Analysis of PCBs and Pesticides

in

Air and Precipitation Samples

IADN Project
Gas Chromatography Procedure

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I. INTRODUCTION

This document describes the instrumental analysis and quantitation of PCBs and pesticides in air and precipitation samples collected from five sites on the Great Lakes. This research is conducted at the 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. The following summarizes the gas chromatographic technique used for quantitation.

Gas Chromatographs used for analysis of PCBs and pesticides are:

1. Hewlett Packard 5890 with 7673A autosampler and Ni^{63} electron capture detector; it is referred as GC North or Instrument #1
2. Hewlett Packard 5890 with 7673A autosampler and Ni^{63} electron capture detector; it is referred as GC South or Instrument #2
   The Integrator Hewlett Packard 3396 controls operations of these two GCs. GCs and autosamplers are connected with Multichannel Interface Hewlett Packard 35900E.

3. Hewlett Packard 6890 with electronic pressure control, 7683 Autosampler and a micro ECD; This GC is mainly used for confirmation of certain compounds.

A 60m, DB-5 column with 0.25mm i.d and 0.1µ film thickness is used for good resolution. Data acquisition and quantitation are done in Hewlett Packard 3365 ChemStation Revision A.06.03 (509). Hydrogen and Nitrogen, ultrapure grade, are used as carrier gas and detector make-up gas. A 60m, DB-1701column with 0.25mm i.d and 0.25µ film thickness is used as a second confirmation column.

Hexane fraction of a sample after silica gel cleanup is used for the analysis of PCBs, HCB, p,p'-DDE, p,p'-DDT, t-Nonachlor, aldrin, o,p'-DDT, and Octachlorostyrene. The 50% dichloromethane fraction in hexane is used for analyses of the other pesticides. After GC work the mass of the analytes are calculated by internal standard (ISTD) quantitaion 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 second 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,

$$\mathrm{RRF}_{\mathrm{std}} = \left(\frac{\mathrm{mass}_{\mathrm{a}}}{\mathrm{area}_{\mathrm{a}}}\right)_{\mathrm{std}} \div \left(\frac{\mathrm{mass}_{\mathrm{istd}}}{\mathrm{area}_{\mathrm{istd}}}\right)_{\mathrm{std}}$$

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:

$$\left(\text{mass}_{\text{a}}\right)_{\text{sample}} = \left(\text{area}_{\text{a}}\right)_{\text{sample}} \times \text{RRF}_{\text{std}} \times \left(\frac{\text{mass}_{\text{istd}}}{\text{area}_{\text{istd}}}\right)_{\text{sample}}$$

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. Gaz 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

Check the column head pressure. It should be at 22-24 psi. If the pressure falls, tighten the septum nut. If the pressure is still low check for leaks and tighten other connections.

3. GC oven Baking

After every GC run bake the oven at 280^0C for 1 hour. After every other run also bake the injector and the detector at 280^0C and 380^0C.

4. Septum

a) After every 50- 60 samples or so change the septum.
b) Cool the oven down to 40°C.
c) Remove autosampler tower.
d) Remove septum nut and take the old septum out. Discard.
e) Using clean Q-tips soaked 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. Column head pressure should go up to 24 psi if nut is tight enough. Check the tightness of the nut after injecting the first sample. Make sure that the head pressure is still 24 psi

5. Background

Background signals in both GC 5890 are usually around 20. For 6890 the output is 170-200 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

Mullin 94 standard 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 118 peaks (including PCBs, pesticides, surrogate and Internal standards) should be detected in PCB standards and congener 17, 18, and 77 should be separated. If not, install a new column.

7. Checking Leaks and Gas Flow in 5890

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.
Check the gas flow once in two weeks with a flow meter. Approximate gas flows are as follows:

Split vent	120 ml/min.
Purge vent	2 ml/min.
Total flow through detector	22 ml/min.

8. **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	10 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 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 one gold seal and a washer in it. The washer and the seal need to be replaced each time they are taken apart. Clean these parts by ultrasonication with dichloromethane and hexane and air dry. **This step is done when there is a problem with signal or base line.**

i) Put a beaker inside the oven underneath the injection port and pour some hexane through the injection port. Clean the injection port with Q-tips and rinse again with hexane.

2. Assembling Injection Port and Liner

a) **If step h is performed**, assemble the holder nut. Place the gold washer first and then the seal. 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) Put a viton O-ring on the liner. Put the big nut on and tighten it. Put in a clean septum. Cover the septum with septum nut. 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. Clean all different parts with Q-tips soaked in DCM and ultrasonicate these with DCM and Hexane for 10 minutes with each solvent. Onto the column, 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 hole with magnifying glass. It should be a clean hole without any jagged end. **Always clip the column after putting the nut and the ferrule on.**

c) Measure **25mm** from the tip of the column. Mark this point with Liquid Paper.

d) Carefully insert the column with nut and ferrule through the holder nut and screw it in. As soon as it feels tight, pull the column out gently until the white mark is seen. 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. Put the 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. Turn hydrogen on and check the flow of gas through the column by inserting the cut end in a beaker of hexane. Turn hydrogen off.

f) Insert the column all the way up until it stops. Pull down about 1 mm and 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 is 24 psi.

b) Check the gas flow with a flow meter. Approximate gas flows for 5890 are as follows:

Split vent	120 ml/min
Purge vent	2 ml/min.
Total flow through detector	22 ml/min

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

Split vent	61.4 mL/min
Total flow	70 mL/min
Initial column flow	2 mL/min
Detector gas flow	10 mL/min

5. Assembling

a) Replace the autosampler tower.

b) Turn 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.

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

Baking temperature:

Oven:	280^0C
Injector A:	280^0C
Injector B:	280^0C
Detector A:	380^0C
Detector B:	380^0C

e) If it is a new column, bake injector and detector. Column should be conditioned by ramping it 1 or 2 degrees per minute to 280^0C. Hold there for 1 hour.

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

IV. ROUTINE GC OPERATION

1. GC condition and oven temperature program:

PCBs, Hexachlorobenzene, p,p'-DDE, aldrin, o,p'-DDT, octachlorostyrene, about 50% of t-Nonachlor, and p,p'-DDT are eluted in the hexane fraction, whereas the other chlorinated pesticides are eluted in the 50% dichloromethane in hexane fraction after the silica gel column chromatography. The procedure for nitrogen blowdown, spiking with internal standard, and making microvials for the autosampler are described in IADN Project Sample Preparation Procedure, Version 1.2, and May 2000.

GC 5890 :

Carrier gas:	Hydrogen
Make up gas	Nitrogen
Split vent:	120 ml/min
Purge vent	2 ml/min
Total flow through the detector:	22 ml/min
Column:	DB-5, 60m, 0.25mm i.d, 0.1μ film thickness

GC 6890

Carrier gas:	Hydrogen
Make up gas	Nitrogen
Split vent	61.4 mL/min
Total flow	70 mL/min
Initial column flow	2 mL/min
Detector gas flow	10 mL/min

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

2. Temperature Program for 5890 and 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 on	3 min.
Purge off	150 min.
Run time	165 min.

DB-1701	
Initial temp.	100^0 C
Initial time	1 min.
Rate	10^0 C/min
Final temp.	160^0 C
Rate A	1^0 C/min
Final temp A	240^0 C
Rate B	10^0 C/min
Final temp B	260^0 C
Final time	20 min.
Run time	109 min.

Mike Mullin specified the GC condition, column type, and the oven temperature program. The method name is Mullin.m

3. GC Pre-run

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

b) Bake oven at 280^0C for half an hour. Bake injector and detector at 280^0C and 380^0C respectively about every other time the oven is baked.

c) Cool oven to 100^0C, injector to 250^0C, and detector to 350^0C.
Make the samples ready in microvials and load the autosampler tray.

4. Logging into the computer

a) User name pvlab29
b) Password ******
c) Domain IUB

5. Preparing Sequence in ChemStation

Open HPChemStation. Open North GC, South GC or GCSPEA

Method and Run Control	Sequence	Sequence parameter

a) Type in the operator's name and the subdirectory name (Batch ID). Type in 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 J2700 (data acquired on January 27, 2000). Counter should be 001.

c) Prepare a sample table with hexane blank, calibration standard, performance 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 hexane blank and a fresh standard at the end of the sequence.

d) Save the sequence in c:\HPChem\1\Sequence as .S file.

An example of a sequence is given on the next page.

Chart 1

Sequence: C:\HPCHEM\1\DATA\M200CH\M200CH.S

Sequence Parameters:

Operator:	Lidia Strandberg
Data File Naming:	Prefix/Counter
Signal 1 Prefix:	jl1400
Counter:	01
Signal 2 Prefix:	SIG2
Counter:	0001
Data Directory:	C:\HPCHEM\1\DATA\
Data Subdirectory:	M200CH
Part of Methods to run:	According to Runtime Checklist
Barcode Reader:	not used
Shutdown Cmd/Macro:	none

Sequence Comment:
South GC pcbcalst(99) and pcbperfst(98). 7/14/00.

Sequence Table (Front Injector):

Method and Injection Info Part:

Line	Vial	SampleName	Method	Inj	SampleType	InjVolume	DataFile
1	1	hexane blank	MULLIN	1	Sample	1.0	
2	2	pcbcalst 000714	MULLIN	1	Sample	1.0	
3	3	pcbperfst 000714	MULLIN	1	Sample	1.0	
4	4	lbc 000626,h	MULLIN	1	Sample	1.0	
5	5	bh 01c 000314,h	MULLIN	1	Sample	1.0	
6	6	eh 01c 000314,h	MULLIN	1	Sample	1.0	
7	7	eh 02c 000314b,h	MULLIN	1	Sample	1.0	
8	8	sh 01c 000314,h	MULLIN	1	Sample	1.0	
9	9	sh 02c 000314,h	MULLIN	1	Sample	1.0	
10	10	th 02c 000314,h	MULLIN	1	Sample	1.0	
11	11	ch 02c1 000314,h	MULLIN	1	Sample	1.0	
12	12	ch 02c2 000314,h	MULLIN	1	Sample	1.0	
13	13	pcbcalst 000714	MULLIN	1	Sample	1.0	
14	14	hexane blank	MULLIN	1	Sample	1.0	

Sequence Table (Back Injector):

No entries - empty table!

Instrument 1 7/17/00 3:10:26 PM Lidia Strandberg

6. **GC run**

a) Programming the integrator (5890 GCs only)

The integrator is already edited for the new method with the proper initial parameters. It does not need to be edited for each run. In case of power failure or method change, the method needs to be edited on the integrator as shown below.

Edit method

A menu with a list of options will be shown. Only the following 3 options need to be edited.

#1. Cht sp [1.0]:	Change the chart speed to 0.1 cm/in	This will save paper
#6. Report Option:	Suppress local report? y/n	Select y
#7. Print and post run options	Large font? y/n	Select n (North GC only)

b) Start a 5890 GC run

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

HPChemStation	Method and run control	Run Control	Run Sequence

And in the integrator

Shift+ seq	Start

Once the GC makes the injection the sequence will start in ChemStation.

c) Starting a 6890 GC run

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

HPChemStation	Method and run control	Run Control	Run Sequence

d) Post GC run

The data files (*.d folders) will be saved on L:\HitesR\GCData\GCNorth or \GCSouth or \GCSPEA (for the 6890). It can be also stored in C:\Hpchem.

V . HP 3365 CHEMSTATION
GENERAL INTEGRATION AND REPORTING

1. Put all *.d folders in a batch (e.g. N99CH or N99FF1) in individual computer as C:\HpChem\1 or 2\data

2. Load Signal

Load a *.d file. The chromatogram will appear on screen.

3. Integration of a chromatogram

Integrate the chromatogram with the following:

Starting Parameters in Integration Events

Initial slope sensitivity	10	Initial
Initial Peak Width	0.04	Initial
Initial Area Reject	10	Initial
Initial height reject	10	Initial
Shoulder Detection	OFF	Initial
Negative peak on	0.0	

Correct the integration by

Baseline now: This command will maintain a straight baseline.

Area sum on and off: This command will split a peak if two peaks are not well resolved.

4. Creating Method file

PCB or Pesticide methods are created with proper calibration tables and integration events after integrating the standard chromatograms. The procedures are described in Chapter V, and Chapter VI.

5. Preparing Report Template (FRP)

View	Report Style	New Template	Edit	New Section	General

Header, footer, and a general section will appear. Separate header and footer section by dragging.
Put Data file name and Sample name in Header section. This information will repeat on each page of the report.
Put information like method file, injection date and time, operator's name, analyst's name, sample ID etc on the top of the general section.
Put **Chromatogram** in General section and set up all options in Set up Chromatogram.

Create a **Table** underneath the chromatogram. Set up the table for Calibrated Compounds. Put the options like mean retention time, main peak type, main peak area, response factor, amount, ISTD, # ISTD, and compound names for the printed columns.

Save the template as PCB.FRP or Pesticide.FRP

6. Printing report

After integrating the chromatogram and loading correct method and correct FRP create a report through **Specify report.** Save the report as ***.txt file in the data folder (***.d folder) together with the method file in C drive.

7. Data storage

After working on the whole batch and saving data in C drive, copy the complete batch files (*.d, *.txt, *.m) in L:\ IADN\CompletedGCdata folder. Make a 2nd copy on zip disk. Delete the folder from C drive.

VI. PESTICIDE DATA REDUCTION IN 50% FRACTION

1. Creating a Method File

a) Integration and Peak Identification

Inject a Mixed Pesticide Standard and load the standard chromatogram in HPChemStation. 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.

Pesticide Reference Table

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
Aldrin	60	5
Congener 65(Ref)	61	20
Heptachloroepoxide	68	10
Octachlorostyrene	67	9.5
Oxychlordane	69	20
γ-Chlordane	72	20
Congener 155(ISTD)	73	20
Endosulfan I	74	20
α-Chlordane	75	20
t-Nonachlor	76	20
Dieldrin	78	20
p,p'-DDE	82	20
o,p'-DDE	82.7	20
Endrin	83	20
Endosulfan II	84	20
p,p'-DDD	88	20
o,p'-DDT	89	20
Endosulfan sulfate	92	20
p,p'-DDT	93	20
Methoxychlor	100	20
Dibutylchlorendate	112	20

b) Preparation of new Calibration Table

If the peak shapes and the integrations look reasonable prepare a calibration table.

Calibration → New Table

Enter all compound names, amount, and mark congener 155 as reference standard and ISTD.
Set calibration setting to 0.25% for reference and other peaks.
Remove all peaks with zero amounts. **Save file as Method file (Pest. M)**. The calibration table and the integration events will be saved in the method.
Print the calibration table and integration events.

c) Replacing Previous Calibration

Once the calibration table is saved in the method it can be recalibrated and replaced in subsequent GC runs.

Calibration → Recalibrate → Replace

If the GC column has been clipped or running conditions have been changed the analyte peaks shift so much that they are not found in the internal standard report and then a new calibration table will have to be created.

2. Samples, 50% fraction

Load Pest. M
Load signal from .d file of a sample and integrate.
Load Pest.FRP for **Report style**
Check the report on screen first.
Print report and save Text File by clicking options in **Specify Report.**
Save the Method and the Text File in the same data folder.
Such as C:\HPChem\1 or 2\data\batch\m3995.d\m3995.m or M3995.txt
Calibration and the integration events will be saved in the method file.

NOTE: Sometimes it is necessary to increase the window more than 0.25% to find internal standard. If it goes more than 0.5%, rerun the sample in GC.

A Pesticide Standard Chromatogram, Pesticide Calibration Table, Pesticide Sample Chromatogram, Pesticide Internal Standard Report, and a Pesticide Event are added in the following pages.

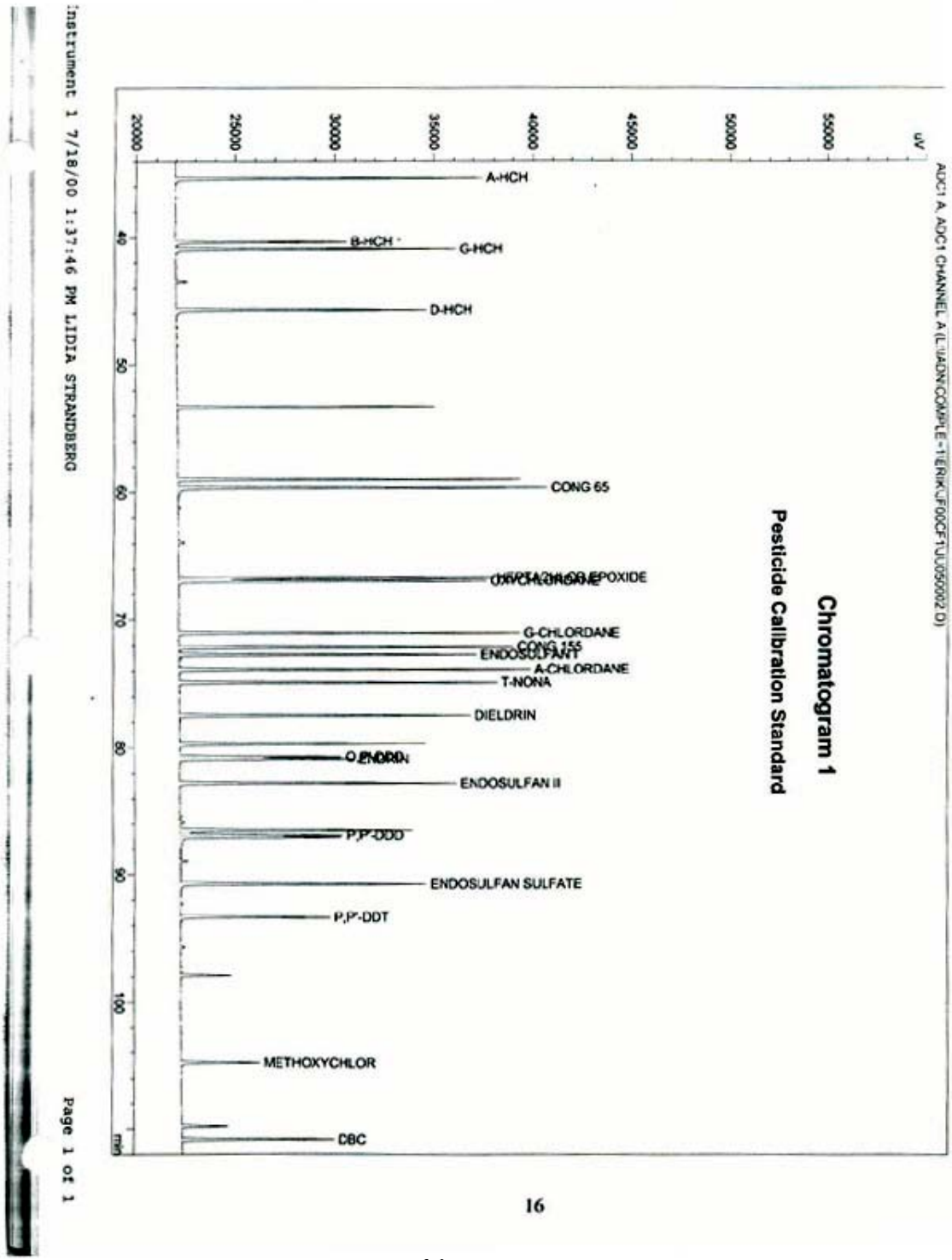
Instrument 1 7/18/00 1:37:46 PM LIDIA STRANDBERG
Page 1 of 1
ADC1 A, ADC1 CHANNEL A (L:\IADN\COMPLE~1\ERIK\LF00CF1\LU050002.D)
uV
20000
25000
30000
35000
40000
45000
50000
55000
40
50
60
70
80
90
100
min
A-HCH
B-HCH
G-HCH
D-HCH
CONG 65
G-CHLORDANE
CONG 155
ENDOSULFAN I
A-CHLORDANE
T-NONA
DIELDRIN
ENDOSULFAN II
P,P'-DDD
ENDOSULFAN SULFATE
P,P'-DDT
METHOXYCHLOR
DBC
Chromatogram 1
Pesticide Calibration Standard

Chart 2

Method C:\HPCHEM\1\DATA\M200CF1\JL130002.D\JL130002.M

Calibration Table

Calib. Data Modified : Monday, July 17, 2000 12:41:15 PM

Calculate : Internal Standard
Based on : Peak Area

Rel. Reference Window : 0.250 %
Abs. Reference Window : 0.000 min
Rel. Non-ref. Window : 0.250 %
Abs. Non-ref. Window : 0.000 min
Uncalibrated Peaks : not reported
Partial Calibration : Yes, identified peaks are recalibrated
Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear
Origin : Included
Weight : Equal

Recalibration Settings:
Average Response : Average all calibrations
Average Retention Time: Floating Average New 75%

Calibration Report Options :
Printout of recalibrations within a sequence:
Calibration Table after Recalibration
Normal Report after Recalibration
If the sequence is done with bracketing:
Results of first cycle (ending previous bracket)

Default Sample ISTD Information (if not set in sample table):

ISTD #	ISTD Amount [ng]	Name
1	20.00000	CONG 155

Signal 1: ADC1 A, ADC1 CHANNEL A

RetTime [min]	Sig	Lvl	Amount [ng]	Area	Amt/Area	Ref	Grp	Name
35.342	1	1	20.00000	1.45176e4	1.37764e-3	1		A-HCH
40.343	1	1	20.00000	9375.86914	2.13314e-3	1		B-HCH
40.893	1	1	20.00000	1.41668e4	1.41175e-3	1		G-HCH
45.696	1	1	20.00000	1.27830e4	1.56457e-3	1		D-HCH
59.638	1	1	20.00000	1.88028e4	1.06367e-3	1		CONG 65
66.689	1	1	20.00000	1.50782e4	1.32642e-3	1		HEPTACHLOR EPOXIDE
66.916	1	1	20.00000	1.56443e4	1.27842e-3	1		OXYCHLORDANE
70.984	1	1	20.00000	1.63924e4	1.22008e-3	1		G-CHLORDANE
72.114	1	1	20.00000	1.67878e4	1.19134e-3	+I1		CONG 155
72.698	1	1	20.00000	1.47724e4	1.35388e-3	1		ENDOSULFAN I
73.842	1	1	20.00000	1.66741e4	1.19946e-3	1		A-CHLORDANE
74.875	1	1	20.00000	1.53551e4	1.30250e-3	1		T-NONA
77.442	1	1	20.00000	1.39928e4	1.42930e-3	1		DIELDRIN
80.668	1	1	20.00000	8656.53027	2.31039e-3	1		O,P'-DDD
80.834	1	1	20.00000	7472.73975	2.67639e-3	1		ENDRIN
82.757	1	1	20.00000	1.39081e4	1.43801e-3	1		ENDOSULFAN II
86.830	1	1	20.00000	8307.47852	2.40747e-3	1		P,P'-DDD
90.692	1	1	20.00000	1.32690e4	1.50728e-3	1		ENDOSULFAN SULFATE
93.308	1	1	20.00000	7388.10938	2.70705e-3	1		P,P'-DDT
104.760	1	1	20.00000	3191.88574	6.26589e-3	1		METHOXYCHLOR
110.785	1	1	20.00000	7584.74463	2.63687e-3	1		DBC

1 Warnings or Errors :

Instrument 1 7/17/00 12:41:19 PM Lidia Strandberg Page 1

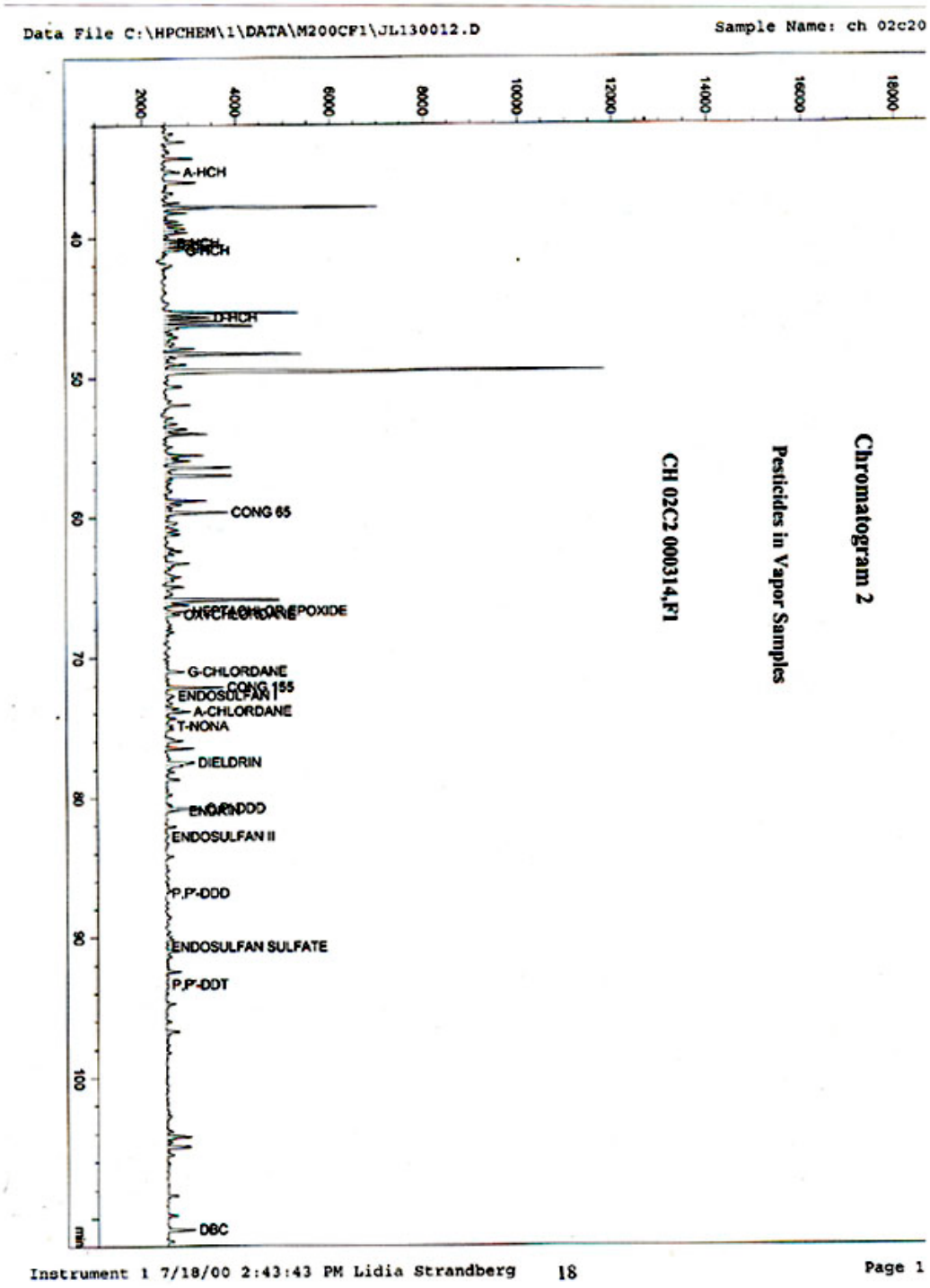
Data File C:\HPCHEM\1\DATA\M200CF1\JL130012.D
Sample Name: ch 02c20
2000
4000
6000
8000
10000
12000
14000
16000
18000
A-HCH
G-HCH
D-HCH
CONG 65
G-CHLORDANE
CONG 155
ENDOSULFAN I
A-CHLORDANE
T-NONA
DIELDRIN
ENDRIN
ENDOSULFAN II
P,P'-DDD
ENDOSULFAN SULFATE
P,P'-DDT
DBC
Chromatogram 2
Pesticides in Vapor Samples
CH 02C2 000314.F1
Instrument 1 7/18/00 2:43:43 PM Lidia Strandberg
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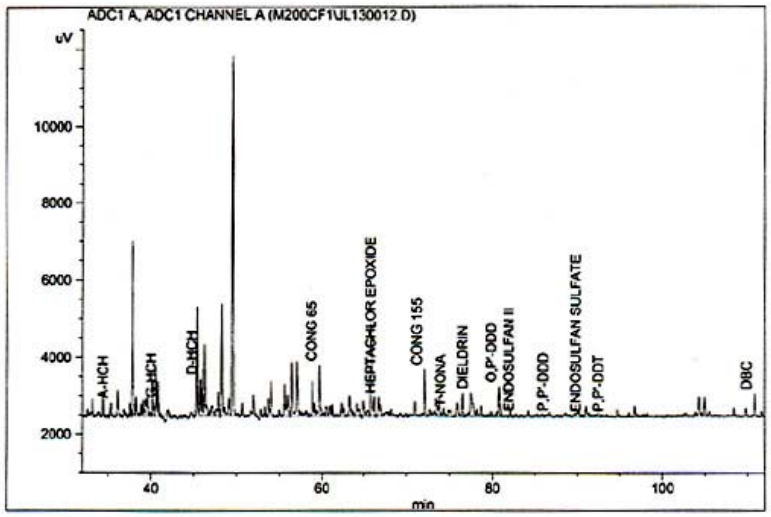
Chart 3

Method File: C:\HPCHEM\1\DATA\M200CF1\JL130012.D\JL130012.M
Data File: C:\HPCHEM\1\DATA\M200CF1\JL130012.D

Injection Date and Time: 5:39:19 PM
Calibration Modification Date and Time: Jul 17, 2000 01:56:53 pm

GC Operator: Lidia Strandberg
Data Analyst: Karen Arnold
Sample: CH 02C2 000314,F1
Comments: TW= 34.70G, C1= 17.31G, C2= 17.39G



Ret. Time (min.)	Peak Type	Peak Area	Rel. Res. Factor	Amount (ng)	ISTD #	Is ISTD	Compound Name
35.319	VP	1990	1.153	4.488	1		A-HCH
40.372	VVA+	845	1.785	2.950	1		B-HCH
40.943	VVA+	1377	1.181	3.180	1		G-HCH
45.702	VV	6828	1.309	17.484	1		D-HCH
59.641	VV	9901	0.891	17.246	1		CONG 65
66.703	PV	4139	1.111	8.992	1		HEPTACHLOR EPOXIDE
66.923	VP	2811	1.072	5.891	1		OXYCHLORDANE
70.998	VV	2987	1.027	5.997	1		G-CHLORDANE
72.127	VP	10228	1.000	20.000	1	X	CONG 155
72.726	PV	1658	1.135	3.679	1		ENDOSULFAN I
73.849	VV	4333	1.004	8.508	1		A-CHLORDANE
74.880	VV	1264	1.090	2.694	1		T-NONA
77.474	VV	7375	1.196	17.252	1		DIELDRIN
80.756	VV	6477	1.934	24.488	1		O,P'-DDD
80.927	VBA+	1653	2.240	7.241	1		ENDRIN
82.803	VBA+	147	1.202	0.345	1		ENDOSULFAN II
86.817	VBA+	121	2.005	0.474	1		P,P'-DDD
90.667	PP N	116	1.257	0.286	1		ENDOSULFAN SULFATE
93.347	VVA+	198	2.266	0.876	1		P,P'-DDT
0.000		0	0.000	0.000	1		METHOXYCHLOR
110.801	PP	5210	2.207	22.483	1		DBC

Chart 4

Method C:\HPCHEM\1\DATA\M200CF1\JL130012.D\JL130012.M

Signal Specific Integration Event Table "Event_ADC1A"

Event	Value	Time
Initial Slope Sensitivity	10.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	10.000	Initial
Initial Height Reject	10.000	Initial
Initial Shoulders	OFF	Initial
Negative Peak ON		0.000
Baseline Now		25.579
Area Sum ON		35.141
Area Sum OFF		35.275
Area Sum ON		40.321
Area Sum OFF		40.419
Area Sum ON		40.873
Area Sum OFF		41.007
Area Sum ON		59.426
Area Sum OFF		59.537
Area Sum ON		59.745
Area Sum OFF		59.880
Area Sum ON		70.725
Area Sum OFF		70.897
Area Sum ON		72.803
Area Sum OFF		73.000
Area Sum ON		73.959
Area Sum OFF		74.119
Area Sum ON		74.723
Area Sum OFF		74.796
Area Sum ON		80.533
Area Sum OFF		80.643
Area Sum ON		80.839
Area Sum OFF		81.011
Area Sum ON		82.728
Area Sum OFF		82.872
Baseline Now		83.953
Area Sum ON		86.752
Area Sum OFF		86.875
Area Sum ON		93.283
Area Sum OFF		93.406
Baseline Now		98.478
Baseline Now		110.134
Baseline Now		115.171

Apply Manual Integration Events: No

Instrument 1 7/17/00 2:05:22 PM Lidia Strandberg

VII. PCB AND PESTICIDE DATA REDUCTION IN HEXANE FRACTION

1. Creating a Method File

a) Integration and Peak Identification

Inject Mullin 94 Standard which was mixed with (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 IV.
Identify PCBs from Mullin's 94 chromatogram (Chromatogram 3) and pesticides from individual pesticide standards in Pesticide Reference Table.

b) Preparation of new Calibration Table

If the peak shapes and integration look good prepare a calibration table.

Calibration → New Table

Enter all compound names, amounts supplied by Mike Mullin, and mark congener 30 and 204 as reference ISTDs.

Set calibration setting to 0.25% for reference and other peaks.
Remove all peaks with zero amounts. Save file as **Method File (PCB. M)**. The calibration table and the integration events will be saved in the method.
Print the calibration table and integration events.

c) Replacing Previous Calibration

Once the calibration table is saved in the method it can be recalibrated and replaced in subsequent GC runs.

Calibration → Recalibrate → Replace

If the GC column has been clipped or running conditions have been changed the analyte peaks shift so much that they are not found in the internal standard report and then a new calibration table will have to be created.

2. Samples, Hexane fraction

Load PCB.M
Load signal from *.d file of a sample and integrate.
Load PCB.FRP for **Report style**

Check the report on screen first.

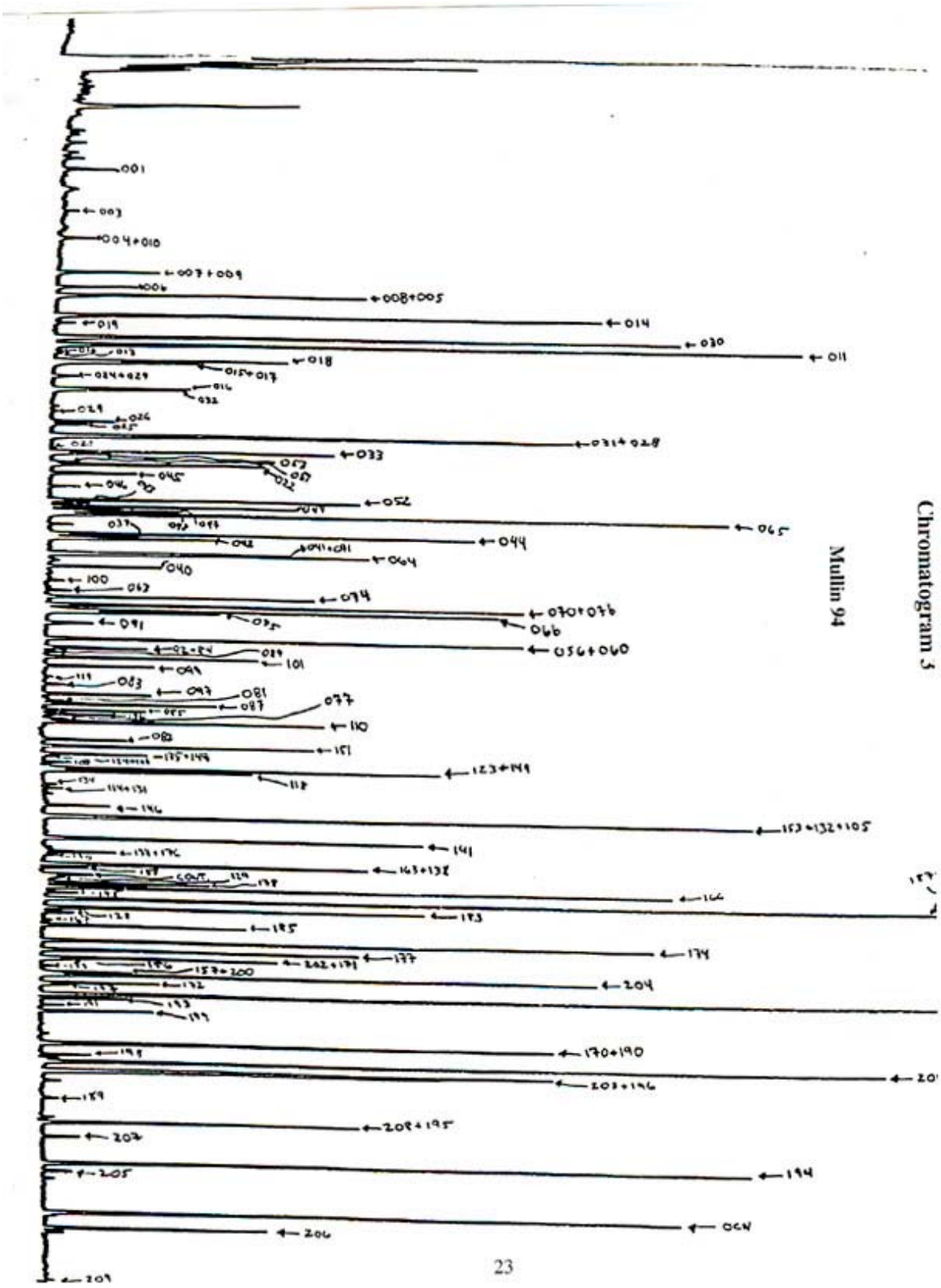
Print report and save Text File by clicking options in **Specify Report.**
Save the Method and the Text File in the same data folder.
Such as C:\HPChem\1\data\batch\M3995.d\m3995.m or m3995.txt
Calibration and the integration events will be saved in the method file.

NOTE: Sometimes it is necessary to increase the window more than 0.25% to find internal standard. If it goes more than 0.5%, rerun the sample in GC.

3. 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.


Chromatogram 3
Mullin 94

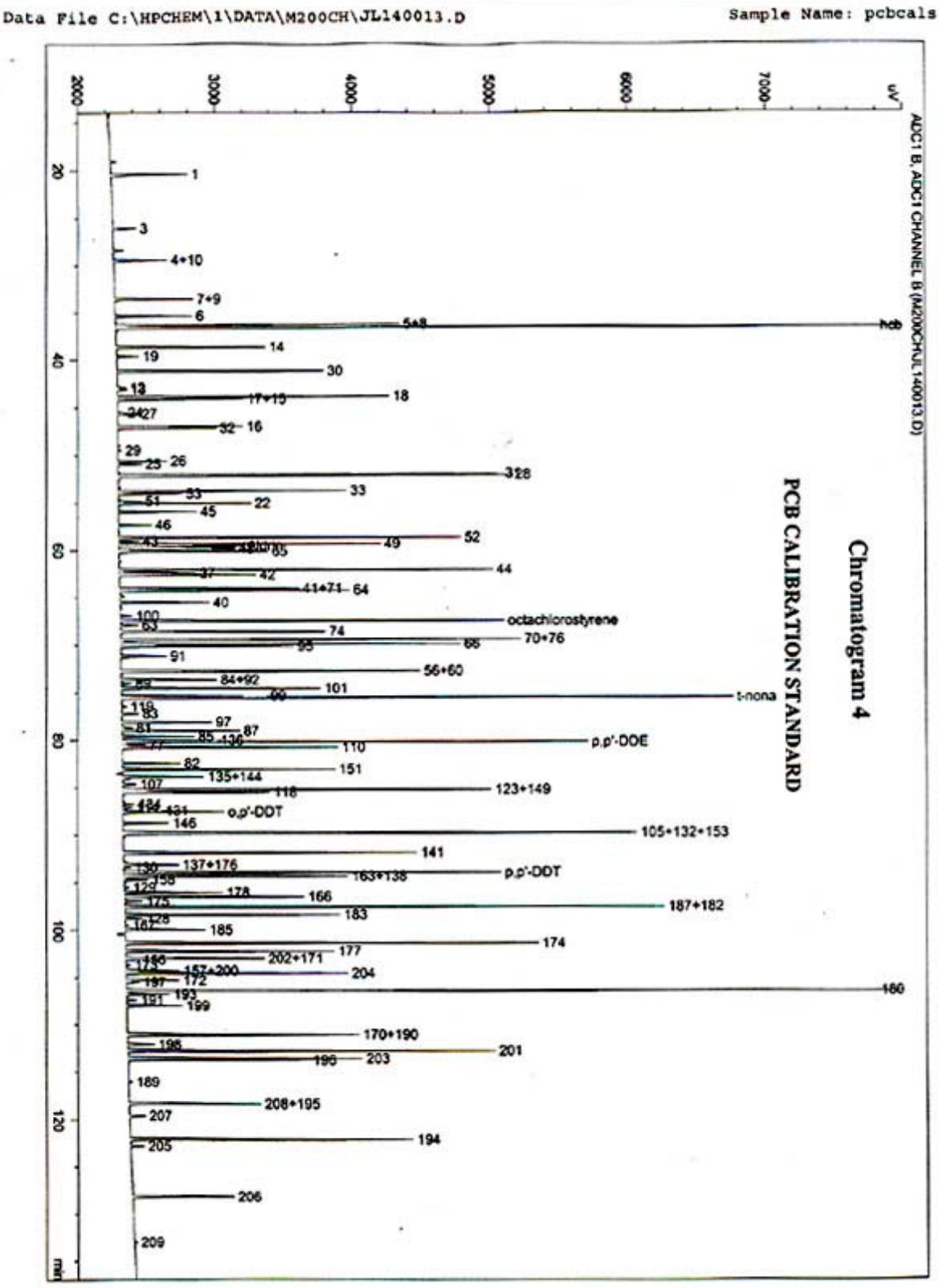

Data File C:\HPCHEM\1\DATA\M200CH\JL140013.D
Sample Name: pcbcals
ADC1 B, ADC1 CHANNEL B (M200CH\JL140013.D)
PCB CALIBRATION STANDARD
Chromatogram 4
octachlorostyrene
t-nona
p,p'-DOE
o,p'-DDT
p,p'-DDT
Instrument 1 7/17/00 4:06:36 PM Lidia Strandberg
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Chart 5

Method C:\HPCHEM\1\DATA\M200CH\JL140013.D\JL40013.M

****
Calibration Table
****

Calib. Data Modified : Monday, July 17, 2000 12:01:18 PM

Calculate : Internal Standard
Based on : Peak Area

Rel. Reference Window : 0.250 %
Abs. Reference Window : 0.000 min
Rel. Non-ref. Window : 0.300 %
Abs. Non-ref. Window : 0.000 min
Uncalibrated Peaks : not reported
Partial Calibration : Yes, identified peaks are recalibrate
Correct All Ret. Times: No, only for identified peaks

Curve Type : Linear
Origin : Included
Weight : Equal

Recalibration Settings:
Average Response : Average all calibrations
Average Retention Time: Floating Average New 75%

Calibration Report Options :
Printout of recalibrations within a sequence:
Calibration Table after Recalibration
Normal Report after Recalibration
If the sequence is done with bracketing:
Results of first cycle (ending previous bracket)

Default Sample ISTD Information (if not set in sample table):

ISTD #	ISTD Amount [ng/ml]	Name
1	8.00000	30
2	6.00000	204

Signal 1: ADC1 B, ADC1 CHANNEL B

RetTime [min]	Sig	Lvl	Amount [ng/ml]	Area	Amt/Area	Ref	Grp	Name
20.389	1	1	48.00000	3889.64404	1.23405e-2	1		1
26.026	1	1	28.00000	1084.25293	2.58242e-2	1		3
29.392	1	1	13.60000	2538.11865	5.35830e-3	1		4+10
33.553	1	1	4.80000	3977.69653	1.20673e-3	1		7+9
35.322	1	1	7.60000	3504.37280	2.16872e-3	1		6
36.267	1	1	56.00000	1.34119e4	4.17539e-3	1		5+8
36.565	1	1	20.00000	4.23298e4	4.72480e-4	1		hcb
38.665	1	1	20.00000	6948.48096	2.87833e-3	1		14
39.582	1	1	1.12000	1034.55811	1.08259e-3	1		19
41.170	1	1	8.00000	9296.78809	8.60512e-4	+I1		30
42.857	1	1	6.80000e-1	440.30130	1.54440e-3	1		12
43.052	1	1	3.90000e-1	477.72354	8.16372e-4	1		13
43.812	1	1	14.80000	1.42710e4	1.03707e-3	1		18
44.060	1	1	14.80000	6292.15820	2.35213e-3	1		17+15
45.475	1	1	5.20000e-1	270.63330	1.92142e-3	1		24
45.621	1	1	5.20000e-1	1004.82367	5.17504e-4	1		27
46.923	1	1	8.00000	5726.21045	1.39708e-3	1		16
47.086	1	1	7.60000	4498.63086	1.68940e-3	1		32
49.436	1	1	2.10000e-1	114.35308	1.83642e-3	1		29
50.559	1	1	2.80000	2149.41968	1.30268e-3	1		26
50.876	1	1	1.30000	1028.73389	1.26369e-3	1		25
51.992	1	1	18.80000	1.37926e4	1.36305e-3	1		31

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Chart 5

Method C:\HPCHEM\1\DATA\M200CH\JL140013.D\JL40013.M

RetTime [min]	Sig	Lvl	Amount [ng/ml]	Area	Amt/Area	Ref	Grp	Name
52.048	1	1	18.80000	1.56554e4	1.20086e-3	1		28
53.725	1	1	13.20000	1.05055e4	1.25649e-3	1		33
54.022	1	1	2.60000	3093.39404	8.40501e-4	1		53
54.790	1	1	7.20000e-1	1059.86914	6.79329e-4	1		51
55.023	1	1	11.60000	6039.63135	1.92065e-3	1		22
55.896	1	1	3.56000	3742.73145	9.51177e-4	1		45
57.310	1	1	1.60000	1543.56396	1.03656e-3	1		46
58.695	1	1	18.00000	1.65152e4	1.08991e-3	1		52
59.052	1	1	1.10000	974.98175	1.12823e-3	1		43
59.360	1	1	9.20000	1.23190e4	7.46813e-4	1		49
59.533	1	1	5.00000	5407.13623	9.24704e-4	1		Aldrin
59.741	1	1	4.00000	5318.77148	7.52053e-4	1		47
59.863	1	1	4.00000	5378.94531	7.43640e-4	1		48
60.124	1	1	5.00000	7042.40723	7.09985e-4	1		65
62.131	1	1	17.20000	1.75842e4	9.78151e-4	1		44
62.447	1	1	4.80000	3685.51465	1.30240e-3	1		37
62.618	1	1	5.60000	6644.91797	8.42749e-4	1		42
64.081	1	1	9.20000	8655.43555	1.06292e-3	1		41+71
64.243	1	1	7.20000	1.06757e4	6.74431e-4	1		64
65.515	1	1	3.76000	4225.79297	8.89774e-4	1		40
66.950	1	1	4.40000e-1	553.29071	7.95242e-4	1		100
67.452	1	1	9.38000	1.74705e4	5.36905e-4	1		octachlorostyrene
67.909	1	1	8.40000e-1	866.09753	9.69868e-4	1		63
68.556	1	1	7.60000	9437.62793	8.05287e-4	1		74
69.357	1	1	13.60000	1.89675e4	7.17017e-4	1		70+76
69.848	1	1	20.80000	1.56777e4	1.32673e-3	1		66
70.085	1	1	8.00000	8798.16504	9.09281e-4	1		95
71.130	1	1	2.04000	2313.68311	8.81711e-4	1		91
72.684	1	1	14.00000	1.46045e4	9.58612e-4	1		56+60
73.575	1	1	7.20000	5463.67969	1.31779e-3	1		84+92
73.994	1	1	4.00000e-1	490.58990	8.15345e-4	1		89
74.501	1	1	7.20000	9996.39258	7.20260e-4	1		101
75.260	1	1	2.96000	3456.45996	8.56368e-4	1		99
75.443	1	1	20.00000	2.92912e4	6.82799e-4	1		t-nona
76.404	1	1	1.12000e-1	232.90166	4.80890e-4	1		119
77.140	1	1	6.00000e-1	797.85919	7.52012e-4	1		83
78.039	1	1	2.24000	4307.79102	5.19988e-4	1		97
78.680	1	1	6.40000e-1	469.90161	1.36199e-3	1		81
78.969	1	1	4.00000	5913.79590	6.76385e-4	1		87
79.587	1	1	2.80000	2961.62817	9.45426e-4	1		85
79.950	1	1	3.00000	2478.11670	1.21060e-3	1		136
80.145	1	1	20.00000	2.13620e4	9.36240e-4	1		p,p'-DDE
80.446	1	1	9.20000e-1	1118.49402	8.22535e-4	1		77
80.712	1	1	7.60000	1.04039e4	7.30497e-4	1		110
82.418	1	1	1.80000	2784.20972	6.46503e-4	2		82
83.063	1	1	6.80000	1.00404e4	6.77263e-4	2		151
83.838	1	1	3.56000	4304.95361	8.26954e-4	2		135+144
84.643	1	1	5.20000e-1	650.19952	7.99755e-4	2		107
85.188	1	1	11.20000	1.88850e4	5.93065e-4	2		123+149
85.447	1	1	4.80000	7182.07373	6.68331e-4	2		118
86.750	1	1	2.90000e-1	574.60773	5.04692e-4	2		134
87.126	1	1	5.20000e-1	434.25958	1.19744e-3	2		114
87.400	1	1	1.16000e-1	261.68332	4.43284e-4	2		131
87.550	1	1	5.00000	4927.52783	1.01471e-3	2		o,p'-DDT
88.728	1	1	1.56000	2307.48218	6.76062e-4	2		146
89.756	1	1	17.20000	2.92835e4	5.87362e-4	2		105+132+153
91.838	1	1	6.80000	1.62132e4	4.19411e-4	2		141
93.098	1	1	1.04000	2725.89111	3.81527e-4	2		137+176
93.369	1	1	3.00000e-1	313.48572	9.56981e-4	2		130
93.877	1	1	20.00000	1.66085e4	1.20420e-3	2		p,p'-DDT
94.275	1	1	10.80000	1.44900e4	7.45344e-4	2		163+138
94.644	1	1	1.00000	1208.53186	8.27450e-4	2		158
95.501	1	1	5.20000e-2	278.49811	1.86716e-4	2		129
96.014	1	1	4.40000	4934.45020	8.91690e-4	2		178
96.422	1	1	5.00000	8805.98633	5.67796e-4	2		166

Instrument 1 7/17/00 12:18:33 PM Lidia Strandberg

Chart 5

Method C:\HPCHEM\1\DATA\M200CH\JL140013.D\JL40013.M

RetTime [min]	Sig	Lvl	Amount [ng/ml]	Area	Amt/Area	Ref	Grp	Name
96.910	1	1	8.00000e-1	887.94708	9.00955e-4	2		175
97.455	1	1	14.40000	2.75868e4	5.21990e-4	2		187+182
98.287	1	1	6.80000	1.00579e4	6.76084e-4	2		183
98.720	1	1	4.00000e-1	892.36230	4.48248e-4	2		128
99.425	1	1	1.96000e-1	160.08366	1.22436e-3	2		167
99.894	1	1	1.90000	3863.93311	4.91727e-4	2		185
101.299	1	1	12.80000	2.09136e4	6.12042e-4	2		174
102.144	1	1	6.80000	1.01970e4	6.66864e-4	2		177
102.877	1	1	3.16000	6654.34082	4.74878e-4	2		202+171
103.066	1	1	2.60000e-1	507.55533	5.12259e-4	2		156
103.658	1	1	1.52000e-1	239.47127	6.34732e-4	2		173
104.226	1	1	1.56000	2617.33374	5.96026e-4	2		157+200
104.480	1	1	6.00000	1.11033e4	5.40380e-4	2	+I2	204
105.243	1	1	2.24000	2631.60449	8.51192e-4	2		172
105.467	1	1	4.40000e-1	600.81616	7.32337e-4	2		197
106.320	1	1	24.40000	4.01548e4	6.07649e-4	2		180
106.747	1	1	1.68000	2265.75977	7.41473e-4	2		193
107.378	1	1	4.80000e-1	532.24811	9.01835e-4	2		191
107.965	1	1	1.72000	2773.85815	6.20075e-4	2		199
111.020	1	1	6.80000	1.32843e4	5.11882e-4	2		170+190
112.125	1	1	4.80000e-1	837.95673	5.72822e-4	2		198
112.765	1	1	16.80000	1.86500e4	9.00805e-4	2		201
113.565	1	1	8.40000	1.18791e4	7.07125e-4	2		203
113.760	1	1	8.00000	7149.11963	1.11902e-3	2		196
116.082	1	1	1.60000e-1	178.99550	8.93877e-4	2		189
118.323	1	1	3.20000	6724.45508	4.75875e-4	2		208+195
119.646	1	1	3.70000e-1	790.01111	4.68348e-4	2		207
122.075	1	1	7.20000	1.41756e4	5.07917e-4	2		194
122.844	1	1	4.40000e-1	751.04803	5.85848e-4	2		205
128.164	1	1	2.72000	5193.57422	5.23724e-4	2		206
133.053	1	1	4.80000e-2	110.64144	4.33834e-4	2		209

13 Warnings or Errors (10 first messages follow) :

Warning : Overlapping peak time windows at 51.992 min, signal 1
Warning : Overlapping peak time windows at 59.36 min, signal 1
Warning : Overlapping peak time windows at 59.741 min, signal 1
Warning : Overlapping peak time windows at 62.447 min, signal 1
Warning : Overlapping peak time windows at 64.081 min, signal 1
Warning : Overlapping peak time windows at 75.26 min, signal 1
Warning : Overlapping peak time windows at 79.95 min, signal 1
Warning : Overlapping peak time windows at 87.4 min, signal 1
Warning : Overlapping peak time windows at 93.098 min, signal 1
Warning : Overlapping peak time windows at 102.877 min, signal 1

Peak Sum Table

No Entries in table

Instrument 1 7/17/00 12:18:33 PM Lidia Strandberg

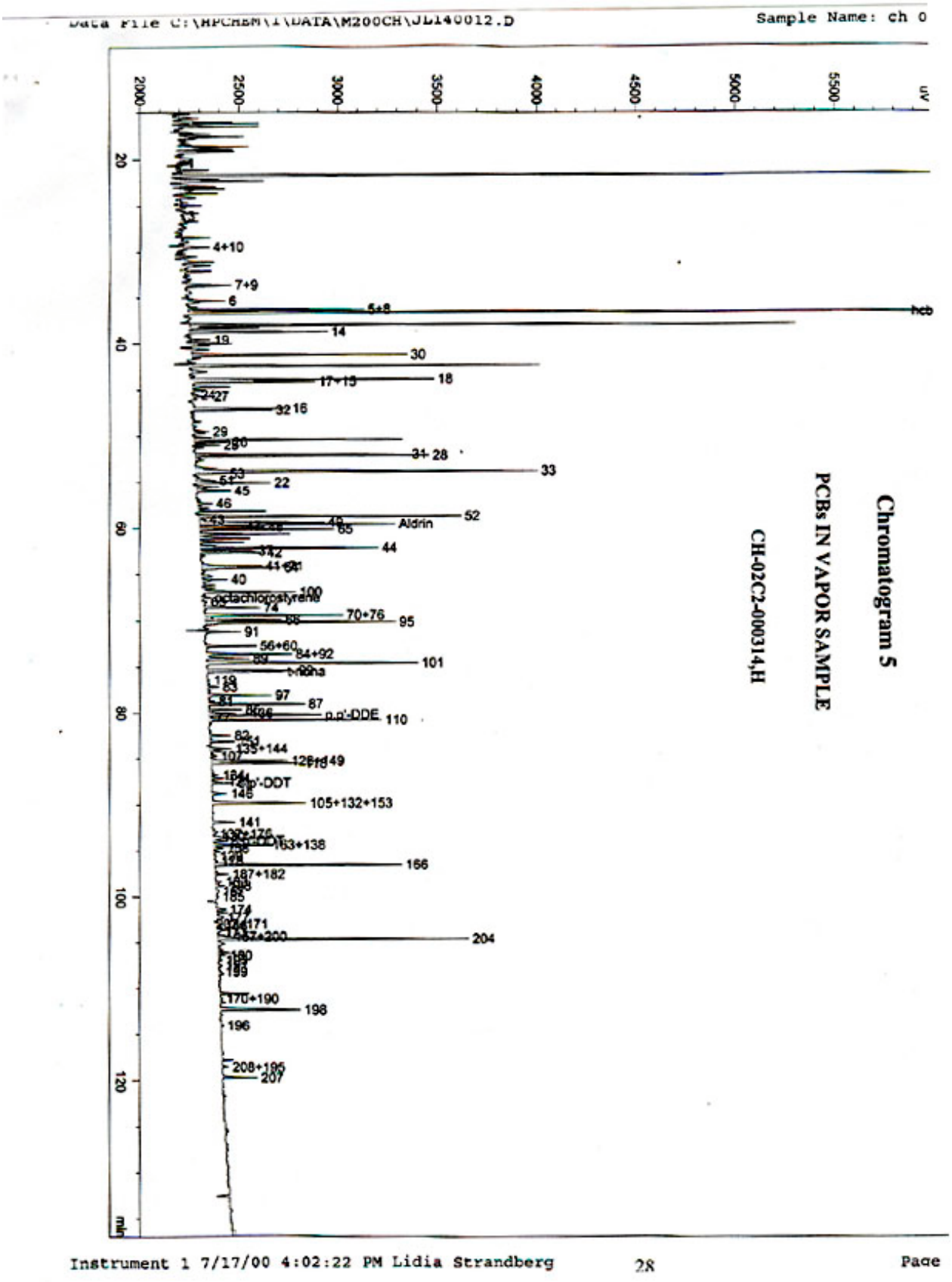
Data File C:\MPCHEM\1\DATA\M200CH\JD140012.D
Sample Name: ch 0
Chromatogram 5
PCBs IN VAPOR SAMPLE
CH-02C2-000314,H
4+10
7+9
6
5+8
hcb
14
19
30
17+15
18
32 16
29
31 28
33
22
45
46
52
Aldrin
44
40
74
70+76
95
91
56+60
84+92
101
97
87
p,p'-DDE
110
135+144
49
p,p'-DDT
105+132+153
141
163+138
166
187+182
174
171
204
170+190
198
196
208+195
207
Instrument 1 7/17/00 4:02:22 PM Lidia Strandberg
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Chart 6

Data File: C:\HPCHEM\1\DATA\M200CH\JL140012.D\JL140012.M
Sample name: ch 02c2 000314,h

Ret. Time (min.)	Peak Type	Internal Standard Peak Area	Rel. Res. Factor	Report Amount (ng)	ISTD #	Is ISTD	Compound Name
20.386	PP N	48	14.341	0.690	1		1
26.035	VV	59	30.010	1.804	1		3
29.419	PV	1054	6.227	6.647	1		4+10
33.599	VV	2147	1.402	3.047	1		7+9
35.352	VV	1337	2.520	3.410	1		6
36.294	VV	6006	4.852	29.500	1		5+8
36.601	VV	39080	0.549	21.723	1		hcb
38.699	VV	5149	3.345	17.437	1		14
39.614	VV	438	1.258	0.558	1		19
41.205	PV	7902	1.000	8.000	1	X	30
0.000		0	0.000	0.000	1		12
0.000		0	0.000	0.000	1		13
43.840	PV	10419	1.205	12.711	1		18
44.085	VV	4803	2.733	13.290	1		17+15
45.494	PV	114	2.233	0.257	1		24
45.648	VV	599	0.601	0.365	1		27
46.947	VV	3363	1.624	5.528	1		16
47.111	VP	2772	1.963	5.509	1		32
49.464	VV	468	2.134	1.011	1		29
50.588	VV	1192	1.514	1.827	1		26
50.895	VP	852	1.469	1.267	1		25
51.950	VVA+	6338	1.584	10.164	1		31
52.086	VV	7704	1.396	10.884	1		28
53.819	VV	16058	1.460	23.737	1		33
54.044	VP	998	0.977	0.987	1		53
54.809	VV	728	0.789	0.582	1		51
55.049	VV	2524	2.232	5.704	1		22
55.922	VV	1320	1.105	1.477	1		45
57.330	VV	528	1.205	0.644	1		46
58.718	VV	9993	1.267	12.813	1		52
59.091	VV	343	1.311	0.455	1		43
59.386	VV	4750	0.868	4.174	1		49
59.562	VV	7767	1.075	8.449	1		Aldrin
59.769	VV	1318	0.874	1.166	1		47
59.895	VV	2892	0.864	2.530	1		48
60.151	VV	5209	0.825	4.351	1		65
62.156	VV	6912	1.137	7.954	1		44
62.443	VVA+	1804	1.514	2.764	1		37
62.622	VV	2818	0.979	2.794	1		42
64.103	VV	2455	1.235	3.070	1		41+71
64.269	VV	3064	0.784	2.431	1		64
65.540	VV	916	1.034	0.958	1		40
66.880	VV	3961	0.924	3.705	1		100
67.477	VV	219	0.624	0.138	1		octachlorostyrene
67.946	VV	57	1.127	0.065	1		63
68.582	VV	2165	0.936	2.051	1		74
69.382	VV	5310	0.833	4.479	1		70+76
69.869	VV	2854	1.542	4.454	1		66
70.109	VV	7897	1.057	8.447	1		95
71.148	PV	1133	1.025	1.175	1		91
72.705	VV	1848	1.114	2.084	1		56+60
73.589	PV	4237	1.531	6.569	1		84+92
74.059	VP	1742	0.948	1.671	1		89
74.525	PP	8856	0.837	7.504	1		101
75.338	VV	3373	0.995	3.398	1		99
75.453	VV	2619	0.793	2.104	1		t-nona
76.428	VV	35	0.559	0.020	1		119
77.159	VV	365	0.874	0.323	1		83
78.057	VV	2642	0.604	1.616	1		97
78.701	VV	233	1.583	0.373	1		81
78.989	VV	3839	0.786	3.055	1		87
79.606	VV	1295	1.099	1.440	1		85
80.010	VVA+	854	1.407	1.216	1		136
80.162	VV	4361	1.088	4.803	1		p,p'-DDE
80.466	VV	126	0.956	0.122	1		77

Chart 6

Data File: C:\HPCHEM\1\DATA\M200CH\JL140012.D\JL140012.M
Sample name: ch 02c2 000314,h

Ret. Time (min.)	Peak Type	Peak Area	Rel. Res. Factor	Amount (ng)	ISTD #	Is ISTD	Compoun Name
80.731	VV	6876	0.849	5.909	1		110
82.438	BV	840	1.196	0.590	2		82
83.082	VV	958	1.253	0.705	2		151
83.854	PV	892	1.530	0.802	2		135+144
84.661	VV	214	1.480	0.186	2		107
85.209	VV	3232	1.097	2.084	2		123+149
85.466	VB	3535	1.237	2.568	2		118
86.766	VV	279	0.934	0.153	2		134
87.083	VV	557	2.216	0.725	2		114
87.433	VVA+	186	0.820	0.090	2		131
87.570	VP	822	1.878	0.907	2		o,p'-DDT
88.733	PB	780	1.251	0.573	2		146
89.767	BB	5357	1.087	3.421	2		105+132+153
91.834	VB	1033	0.776	0.471	2		141
93.106	VV	140	0.706	0.058	2		137+176
93.394	VV	199	1.771	0.207	2		130
93.891	VV	578	2.228	0.757	2		p,p'-DDT
94.306	VV	2586	1.379	2.095	2		163+138
94.659	VV	403	1.531	0.362	2		158
95.519	VV	121	0.346	0.024	2		129
96.043	VV	189	1.650	0.183	2		178
96.439	VB	7536	1.051	4.652	2		166
0.000		0	0.000	0.000	2		175
97.470	VV	563	0.966	0.319	2		187+182
98.303	VV	266	1.251	0.196	2		183
98.734	VV	395	0.830	0.193	2		128
99.324	VV	307	2.266	0.409	2		167
99.901	VV	96	0.910	0.051	2		185
101.306	VV	430	1.133	0.286	2		174
102.174	VV	467	1.234	0.338	2		177
102.901	PV N	334	0.879	0.173	2		202+171
103.079	VBA	156	0.948	0.087	2		156
103.773	VVA+	150	1.175	0.104	2		173
104.224	VV	618	1.103	0.401	2		157+200
104.495	VV	10213	1.000	6.000	2	X	204
0.000		0	0.000	0.000	2		172
0.000		0	0.000	0.000	2		197
106.334	VV	312	1.124	0.206	2		180
106.854	VV	144	1.372	0.116	2		193
107.412	VV	99	1.669	0.097	2		191
108.087	VV	107	1.147	0.072	2		199
111.045	VV	163	0.947	0.091	2		170+190
112.173	VV	4336	1.060	2.700	2		198
0.000		0	0.000	0.000	2		201
0.000		0	0.000	0.000	2		203
113.946	VV	146	2.071	0.178	2		196
0.000		0	0.000	0.000	2		189
118.456	VV	175	0.881	0.091	2		208+195
119.581	VV	1252	0.867	0.638	2		207
0.000		0	0.000	0.000	2		194
0.000		0	0.000	0.000	2		205
0.000		0	0.000	0.000	2		206
0.000		0	0.000	0.000	2		209
				343.749			

Chart 7

Method C:\HPCHEM\1\DATA\M200CH\JL140012.D\JL140012.M

Detector Default Integration Event Table "Event_ADC"

Event	Value	Time
Initial Slope Sensitivity	10.000	Initial
Initial Peak Width	0.040	Initial
Initial Area Reject	10.000	Initial
Initial Height Reject	10.000	Initial
Initial Shoulders	OFF	Initial
Negative Peak ON		0.000
Baseline Now		17.386
Baseline Now		28.595
Area Sum ON		33.352
Area Sum OFF		33.476
Area Sum ON		33.706
Area Sum OFF		33.848
Baseline Now		40.998
Baseline Now		47.476
Area Sum ON		49.301
Area Sum OFF		49.425
Area Sum ON		51.853
Area Sum OFF		52.042
Area Sum ON		57.144
Area Sum OFF		57.257
Area Sum ON		62.349
Area Sum OFF		62.536
Area Sum ON		68.042
Area Sum OFF		68.116
Area Sum ON		69.649
Area Sum OFF		69.768
Area Sum ON		71.258
Area Sum OFF		71.360
Baseline Now		73.132
Area Sum ON		79.938
Area Sum OFF		80.075
Baseline Now		82.187
Area Sum ON		87.352
Area Sum OFF		87.506
Area Sum ON		102.305
Area Sum OFF		102.490
Baseline Now		103.305
Area Sum ON		103.617
Area Sum OFF		103.919
Baseline Now		126.269
Baseline Now		129.625
Baseline Now		134.182
Baseline Now		135.477

Apply Manual Integration Events: No

Instrument 1 7/17/00 2:28:30 PM Lidia Strandberg

Appendix

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Method: C:\HPCHEM\1\METHODS\MULLIN.M of 10/1/99 8:03:05 AM


                              Method Information

     Method was originally suggested by Michael Mullin in August, 1994.


                          Injection Source and Location

     Injection Source:    HP GC Injector

     Injection Location: Front
================================================================================
                                HP6890 GC 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  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:  70.0 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 DB-5
   Max temperature:  325 '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:  10.0 mL/min (On)
   Makeup Gas Type: Nitrogen
   Electrometer:  On

SIGNAL 1                                    SIGNAL 2


Instrument 1 6/29/00 2:12:25 PM LIDIA STRANDBERG                        Page 1
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Appendix

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Method: C:\HPCHEM\1\METHODS\MULLIN.M of 10/1/99 8:03:0


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


                                7673 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


Instrument 1 6/29/00 2:12:25 PM LIDIA STRANDBERG
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Appendix

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Method: C:\HPCHEM\1\METHODS\1701.M of 7/19/00 10:13:50 AM


================================================================
                          HP6890 GC METHOD
================================================================

OVEN
   Initial temp:  100 'C (On)                    Maximum temp:
   Initial time:  1.00 min                       Equilibration t
   Ramps:
      #  Rate  Final temp  Final time
      1 10.00       160        0.00
      2  1.00       240        0.00
      3 10.00       260       20.00
      4   0.0(Off)
   Post temp:  100 'C
   Post time:  0.00 min
   Run time:  109.00 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:  70.0 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 DB-1701
   Max temperature:  280 'C
   Nominal length:  60.0 m
   Nominal diameter:  250.00 um
   Nominal film thickness:  0.25 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 (NC
   Temperature:  350 'C (On)
   Mode:  Constant makeup flow
   Makeup flow:  10.0 mL/min (On)
   Makeup Gas Type: Nitrogen
   Electrometer:  On


                          7673 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


Instrument 1 7/19/00 10:14:06 AM LIDIA STRANDBERG
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Appendix

Method: C:\HPCHEM\1\METHODS\1701.M of 7/19/00 1

Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes
PostInjection Dwell	0.00 minutes

Back Injector:
No parameters specified

Instrument 1 7/19/00 10:14:06 AM LIDIA STRANDBERG