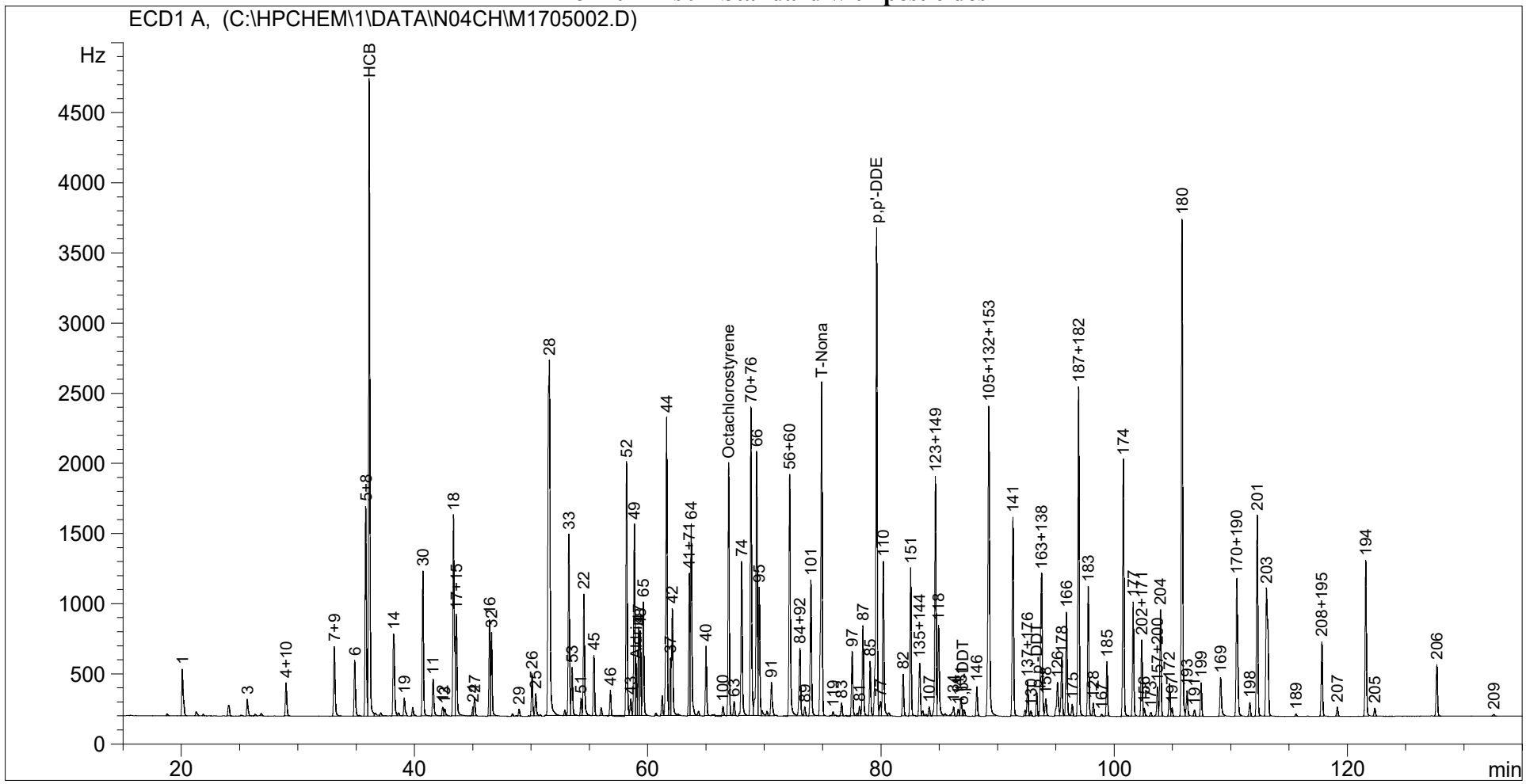
The text files are imported to excel temporarily for statistical calculations. A summary sheet with Total PCBs, percent recoveries of different surrogate standards is generated and printed out.

A Chromatogram from Mike Mullin, PCB Standard Chromatogram, PCB Calibration Table, PCB Sample Chromatogram, PCB Internal Standard Report, and a PCB Integration Events are added in the following pages.

A standard chromatogram of PCB Common Calibration Standard, custom made by AccuStandard, is also added (Chromatogram 7). This standard has replaced the Mike Mullin's 94 standard. This standard is used by all IADN participating laboratories.

Chromatogram 5

Mike Mullin's 94 Standard with pesticides



Chromatogram 6

PCB Common Calibration Standard Chromatogram
(Custom made by AccuStandard, 2009)
Lot# B5020104, B5020105, B5020115

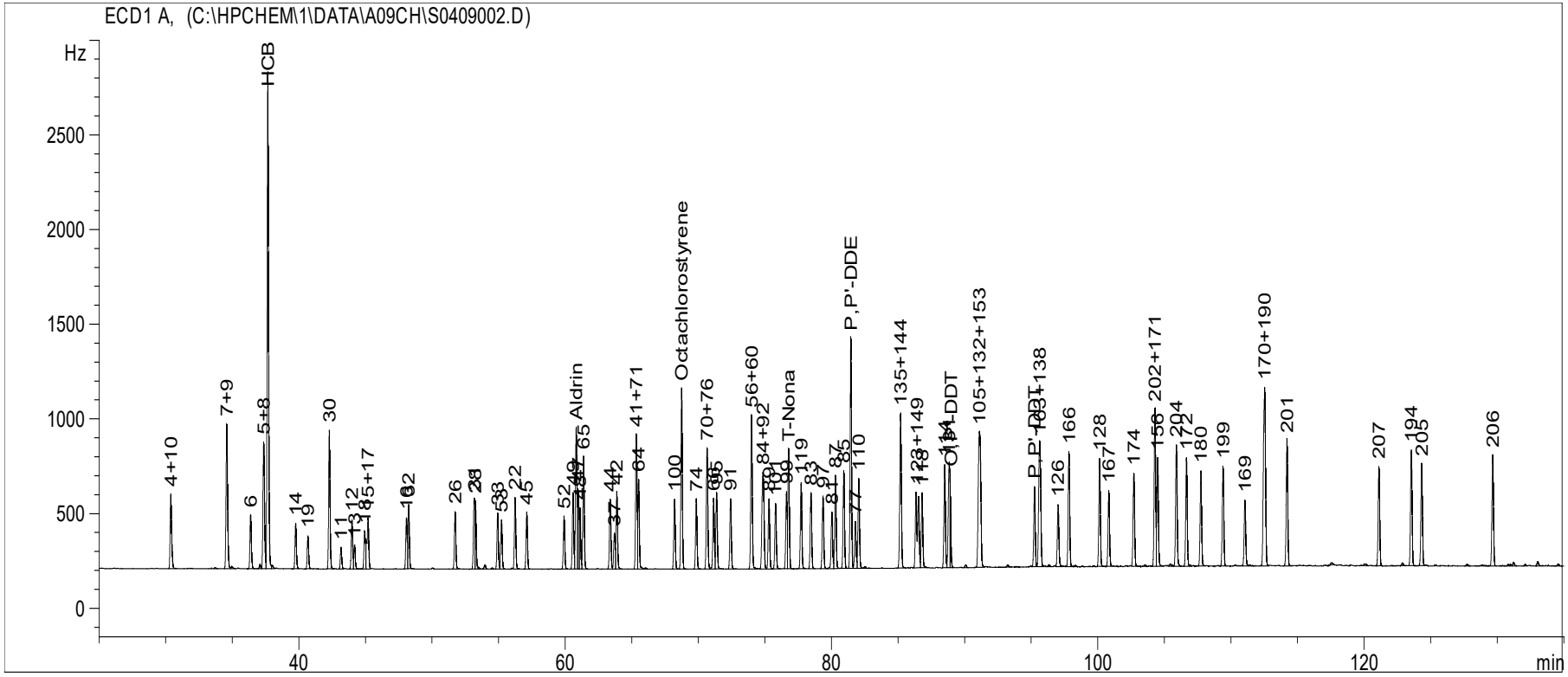


Chart 11
PCB Calibration Table (Common Calibration Standard)

Compound Method	ccs(b10) 200810			ISTD Method		ISTD Results	
Name	RT	Final Conc.	Area	Name	ISTD Conc.	RT	Area
4+10	29.749	20	25422632	30	8	41.577	20942964
7+9	33.92	20	36503648	30	8	41.577	20942964
6	35.7	10	13614230	30	8	41.577	20942964
5+8	36.676	20	38796027	30	8	41.577	20942964
HCB	36.968	20	79478054	30	8	41.577	20942964
14	39.067	10	6968882	30	8	41.577	20942964
19	39.969	5	14561904	30	8	41.577	20942964
30	41.577	8	20942964				
11	42.446	10	2567952	30	8	41.577	20942964
12	43.253	10	10498128	30	8	41.577	20942964
13	43.455	10	3232276	30	8	41.577	20942964
18	44.211	5	15510936	30	8	41.577	20942964
17+15	44.465	15	22389201	30	8	41.577	20942964
16	47.33	5	19547493	30	8	41.577	20942964
32	47.501	5	15263803	30	8	41.577	20942964
26	50.982	5	10408501	30	8	41.577	20942964
31	52.41	5	12106487	30	8	41.577	20942964
28	52.507	5	9817167	30	8	41.577	20942964
33	54.16	5	18772644	30	8	41.577	20942964
53	54.448	5	13784250	30	8	41.577	20942964
22	55.467	5	12851611	30	8	41.577	20942964
45	56.328	5	15617325	30	8	41.577	20942964
52	59.142	5	10811630	30	8	41.577	20942964
49	59.81	5	15085289	30	8	41.577	20942964
Aldrin	60.021	5	9477576	30	8	41.577	20942964
47	60.197	5	12897700	30	8	41.577	20942964
48	60.319	5	16486618	30	8	41.577	20942964
Cong. 65	60.587	10	23217956	30	8	41.577	20942964
44	62.585	5	15382577	30	8	41.577	20942964
37	62.919	5	5139142	30	8	41.577	20942964
42	63.087	5	14554268	30	8	41.577	20942964
41+71	64.543	10	35294852	30	8	41.577	20942964
64	64.715	5	14590603	30	8	41.577	20942964
100	67.416	5	11835174	30	8	41.577	20942964
Octachlorostyrene	67.942	5	14302737	30	8	41.577	20942964
74	69.049	5	9719061	30	8	41.577	20942964
70+76	69.853	10	21055922	30	8	41.577	20942964
66	70.343	5	9919027	30	8	41.577	20942964

Compound Method	ccs(b10) 200810		ISTD Method			ISTD Results	
Name	RT	Final Conc.	Area	Name	ISTD Conc.	RT	Area
95	70.562	5	15327327	30	8	41.577	20942964
91	71.613	5	13343461	30	8	41.577	20942964
56+60	73.173	10	26500718	30	8	41.577	20942964
84+92	74.052	10	26360747	30	8	41.577	20942964
89	74.485	5	15012494	30	8	41.577	20942964
101	74.993	5	11400264	30	8	41.577	20942964
99	75.813	5	12025791	30	8	41.577	20942964
T-Nona	75.956	5	7999780	30	8	41.577	20942964
119	76.907	5	13259268	30	8	41.577	20942964
83	77.634	5	11778198	30	8	41.577	20942964
97	78.543	5	15758744	30	8	41.577	20942964
81	79.198	5	7253981	30	8	41.577	20942964
87	79.475	5	15089741	30	8	41.577	20942964
85	80.099	5	14498172	30	8	41.577	20942964
P,P'DDE	80.648	5	21908962	30	8	41.577	20942964
77	80.956	10	6081020	30	8	41.577	20942964
110	81.219	5	15717235	30	8	41.577	20942964
135+144	84.362	10	30461663	204	6	105.057	16412957
123+149	85.704	10	22513669	204	6	105.057	16412957
118	85.972	5	10960628	204	6	105.057	16412957
114	87.656	5	13436505	204	6	105.057	16412957
131	87.971	5	14066255	204	6	105.057	16412957
O,P',-DDT	88.072	5	1445538	204	6	105.057	16412957
105+132+153	90.298	15	35925733	204	6	105.057	16412957
P,P' DDT	94.42	5	2311967	204	6	105.057	16412957
163+138	94.79	10	29607760	204	6	105.057	16412957
126	96.173	5	8906855	204	6	105.057	16412957
166	96.981	5	14305121	204	6	105.057	16412957
128	99.28	5	15886098	204	6	105.057	16412957
167	99.982	5	10410008	204	6	105.057	16412957
174	101.857	5	15826841	204	6	105.057	16412957
202+171	103.439	10	23890591	204	6	105.057	16412957
156	103.647	5	13775058	204	6	105.057	16412957
204	105.057	6	16412957				
172	105.815	5	15061093	204	6	105.057	16412957
180	106.882	5	13681157	204	6	105.057	16412957
199	108.547	5	15169013	204	6	105.057	16412957
169	110.182	5	8551005	204	6	105.057	16412957
170+190	111.657	10	35220273	204	6	105.057	16412957
201	113.337	5	18071046	204	6	105.057	16412957
207	120.236	5	14386315	204	6	105.057	16412957
194	122.677	5	15798693	204	6	105.057	16412957
205	123.441	5	12824541	204	6	105.057	16412957
206	128.778	5	14470461	204	6	105.057	16412957

Chromatogram 7

PCBs in Vapor Sample
EH 01C 190802, H

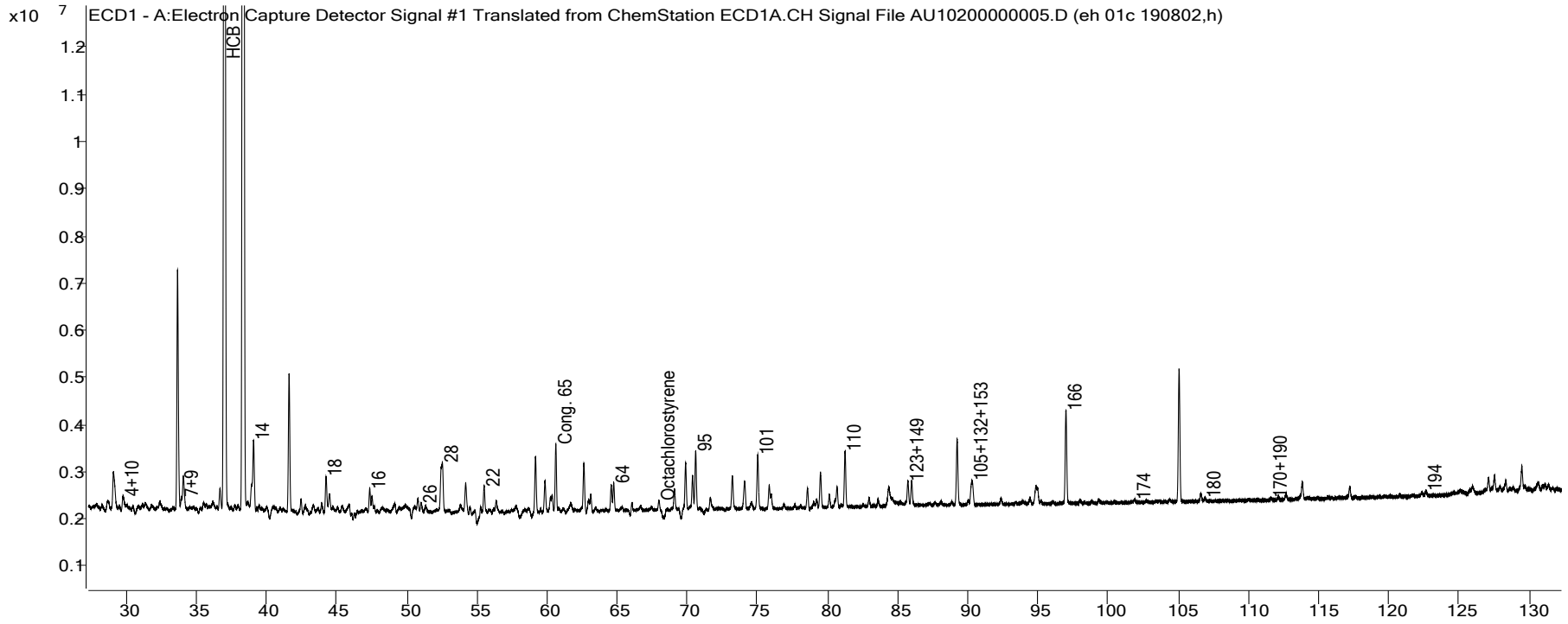


Chart 12

PCB Report

Compound Method	EH 01C 190802,H			ISTD Method		ISTD Results	
Name	RT	Final Conc.	Area	Name	ISTD Conc.	RT	Area
4+10	29.793	2.579	3203931	30	8	41.577	20942964
7+9	34.008	0.585	1042716	30	8	41.577	20942964
6	35.738		0	30	8	41.577	20942964
5+8	36.695	1.646	3121566	30	8	41.577	20942964
HCB	37.009	64.684	251235495	30	8	41.577	20942964
14	39.097	17.013	11588357	30	8	41.577	20942964
19	40.018		0	30	8	41.577	20942964
30	41.577	8.000	20942964				
11	42.479	7.897	1982021	30	8	41.577	20942964
12	43.348		0	30	8	41.577	20942964
13	43.495		0	30	8	41.577	20942964
18	44.257	1.892	5736031	30	8	41.577	20942964
17+15	44.503	2.206	3217552	30	8	41.577	20942964
16	47.368	0.891	3406088	30	8	41.577	20942964
32	47.540	0.751	2240492	30	8	41.577	20942964
26	51.035	0.756	1538701	30	8	41.577	20942964
31	52.507	2.330	5514923	30	8	41.577	20942964
28	52.545	3.550	6811957	30	8	41.577	20942964
33	54.211	1.124	4126056	30	8	41.577	20942964
53	54.521	0.403	1086069	30	8	41.577	20942964
22	55.521	1.460	3668582	30	8	41.577	20942964
45	56.377	0.421	1286586	30	8	41.577	20942964
52	59.186	4.144	8758711	30	8	41.577	20942964
49	59.860	1.655	4879384	30	8	41.577	20942964
Aldrin	60.105		0	30	8	41.577	20942964
47	60.247	0.618	1556970	30	8	41.577	20942964
48	60.372	0.941	3032163	30	8	41.577	20942964
Cong. 65	60.633	4.072	9239902	30	8	41.577	20942964
44	62.634	2.257	6788051	30	8	41.577	20942964
37	62.961	2.097	2106354	30	8	41.577	20942964
42	63.119	0.909	2585356	30	8	41.577	20942964
41+71	64.583	1.090	3758747	30	8	41.577	20942964
64	64.753	1.362	3884668	30	8	41.577	20942964
100	67.467		0	30	8	41.577	20942964
Octachlorostyrene	67.980	0.418	1169401	30	8	41.577	20942964
74	69.088	1.479	2810370	30	8	41.577	20942964
70+76	69.882	3.288	6766157	30	8	41.577	20942964
66	70.377	2.325	4508738	30	8	41.577	20942964

Compound Method	EH 01C 190802,H			ISTD Method		ISTD Results	
95	70.601	2.884	8640454	30	8	41.577	20942964
91	71.646	0.813	2119930	30	8	41.577	20942964
56+60	73.213	2.072	5367953	30	8	41.577	20942964
84+92	74.086	2.271	5850224	30	8	41.577	20942964
89	74.582	0.296	868150	30	8	41.577	20942964
101	75.022	3.750	8357220	30	8	41.577	20942964
99	75.839	1.496	3516911	30	8	41.577	20942964
T-Nona	75.982	1.187	1856500	30	8	41.577	20942964
119	76.905	0.248	643489	30	8	41.577	20942964
83	77.662	0.173	398608	30	8	41.577	20942964
97	78.568	0.930	2865694	30	8	41.577	20942964
81	79.218	0.757	1073240	30	8	41.577	20942964
87	79.493	1.831	5400542	30	8	41.577	20942964
85	80.122	0.587	1664368	30	8	41.577	20942964
P,P'DDE	80.670	0.720	3082912	30	8	41.577	20942964
77	80.994	0.477	283726	30	8	41.577	20942964
110	81.246	2.772	8515596	30	8	41.577	20942964
135+144	84.359	1.137	4545336	204	6	105.057	16412957
123+149	85.725	1.157	3419852	204	6	105.057	16412957
118	85.992	1.251	3598430	204	6	105.057	16412957
114	87.658	0.108	382105	204	6	105.057	16412957
131	87.969		0	204	6	105.057	16412957
O,P',-DDT	88.081		0	204	6	105.057	16412957
105+132+153	90.296	1.898	5967475	204	6	105.057	16412957
P,P' DDT	94.429	1.446	877598	204	6	105.057	16412957
163+138	94.851	1.414	5492791	204	6	105.057	16412957
126	96.181		0	204	6	105.057	16412957
166	96.987	3.971	14910853	204	6	105.057	16412957
128	99.287		0	204	6	105.057	16412957
167	99.946		0	204	6	105.057	16412957
174	101.888	0.103	427339	204	6	105.057	16412957
202+171	103.432	0.057	178994	204	6	105.057	16412957
156	103.625		0	204	6	105.057	16412957
204	105.057	6.000	16412957				
172	105.798		0	204	6	105.057	16412957
180	106.894	0.143	513722	204	6	105.057	16412957
199	108.533		0	204	6	105.057	16412957
169	110.148		0	204	6	105.057	16412957
170+190	111.610	0.037	172712	204	6	105.057	16412957
201	113.351	0.040	187978	204	6	105.057	16412957
207	120.206		0	204	6	105.057	16412957
194	122.649	0.172	712008	204	6	105.057	16412957
205	123.448		0	204	6	105.057	16412957
206	128.789		0	204	6	105.057	16412957

VIII. Appendix: Method Information from GC 6890

```
======================================================================

                                6890 GC1 METHOD
======================================================================


OVEN for GC1
   Initial temp:  100 'C (On)              Maximum temp:  350 'C
   Initial time:  1.00 min                 Equilibration time:  1.00 min
   Ramps:
      #  Rate  Final temp  Final time
      1  1.00      240        0.00
      2 10.00      280        20.00
      3 0.0(Off)
   Post temp:  100 'C
   Post time:  0.00 min
   Run time:   165.00 min


FRONT INLET (UNKNOWN)                   BACK INLET ()
   Mode:  Splitless
   Initial temp:  250 'C (On)
   Pressure:      22.00 psi (On)
   Purge flow:    61.4 mL/min
   Purge time:    0.50 min
   Total flow:    69.1 mL/min
   Gas saver:     On
   Saver flow:    20.0 mL/min
   Saver time:    3.00 min
   Gas type:      Hydrogen

COLUMN 1                                COLUMN 2
   Capillary Column                     (not installed)
   Model Number:        J&W
   Max temperature:     350 'C
   Nominal length:      60.0 m
   Nominal diameter:    250.00 um
   Nominal film thickness:  0.10 um
   Mode:  constant pressure
   Pressure:  22.00 psi
   Nominal initial flow:  1.0 mL/min
   Average velocity:      35 cm/sec
   Inlet:  Front Inlet
   Outlet:  Front Detector
   Outlet pressure:  ambient

FRONT DETECTOR (µECD)                   BACK DETECTOR (NO DET)
   Temperature:  350 'C (On)
   Mode:  Constant makeup flow
   Makeup flow:  40.0 mL/min (On)
   Makeup Gas Type: Nitrogen
   Electrometer:  On
```

```
SIGNAL 1                                SIGNAL 2
   Data rate:  20 Hz                       Data rate:  20 Hz
   Type:  front detector                   Type:  front detector
   Save Data:  On                          Save Data:  Off
   Zero:  0.0 (Off)                        Zero:  0.0 (Off)
   Range:  0                               Range:  0
   Fast Peaks:  Off                        Fast Peaks:  Off
   Attenuation:  0                         Attenuation:  0

COLUMN COMP 1                           COLUMN COMP 2
Derive from front detector              Derive from front detector

                                        POST RUN
                                        Post Time: 0.00 min
TIME TABLE
   Time       Specifier                 Parameter & Setpoint
```

GC Injector

```
     Front Injector:
        Sample Washes                 1
        Sample Pumps                  3
        Injection Volume            2.0 microliters
        Syringe Size               10.0 microliters
        PostInj Solvent A Washes      3
        PostInj Solvent B Washes      3
        Viscosity Delay               0 seconds
        Plunger Speed              Fast
        PreInjection Dwell         0.00 minutes
        PostInjection Dwell        0.00 minutes

     Back Injector:
No parameters specified
```

```
================================================================================
                                6890 GC2 METHOD
================================================================================

OVEN
   Initial temp:  100 'C (On)              Maximum temp:  325 'C
   Initial time:  1.00 min                 Equilibration time:  1.00 min
   Ramps:
      #  Rate  Final temp  Final time
      1 10.00      160        0.00
      2  0.60      222        0.00
      3 10.00      260       20.00
      4   0.0(Off)
   Post temp:  100 'C
   Post time:  0.00 min
   Run time:  134.13 min

FRONT INLET (UNKNOWN)                   BACK INLET ()
   Mode:  Splitless
   Initial temp:  250 'C (On)
   Pressure:  22.00 psi (On)
   Purge flow:  61.4 mL/min
   Purge time:  0.50 min
   Total flow:  70.7 mL/min
   Gas saver:  On
   Saver flow:  20.0 mL/min
   Saver time:  3.00 min
   Gas type:  Hydrogen

COLUMN 1                                COLUMN 2
   Capillary Column                        (not installed)
   Model Number:  Restek rtx-1701
   Max temperature:  260 'C
   Nominal length:  60.0 m
   Nominal diameter:  250.00 um
   Nominal film thickness:  0.10 um
   Mode:  constant pressure
   Pressure:  22.00 psi
   Nominal initial flow:  2.0 mL/min
   Average velocity:  45 cm/sec
   Inlet:  Front Inlet
   Outlet:  Front Detector
   Outlet pressure:  ambient

FRONT DETECTOR (µECD)                   BACK DETECTOR (NO DET)
   Temperature:  350 'C (On)
   Mode:  Constant makeup flow
   Makeup flow:  30.0 mL/min (On)
   Makeup Gas Type: Nitrogen
   Electrometer:  On

SIGNAL 1                                SIGNAL 2
   Data rate:  20 Hz                       Data rate:  20 Hz
```

```
   Type:  front detector                  Type:  front detector
   Save Data:  On                         Save Data:  Off
   Zero:  0.0 (Off)                       Zero:  0.0 (Off)
   Range:  0                              Range:  0
   Fast Peaks:  Off                       Fast Peaks:  Off
   Attenuation:  0                        Attenuation:  0

COLUMN COMP 1                          COLUMN COMP 2
   Derive from front detector             Derive from front detector


                                       POST RUN
                                          Post Time: 0.00 min

TIME TABLE
   Time       Specifier                    Parameter & Setpoint


                                GC Injector


     Front Injector:
        Sample Washes                 1
        Sample Pumps                  3
        Injection Volume            1.0 microliters
        Syringe Size               10.0 microliters
        PostInj Solvent A Washes      3
        PostInj Solvent B Washes      3
        Viscosity Delay               0 seconds
        Plunger Speed              Fast
        PreInjection Dwell         0.00 minutes
        PostInjection Dwell        0.00 minutes

     Back Injector:
No parameters specified
```