Analysis of PCBs and Pesticides in Air and Precipitation Samples:

IADN Project

Gas Chromatography Procedure

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1.0 Routine GC Maintenance

1.1 Gas Tanks

1.1.1 Check gas tanks. Tanks should not go dry. While changing the tank, lower the temperature of the oven down to 40°C. Leave it at 40°C for about half an hour after changing the tank to get rid of air or oxygen that was drawn in.

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

1.2 GC Baking

After every GC run bake the oven at 280°C for one hour. After every other run also bake the injector and the detector at 280°C and 380°C.

1.3 Septum

1.3.1 Preparation of Septum

1.3.1.1 Place the septa in a small beaker. Cover with aluminum foil.

1.3.1.2 Place the beaker in GC oven and bake the septa while baking the column at 280°C.

1.3.1.3 Store the clean septa in the beaker keeping the foil lid intact and use as needed. Always use tweezers to get clean septum out.

1.3.2 Changing of Septum

1.3.2.1 After every 60 samples or so change the septum.

1.3.2.2 Cool the oven down to 40°C.

1.3.2.3 Remove autosampler towers.

1.3.2.4 Remove septum nut and take the old septum out. Discard.

1.3.2.5 Using clean Q-tips soaked in hexane, wipe off the septum holder until no more dirt is seen on Q-tips.

1.3.2.6 Put in pre-cleaned 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.

1.4 Checking Background

1.4.1 Background signal in GC varies from 28 to 32. 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.

1.5 Checking Standard

1.5.1 Mullin 94 standard and Mixed Pesticide Standard should be monitored to check peak detection and peak broadening or tailing. If the peak shapes are not satisfactory, column should be clipped. More than 90 peaks should be detected in PCB standards and cong. 17 and 18 should be separated. If not, install a new column.

1.6 Checking Leaks and Gas Flow

1.6.1 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.

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

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

2.0 GC Cleaning: Clipping Old Column or Installing a New Column

2.1 Taking Apart

2.1.1 Turn oven, injector and detector off.

2.1.2 Turn hydrogen and nitrogen off. Wait until everything cools down.

2.1.3 Take the autosampler tower off.

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

2.1.5 Open the oven. Take the columns out (by detaching from injector and detector ends).

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

2.1.7 Place the columns on the workbench.

2.1.8 Unscrew the holder nut underneath the injection liner. There is one gold seal and a washer in it. Washer and seal need to be replaced each time it is taken apart. Clean these parts by ultrasonication with Dichloromethane and Hexane.

2.1.9 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.

2.2 Assembling Injection Port and Liner

2.2.1 After ultrasonication air dry each part. 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.

2.2.2 Insert a new liner.

2.2.3 Put a viton O-ring on the liner. Put the big nut on and tighten it. Put a clean septum. Cover the septum with septum nut. Tighten with wrench.

2.3 Clipping Column

2.3.1 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 the ferrule with conical end pointing towards the open end of the column.

2.3.2 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.

2.3.3 Measure 25mm from the tip of the column. Mark this point with Liquid Paper®.

2.3.4 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 ¼ turn after hand tight. Do not overtighten.

2.3.5 Take the nut off the detector end of the column. Put the nut and the ferrule on the column in the same way as in the injector end. Clip the column and check for the nice clean cut. Insert the column all the way up until it does not go any more. Pull down about 1 mm and tighten the screw. 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.

2.4 Checking Leaks and Gas Flow

2.4.1 Turn H_2 and N_2 on. Check leaks with a leak detector. Check around the septum, at the injector and at the detector end of the column. Check that the head pressure is 24 psi.

2.4.2 Check the gas flow with a bubble meter. Approximate gas flow are as follows:

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

2.5 Assembling

2.5.1 Replace autosampler tower.

2.5.2 Turn heated zones on.

2.5.3 Turn oven on and set the temperature to 40°C for an hour. Change oven temperature to 70°C and leave another hour.

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

Baking temperature :

Oven:	280°C
Injector A:	280°C
Injector B:	280°C
Detector A:	380°C
Detector B:	380°C

2.5.5 If it is a new column, bake injector and detector. Column can be baked by ramping it 1° or 2° per minute to 280°C. Hold there for one hour.

2.5.6 If blank run looks satisfactory, put in a standard and check.

3.0 Routine GC Operation

3.1 GC condition and oven temperature program:

PCBs, Hexachlorobenzene, and DDE are eluted in the hexane fraction, whereas the other chlorinated pesticides are eluted in the 50% dichloromethane in hexane fraction after 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 1.0, June 1995.

Gas Chromatograph used for analysis of PCBs and pesticides is Hewlett Packard 5890 with 7673A Autosampler. Operation is controlled by the Integrator Hewlett Packard 3396. Data acquisition is done by Hewlett Packard Peak 96. Finally, data analyses are done in Hewlet Packard 3365 ChemStation.

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

Temperature Program:	Initial temp.100°C
Initial time:	1 min.
Rate:	1°C/min.
Final temp.:	240°C
Rate A:	10°C
Final temp A:	280°C
Final time:	20 min.
Purge on:	3 min.
Purge off:	150 min.
Run time:	165 min.

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

3.2 GC Pre-run

3.2.1 Check to see if there is sufficient H_2 for operation. If not, change tanks.

3.2.2 If necessary, change septum.

3.2.3 Bake oven at 280°C for one hour. Bake injector and detector at 280°C and 380°C respectively about every other time the oven is baked.

3.2.4 Cool oven to 100°C, injector to 250°C, and detector to 350°C.

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

3.3 Preparing Sequence in ChemStation

3.3.1 Editing Sequence

3.3.1.1 Sequence Parameters

Sequence	Edit Sequence Parameters

Type in operator's name and subdirectory name. Type in information about calibration standard and spikes in comment.

3.3.1.2 Sequence Table

Enter "From Vial" and "To Vial" number.

3.3.1.3 Sample Table

Type in sample ID's. First vial should be hexane. Second vial should be calibration standard. Third vial should be a Performance standard. Other vials are actual samples. At the end of each sample ID indicate whether the sample is a hexane fraction or 50% fraction (H,F1). Repeat hexane blank and a fresh standard after every set of samples.

3.3.1.4 Print Sequence

x Sequence Parameters
x Sample Table

3.4 Starting a GC run

3.4.1 Programming integrator

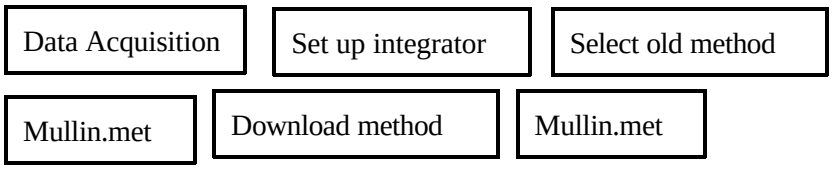
Set runnum 1
Set Date mo/day/year
Shift edit seq

The integrator will ask questions about method to load and autosampler sequence. Enter all information.

3.4.2 Programming peak 96

Integrator 2

Follow the keys:



3.4.3 Set up PC

Follow the keys:

Generate new

Data prefix J3095 (June 30, 95. It will pick up the vial number from the integrator)

Check export path/peak/export 2

Data to ChemStation Y

3.4.4 Transfer information from integrator to PC

Follow the keys:

Utilities → Transfer → Integrator to PC → IBCOL.SEQ

3.4.5 Start a GC run

Follow the keys:

Data Acquisition → Start Seq

3.4.6 Post GC run

After the GC run is over copy the .d files on floppy disks.

Sequence

27 Apr 95 05:05 PM
age 1
Sequence: C:\HPCHEM\1\SEQUENCE\DEFAULT.SEQ

Operator: Ilora Basu

Sequence preparation date: 15 Jun 92 09:14 AM

Data File Subdirectory: ON94CH

Part of methods to run: full method

On a barcode mismatch: inject anyway

Comment:
was spiked with 8 ng of 30 and 6 ng of 204

Sequence Table

Inj.	Seq. Line	Cal. Line	Method Name	From Vial	To Vial	Inj/ Vial
FRONT	1			1	16	1
REAR	1					

Sample Table

Vial Num.	Sample Name	Sample Amount	Multiplier	ISTD Amount
1	hexane blank			
2	pcbcalst950427			
3	pcbperfst950427			
4	LBC 950113,H			
5	MSC 950112,H			
6	EH02C1 941024,H			
7	EH02C2 941024,H			
8	EH01C 941105,H			
9	SH01C 941024,H			
10	SH02C 941025B,H			
11	SH01C 941105,H			
12	TH01C 941024,H			
13	TH02C 941024,H			
14	TH01C 941105,H			
15	PCBCALST950427			
16	Hexane blank			

Chart 1

4.0 HP 3365 Chemstation Initialization and Baseline Correction

4.1 Copy the .d files from Peak 96 on to the floppy disks.
Copy the .d files to HP 3365 ChemStation.

4.2 Load IBPCBN.MTH or IBPEST2.MTH

4.3 Loading Chromatogram

Files	Load Data File

Sub directory	Sample Vial Number

Chromatogram will appear on screen.

4.4 Baseline Correction

4.4.1 By Integration Event

Go to "command line" and type in Clrevents.
Correct starting parameters and then make baseline corrections by using Baseline Now, and Area Sum.

Starting Parameters

Integration	Integration Events

Initial Area Reject	10	Initial
Initial Peak Width	0.04	Initial
Shoulder Detection	OFF	Initial
Initial Threshold	-4	Initial
Integrator On	0.0	
Negative peak on	0.0	

4.4.2 Integrate the chromatogram

Change the scale axis for close view of each peak.

4.4.3 Baseline Now:
This command is used to get a straight base line.

Integration	Possible Events	Baseline Now

Click cursor where you want the baseline.

Integration | Integrate

If do not like changes go to and delete. Integration Events

4.4.4 Area Sum and Negative Peak:

This command is used to split a peak.

Integration | Possible Events | Neg. Peak

Turn negetive peak on and off in the area to split the peak

Integration | Possible Events | Area Sum

Move the cursor and click on the area to split.

Notice: Area Sum On\Off does not work with Neg. Peak On.

5.0 Pesticide Data Reduction in 50% Fraction

5.1 Standard

5.1.1 Integration and Peak Identification

Call standard chromatogram and correct baseline and integrate

Compounds	GC Retention time min.(approx.)	concentration ng/mL
á-HCH	35	20
ã-HCH	40	20
Cong 65(ISTD)	59	20

Compounds	GC Retention time min.(approx.)	concentration ng/mL
ã-Chlordane	70	20
cong 155(ISTD)	72	20
á-Chlordane	73	20
t-Nonachlor	74	20
Dieldrin	77	20
DDD	86	20
DDT	93	20
Dibutylchlorendate	110	10

5.1.2 Printing Report and Saving Text File

Graphics | Annotate

Type in the sample name and add it on the chromatogram.

Check Calibration Settings under Reports.

Mixed Pest.	Standard.
Ref. Window	1
Nonref. Window	1
Units of Amount	ng
Sample ISTD	20
Fit: Linear	Origin: Force

Check report on screen before printing

Reports | Specify Report

X File (X Auto)	X Area
X Screen	X ISTD (if calib. is replaced).
X Report	X Percent (if new calib is made).

If the report looks alright, print the report.

Reports | Print Report

To print report on paper go back to Specify Report and add Printer and Chromatogram to the options selected.

Print integration events and calibration settings

File → Return to Top

Method → File → Integration Events

5.1.3 Preparing New Calibration

Set up initial calibration table by identifying the standard peaks in the area percent report.

Reports → Prp Calib/Recalib. → New Table

Manually type in the amount and name of each analyte with the retention times from the area percent printout.

Highlight "yes" in Reference ISTD peak boxes for Congener 65 .

OK

Do you want to delete all the peaks with the amount of zero? Yes

Save to method? Yes

You will get a printout for calibration table.

5.1.4 Replacing Previous Calibration

For subsequent standard runs with analyte peaks all correctly identified in ISTD report the calibration table is replaced.

Reports → Prep Calib/Recalib.

Recalibrate → Replace → OK

Save to method? Yes

Will get a printout of calibration table.

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.

5.1.5 Saving Calibration on Data File

File	Command Line

Type in: SAVETBL C:\HPCHEM\1\DATA\SUBDIR.\0**R0101.CAL

5.1.6. Saving Event on Data File

File	Command Line

Type in: SAVE C:\HPCHEM\1\DATA\SUBDIR.\0**R0101.EVT

5.2 Samples, 50% Fraction

5.2.1 Integration - See Section 4.4.

5.2.2 Printing Report and Saving Text File.
See previous Section 5.1.2 with the following exceptions:

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.

5.2.3 Saving Events - See Section 5.1.6

5.3 Copying .Txt Files

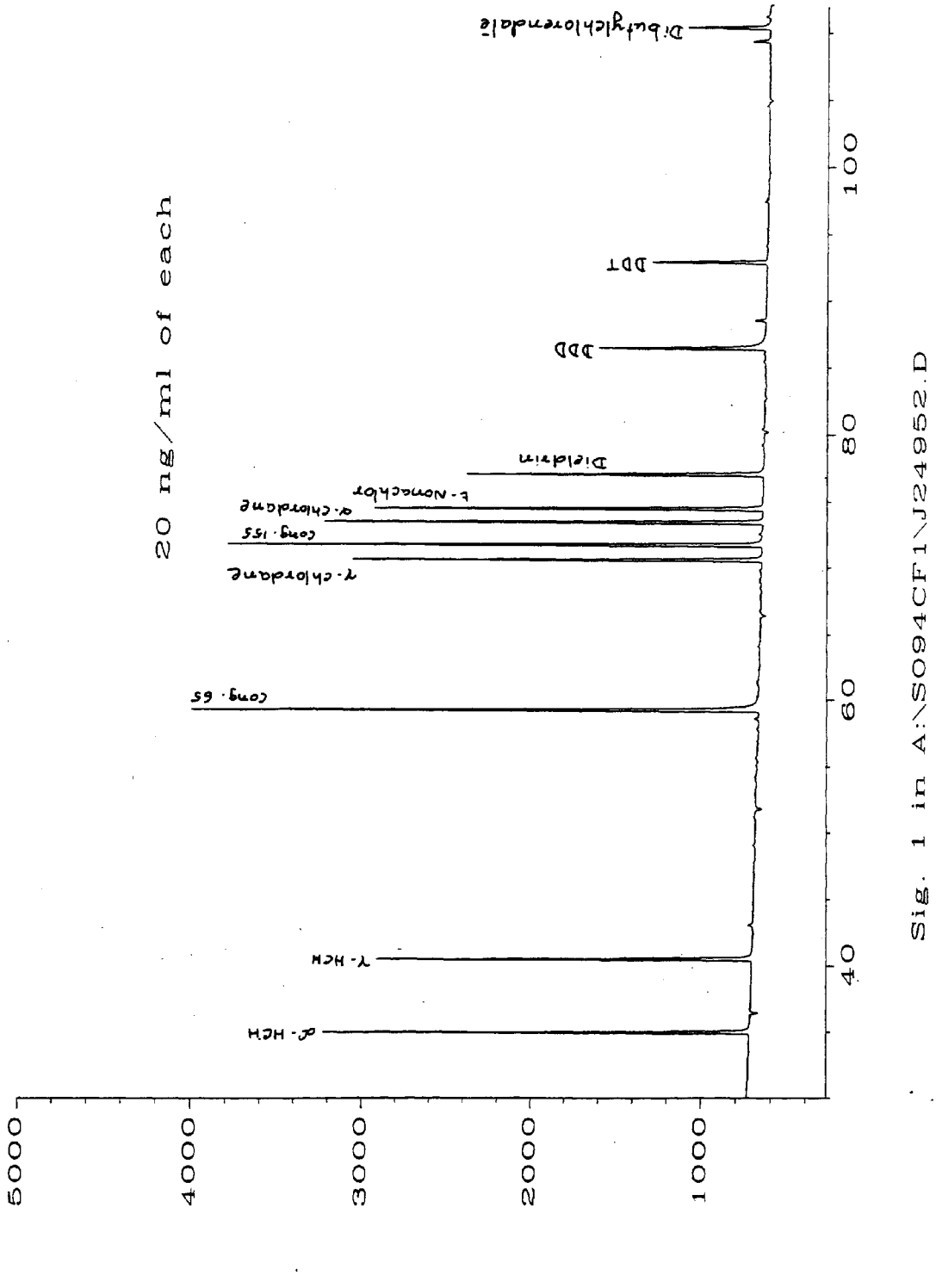
After one set of data is reduced write comments in the text files.

Program manager	Accessories	Note pad

Click on C, HPCHEM 1, data, subdirectory and .txt files.

Call .txt file one by one and write down comments about spike, dilution, reinjection etc.

Save .txt files after writing comments.



Chromatogram 1. Pesticide Calibration Standard

09 Aug 95 08:33 AM
Method: C:\HPCHEM\1\METHODS\IBPEST2.MTH

Calibration Table

Pk#	RT	Lvl	ng	Amt/Area	Ref Istd	I#	Name
1	34.931	1	20.0	1.2744e-003		1	A-HCH
2	40.469	1	20.0	1.3924e-003		1	G-HCH
3	59.180	1	20.0	7.8613e-004	Ref ISTD	1	CONG 65
4	70.521	1	20.0	1.0422e-003		1	G-CHLORDANE
5	71.658	1	20.0	8.3836e-004		1	CONG 155
6	73.377	1	20.0	9.9121e-004		1	A-CHLORDANE
7	74.405	1	20.0	1.0712e-003		1	T-NONA
8	76.972	1	20.0	1.4431e-003		1	DIELDRIN
9	86.359	1	20.0	2.4822e-003		1	DDD
10	92.832	1	20.0	3.8777e-003		1	DDT
11	110.300	1	10.0	2.2105e-003		1	DBC

Calibration Settings

Title:

Reference window:	0.500 %
Non-reference window:	0.500 %
Units of amount:	ng
Multiplier:	1.0
RF uncal peaks:	0.0
Sample Amount:	0.0

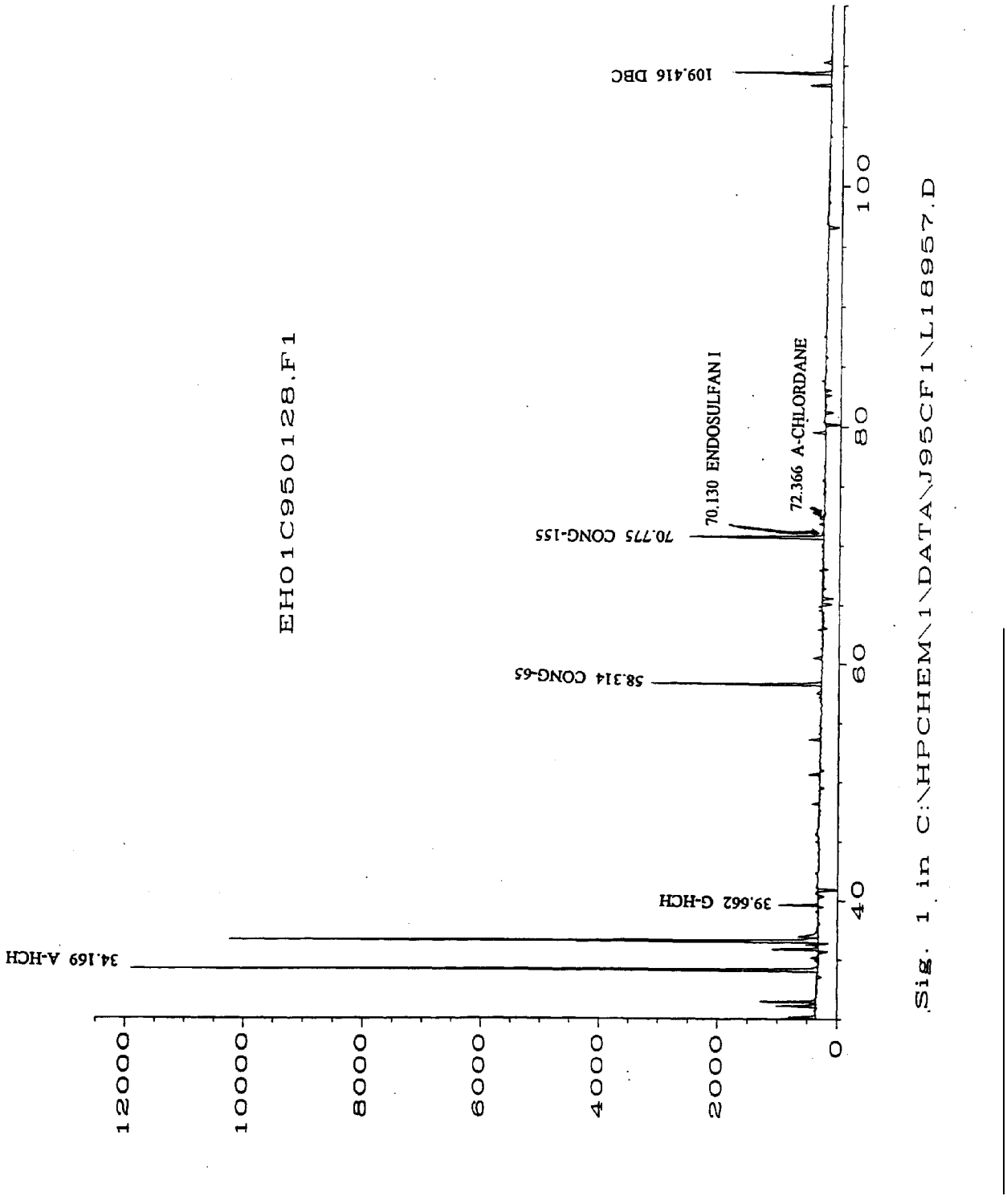
Sample ISTD Information

I#	Amount
1	20.0

Multilevel Information

Fit: Linear
Origin: Force

Chart 2



Chromatogram 2. Pesticides in Vapor Sample

Internal Standard Report

Data File Name : C:\HPCHEM\1\DATA\J95CF1\L18957.D
Operator :
Instrument : HP5890A
Sample Name :
Run Time Bar Code:
Acquired on : JUL 19, 1995 02:43:09
Report Created on: 09 Aug 95 12:33 PM
Last Recalib on : 09 AUG 95 10:20 AM
Multiplier : 1

Page Number : 1
Vial Number : 0
Injection Number :
Sequence Line :
Instrument Method:
Analysis Method : IBPEST2.MTH
Sample Amount : 0
ISTD Amount : 20

Sig. 1 in C:\HPCHEM\1\DATA\J95CF1\L18957.D

Ret Time	Area	Type	Width	Ref#	ng	Name
34.169	70370	PV	0.093	1	71.797	A-HCH
39.662	3866	PV	0.096	1	4.038	G-HCH
58.314	22586	VV	0.123	1	20.759	CONG 65
69.780	142	VV -	0.000	1	0.142	G-CHLORDANE
70.775	19005	BV	0.132	1-IR	20.000	CONG 155
72.366	545	BVA	0.109	1	0.549	A-CHLORDANE
73.506	* not found *			1		T-NONACHLOR
76.043	* not found *			1		DIELDRIN
85.473	* not found *			1		DDD
91.939	* not found *			1		DDT
109.416	13402	PV	0.128	1	25.822	DBC

Time Reference Peak	Expected RT	Actual RT	Difference
5	70.785	70.775	-0.0%

Not all calibrated peaks were found

Chart 3

09 Aug 95 12:33 PM
Method: C:\HPCHEM\1\METHODS\IBPEST2.MTH

Integration Events

Events:	Value:	Time:
Initial Area Reject	50	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	-4	INITIAL
Baseline Now		0.000
Negative Peak ON		0.000
Baseline Now		33.943
Baseline Now		62.680
Negative Peak OFF		72.300
Area Sum ON		72.400
Area Sum OFF		72.722
Negative Peak ON		72.730
Baseline Now		81.033
Baseline Now		82.107
Baseline Now		87.837
Baseline Now		88.403
Baseline Now		89.180

Calibration Settings

Title:

Reference window:	0.500 %
Non-reference window:	0.500 %
Units of amount:	ng
Multiplier:	1.0
RF uncal peaks:	0.0
Sample Amount:	0.0

Sample ISTD Information

I#	Amount
1	20.0

Multilevel Information

Fit: Linear
Origin: Force

Chart 4

6.0 PCB and Pesticide Data Reduction in Hexane Fraction

6.1 Mullin 94 Standard

6.1.1 Integration and Peak Identification

Load standard chromatogram and correct baseline according to Section 4.2 and 4.3. Integrate and divide the chromatogram in five to six sections. Identify PCBs from Mullin's 94 chromatogram and pesticides (HCB and DDE) from Mixed Pesticide Standard.

6.1.2 Printing Report and Saving Text Files

Graphics | Annotate

Click in text box and type in sample name. Click on | Add | , and then on chromatogram where you want sample name to be placed.

Check Calibration Settings under Reports.

Ref. Window	1.0
Nonref. Window	1.0
Units of Amount	ng
Sample ISTD #1	8
Sample ISTD #2	6

Fit: Linear Origin: Force

Check report on screen before printing

Reports | Specify Reports

X File (X Auto)	X Area
X Screen	X ISTD (if calib. is replaced).
X Report	X Percent (if new calib is made).

Report | Print Reports

To print report on paper go back to Specify Report and add Printer and Chromatogram to the options selected.

Print integration events and calibration settings:

File | Return to Top

Method | Print | Integration Events

Calibration Setting

6.1.3 Preparing New Calibration

Set up initial calibration table by identifying congener peaks from Mullin's chromatogram in the area percent report.

Reports | Prep Calib/Recalib

New Table | OK

Manually type in and enter the amount and name of each analyte with the retention times from the area percent printout.

Highlight "yes" in Reference ISTD peak boxes for Congener 30 and 204.

Ref.ISTD#1 will be used up to cong.110.
Ref.ISTD#2 will be used from cong.82.

* OK *

Do you want to delete all the peaks with the amount of zero?

* Yes *

Save to method?

* Yes *

6.1.4 Replacing Previous Calibration

For subsequent standard runs with analyte peaks all correctly identified in ISTD report the calibration table is replaced.

Reports | Prep Calib/Recalib

Recalibration | Replace | OK

Save to method?

* Yes *

Will get a printout of calibration table.

If the GC column has been clipped or running conditions have been changed, the retention times of analyte peaks shift and they are not identified in the ISTD report. A new calibration table will have to be created. This can be done either by following Section 6.1.3 or by inserting the new retention time in old calibration table and creating a temporary calibration table. Get ISTD report of the new standard with temporary calibration table and replace the calibration according to Section 6.1.4.

6.1.5 Saving Calibration on Data File

File | Command Line

Type in SAVETBL C:\HPCHEM\1\DATA\SUBDIRECTORY\0**R0101.CAL

6.1.6 Saving Event

File | Command Line

Type in: SAVE C:\HPCHEM\1\DATA\SUBDIR.\0**R0101.EVT Enter with keyboard.

6.2 Samples, Hexane Fraction

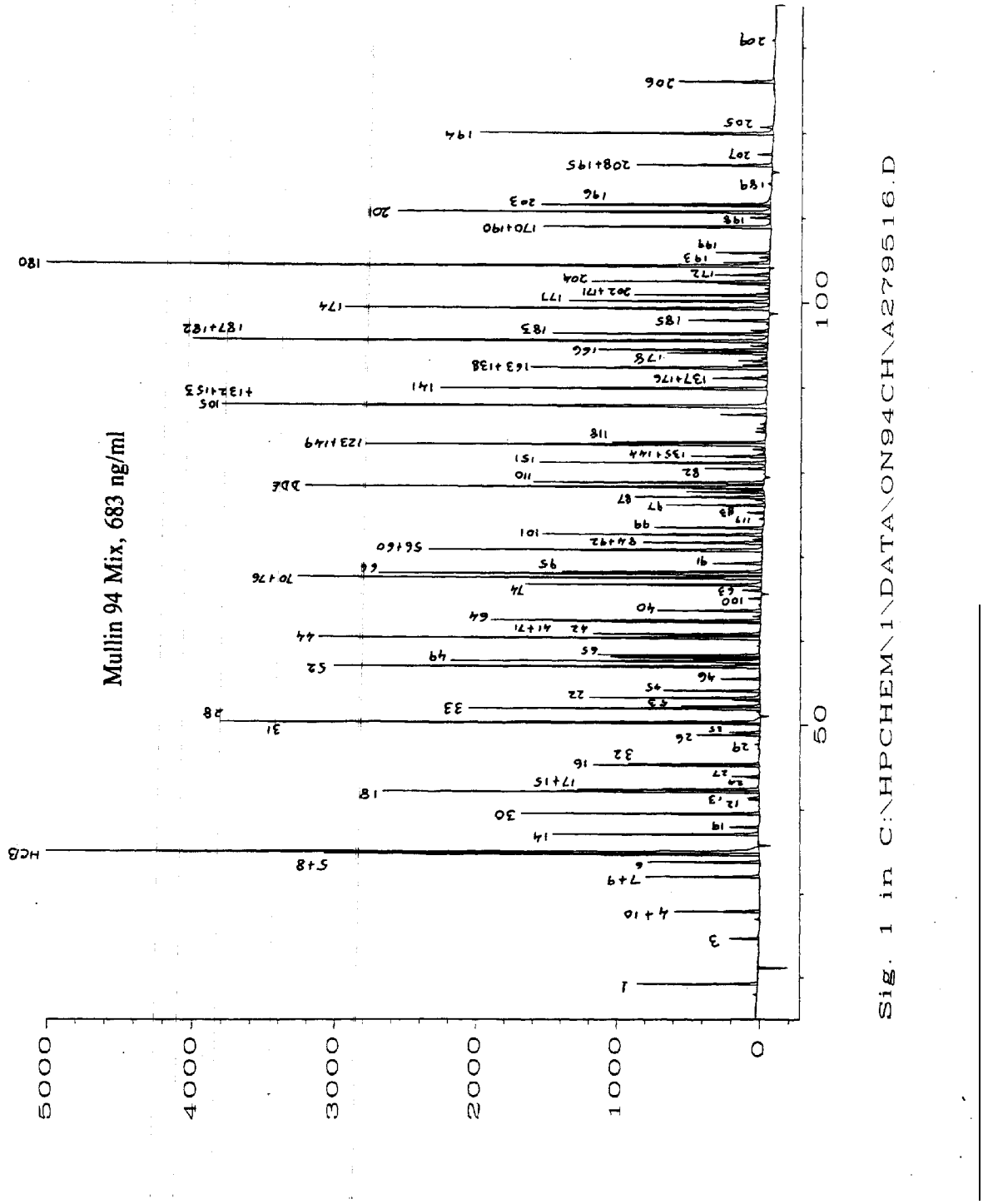
6.2.1 Integration - See previous Section 4.4

6.2.2 Printing Report and Saving Text File

See previous Section 5.1.2 with the following exceptions:
Calibration Settings Ref. and Nonref. Windows should be changed to 0.25%.

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.

6.2.3 Saving Events - See previous Section 5.1.6



Chromatogram 3. PCB Calibration Standard 950427

11 Jul 95 02:52 PM
Method: C:\HPCHEM\1\METHODS\IBPCBN.MTH

Calibration Table

Pk#	RT	Lvl	ng	Amt/Area	Ref Istd	I#	Name
1	19.187	1	48.0	1.0132e-002		1	1
2	24.672	1	28.0	2.2807e-002		1	3
3	27.943	1	13.6	3.6808e-003		1	4+10
4	32.039	1	4.8	9.3258e-004		1	7+9
5	33.775	1	7.6	1.6816e-003		1	6
6	34.701	1	56.0	3.0596e-003		1	5+8
7	34.958	1	20.0	3.6399e-004		1	HCB
8	37.077	1	20.0	2.2436e-003		1	14
9	37.944	1	1.12	9.3145e-004		1	19
10	39.526	1	8.0	8.4154e-004	Ref ISTD	1	30
11	41.222	1	0.68	1.734e-003		1	12
12	41.420	1	0.39	8.1106e-004		1	13
13	42.141	1	14.8	8.6819e-004		1	18
14	42.378	1	14.8	1.7505e-003		1	17+15
15	43.782	1	0.52	2.2385e-003		1	24
16	43.928	1	0.52	4.5844e-004		1	27
17	45.198	1	8.0	1.2027e-003		1	16
18	45.372	1	7.6	1.3979e-003		1	32
19	47.724	1	0.21	1.0343e-003		1	29
20	48.843	1	2.8	1.1252e-003		1	26
21	49.151	1	1.3	1.0363e-003		1	25
22	50.195	1	18.8	1.0569e-003		1	31
23	50.297	1	18.8	1.107e-003		1	28
24	51.959	1	13.2	1.0978e-003		1	33
25	52.245	1	2.6	7.7852e-004		1	53
26	53.008	1	0.72	7.3206e-004		1	51
27	53.246	1	11.6	1.682e-003		1	22
28	54.093	1	3.56	9.1343e-004		1	45
29	55.492	1	1.6	1.0223e-003		1	46
30	56.914	1	18.0	9.9944e-004		1	52
31	57.246	1	1.1	1.0759e-003		1	43
32	57.567	1	9.2	7.1717e-004		1	49
33	57.945	1	4.0	8.0619e-004		1	47
34	58.045	1	4.0	6.8621e-004		1	48
35	58.295	1	5.0	7.5344e-004		1	65
36	60.304	1	17.2	9.4466e-004		1	44
37	60.650	1	4.8	1.6582e-003		1	37
38	60.774	1	5.6	7.105e-004		1	42
39	62.234	1	9.2	1.1125e-003		1	41+71
40	62.395	1	7.2	6.3997e-004		1	64
41	63.646	1	3.76	9.4652e-004		1	40
42	65.123	1	0.44	8.0299e-004		1	100
43	66.067	1	0.84	1.127e-003		1	63
44	66.707	1	7.6	7.905e-004		1	74
45	67.504	1	13.6	7.1319e-004		1	70+76
46	67.988	1	20.8	1.3476e-003		1	66
47	68.205	1	8.0	9.4067e-004		1	95
48	69.243	1	2.04	9.5141e-004		1	91
49	70.795	1	14.0	9.7113e-004		1	56+60
50	71.665	1	7.2	1.4746e-003		1	84+92
51	72.076	1	0.4	8.7453e-004		1	89
52	72.627	1	7.2	7.9384e-004		1	101
53	73.429	1	2.96	6.6508e-004		1	99
54	74.518	1	0.112	2.6071e-004		1	119
55	75.234	1	0.6	6.7352e-004		1	83
56	76.129	1	2.24	5.4267e-004		1	97

Chart 5

11 Jul 95 02:52 PM
Method: C:\HPCHEM\1\METHODS\IBPCBN.MTH

57	76.768	1	0.64	1.1439e-003		1	81
58	77.055	1	4.0	7.372e-004		1	87
59	77.665	1	2.8	9.0102e-004		1	85
60	78.061	1	3.0	1.1743e-003		1	136
61	78.260	1	20.0	1.1061e-003		1	DDE
62	78.596	1	0.92	0.00125		1	77
63	78.788	1	7.6	7.6996e-004		1	110
64	80.467	1	1.8	7.0807e-004		2	82
65	81.135	1	6.8	7.4529e-004		2	151
66	81.903	1	3.56	1.0635e-003		2	135+144
67	82.187	1	0.22	5.1262e-004		2	124+147
68	82.730	1	0.52	7.6193e-004		2	107
69	83.249	1	11.2	6.8246e-004		2	123+149
70	83.524	1	4.8	6.9706e-004		2	118
71	84.783	1	0.29	3.3027e-004		2	134
72	85.727	1	0.56	1.5268e-003		2	114+131
73	86.796	1	1.56	9.0246e-004		2	146
74	87.813	1	17.2	6.8456e-004		2	105+132+153
75	89.856	1	6.8	5.0933e-004		2	141
76	91.096	1	1.04	4.7746e-004		2	137+176
77	91.398	1	0.3	1.3091e-003		2	130
78	92.301	1	10.8	9.6416e-004		2	163+138
79	92.663	1	1.0	9.9319e-004		2	158
80	93.503	1	0.052	2.8924e-004		2	129
81	94.033	1	4.4	1.163e-003		2	178
82	94.411	1	5.0	7.4955e-004		2	166
83	94.927	1	0.8	1.0108e-003		2	175
84	95.471	1	14.4	6.2431e-004		2	187+182
85	96.296	1	6.8	8.1456e-004		2	183
86	96.711	1	0.4	4.7325e-004		2	128
87	97.464	1	0.196	1.9914e-003		2	167
88	97.877	1	1.9	6.255e-004		2	185
89	99.284	1	12.8	7.7724e-004		2	174
90	100.118	1	6.8	8.8769e-004		2	177
91	100.850	1	3.16	6.067e-004		2	202+171
92	101.080	1	0.26	5.3533e-004		2	156
93	101.616	1	0.152	1.4242e-003		2	173
94	102.194	1	1.56	8.4592e-004		2	157+200
95	102.445	1	6.0	8.7429e-004	Ref ISTD	2	204
96	103.240	1	2.24	1.1557e-003		2	172
97	103.432	1	0.44	1.2268e-003		2	197
98	104.309	1	24.4	7.6752e-004		2	180
99	104.737	1	1.68	8.6603e-004		2	193
100	105.361	1	0.48	1.4826e-003		2	191
101	105.905	1	1.72	8.9833e-004		2	199
102	108.970	1	6.8	6.393e-004		2	170+190
103	110.083	1	0.48	5.7375e-004		2	198
104	110.714	1	16.8	1.1407e-003		2	201
105	111.510	1	8.6	9.3343e-004		2	203
106	111.732	1	8.6	1.5565e-003		2	196
107	114.055	1	0.16	1.5706e-003		2	189
108	116.223	1	3.2	6.1544e-004		2	208+195
109	117.547	1	0.37	6.9444e-004		2	207
110	120.001	1	7.2	6.306e-004		2	194
111	120.770	1	0.44	7.3461e-004		2	205
112	126.056	1	2.72	7.3361e-004		2	206
113	130.915	1	0.048	4.7233e-004		2	209

Chart 5 (Cont'd)

11 Jul 95 02:52 PM
Method: C:\HPCHEM\1\METHODS\IBPCBN.MTH

Calibration Settings

Title:

Reference window:	0.500 %
Non-reference window:	0.500 %
Units of amount:	ng
Multiplier:	1.0
RF uncal peaks:	0.0
Sample Amount:	0.0

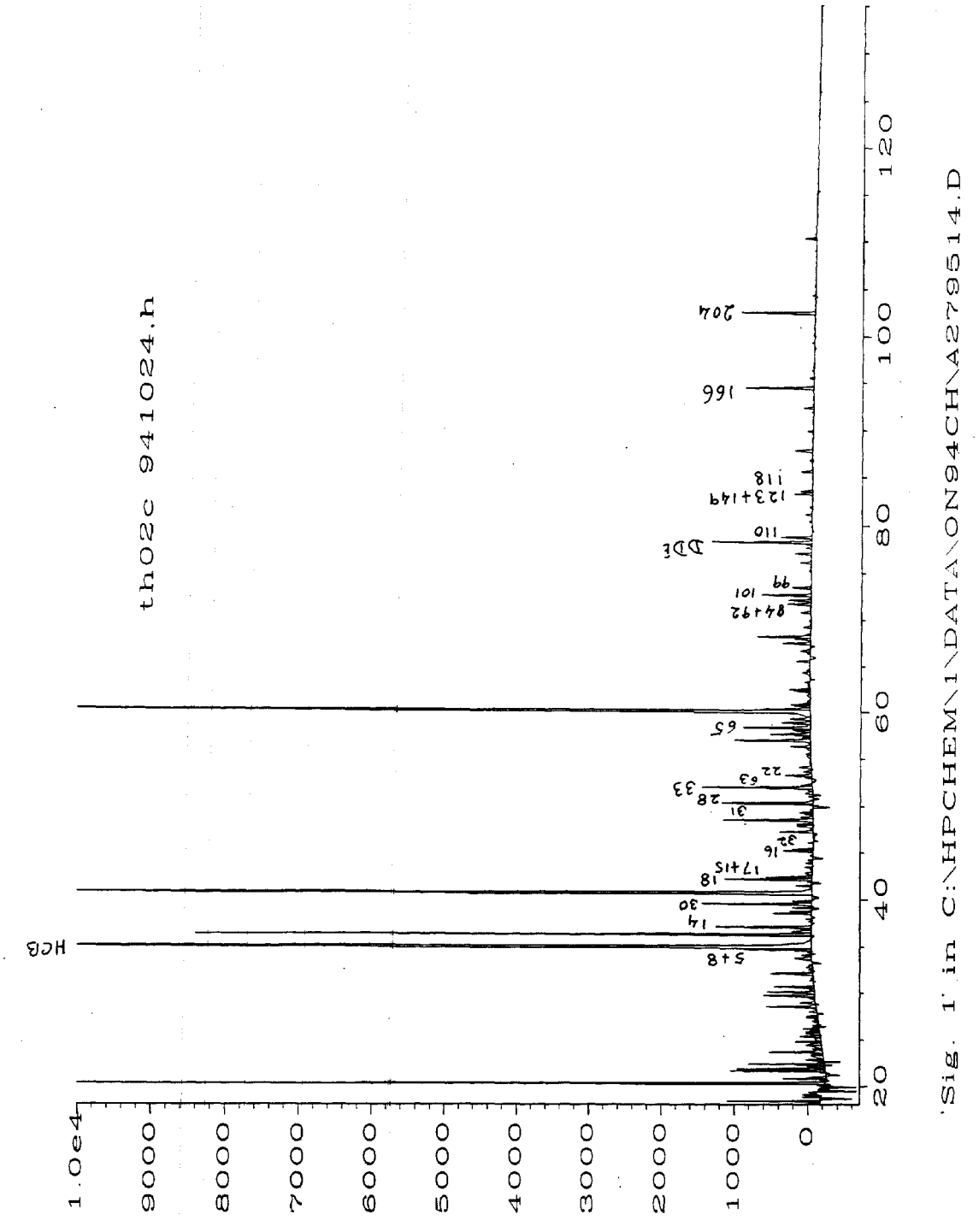
$ample ISTD Information

I#	Amount
1	8.0
2	6.0

Multilevel Information

Fit: Linear
Origin: Force

Chart 5 (Cont'd)



Chromatogram 4. PCBs in Vapor Sample

Internal Standard Report

Data File Name : C:\HPCHEM\1\DATA\ON94CH\A279514.D
Operator :
Instrument : HP5890A
Sample Name :
Run Time Bar Code:
Acquired on : APR 28, 1995 17:32:09
Report Created on: 22 Jun 95 01:39 PM
Last Recalib on : 21 JUN 95 11:36 AM
Multiplier : 1

Page Number : 1
Vial Number : 0
Injection Number :
Sequence Line :
Instrument Method:
Analysis Method : IBPCBN.MTH
Sample Amount : 0
ISTD Amount : 8

Sig. 1 in C:\HPCHEM\1\DATA\ON94CH\A279514.D

Ret Time	Area	Type	Width	Ref#	ng	Name
19.225	50	VBA	0.039	1	0.496	1
24.692	* not found *			1		3
27.971	868	PV	0.110	1	3.027	4+10
32.111	4297	PVA	0.110	1	3.839	7+9
33.752	2840	PP	0.168	1	4.459	6
34.697	6752	PV	0.113	1	19.228	5+8
34.958	235283	VV	0.116	1	80.586	HCB
37.071	9754	VP	0.116	1	20.720	14
37.950	398	PP	0.095	1	0.327	19
39.522	10418	PP	0.109	1-IR	8.000	30
41.195	250	VP	0.110	1	0.393	12
41.464	1313	PP	0.095	1	0.835	13
42.141	9515	VV	0.123	1	7.438	18
42.378	5080	VV	0.122	1	8.112	17+15
43.807	* not found *			1		24
43.922	662	PV	0.104	1	0.272	27
45.191	3020	PV	0.115	1	3.319	16
45.367	2080	VV	0.108	1	2.622	32
47.763	* not found *			1		29
48.833	1350	VV	0.113	1	1.390	26
49.142	890	PV	0.119	1	0.804	25
50.160	3441	PV +	0.000	1	3.219	31
50.304	11483	VP	0.137	1	11.717	28
51.964	12077	VV	0.125	1	11.951	33
52.238	1138	VP	0.113	1	0.779	53
53.020	710	PV	0.108	1	0.446	51
53.236	3123	VV	0.131	1	4.763	22
54.087	1466	PV	0.142	1	1.178	45
55.485	625	VV	0.138	1	0.555	46
56.910	9430	VV	0.137	1	8.396	52
57.204	908	VV	0.110	1	0.866	43
57.566	4527	VV	0.122	1	2.889	49
57.941	1458	VV	0.110	1	1.044	47
58.058	1937	VV	0.115	1	1.181	48
58.291	7669	VV	0.125	1	5.113	65
60.300	4996	VV	0.118	1	4.236	44
60.588	1785	VV +	0.000	1	2.658	37
60.756	2317	VVA	0.133	1	1.532	42
62.234	1399	PV	0.116	1	1.401	41+71
62.393	2446	VVA	0.128	1	1.405	64
63.560	306	PP	0.084	1	0.252	40
65.155	* not found *			1		100
65.997	1094	PP	0.178	1	1.692	63
66.702	1344	VV	0.135	1	0.977	74
67.507	3229	PVA	0.130	1	2.106	70+76

Chart 6

67.986		1823	VV	0.127	1	2.236	66
68.207		6262	VV	0.132	1	5.290	95
69.243		1042	PV	0.137	1	0.924	91
70.796		1228	PV	0.136	1	1.131	56+60
71.659		2814	VV	0.134	1	3.904	84+92
72.058		2434	VVA	0.122	1	2.361	89
72.621		5809	PV	0.028	1	4.210	101
73.438		2358	BV	0.138	1	1.450	99
74.559		74	BV	0.087	1	0.0427	119
75.229		205	PV	0.088	1	0.205	83
76.122		1239	PV	0.126	1	0.708	97
76.759		111	BV	0.097	1	0.191	81
77.050		1895	PV	0.129	1	1.371	87
77.661		730	BV	0.140	1	0.658	85
78.056		507	VV	0.095	1	0.572	136
78.257		10906	VV	0.125	1	11.329	DDE
78.605	* not found *				1		77
78.786		3549	VV	0.131	1	2.766	110
80.467		300	PB	0.116	2	0.224	82
81.131		679	PV	0.107	2	0.420	151
81.906		252	PV	0.080	2	0.219	135+144
82.216	* not found *				2		124+147
82.713		94	PV	0.088	2	0.0876	107
83.246		2093	PV	0.128	2	1.156	123+149
83.522		1475	VV	0.133	2	0.924	118
84.792		152	PV	0.111	2	0.0981	134
85.586		1290	BV	0.125	2	2.220	114+131
86.783		480	VV	0.150	2	0.333	146
87.809		2728	BB	0.163	2	1.495	105+132+153
89.850		723	BV	0.127	2	0.303	141
91.130	* not found *				2		137+176
91.387		109	BB	0.112	2	0.117	130
92.311		1195	BV	0.131	2	0.944	163+138
92.655		157	PB	0.103	2	0.117	158
93.488		72	PV	0.116	2	0.0122	129
94.054		111	BV	0.097	2	0.108	178
94.404		7584	PV	0.127	2	4.854	166
94.960	* not found *				2		175
95.468		562	BV	0.125	2	0.294	187+182
96.299		202	PV	0.128	2	0.141	183
96.698		170	PV	0.115	2	0.0841	128
97.488	* not found *				2		167
97.911	* not found *				2		185
99.281		305	PB	0.138	2	0.202	174
100.114		123	BV	0.094	2	0.0918	177
100.853		112	PV	0.104	2	0.0569	202+171
101.108	* not found *				2		156
101.644	* not found *				2		173
102.226	* not found *				2		157+200
102.442		8529	BV	0.133	2-IR	6.000	204
103.270	* not found *				2		172
103.458	* not found *				2		197
104.311		361	PB	0.132	2	0.242	180
104.764	* not found *				2		193
105.299		106	PB	0.112	2	0.0882	191
105.932	* not found *				2		199
108.986		225	PV	0.167	2	0.139	170+190
110.270		1318	PBA	0.132	2	0.924	198
110.709		105	PB	0.100	2	0.107	201
111.539	* not found *				2		203
111.712	* not found *				2		196
114.085	* not found *				2		189

Chart 6 (Cont'd)

```
116.256 * not found *              2              208+195
117.576 * not found *              2              207
120.031 * not found *              2              194
120.791 * not found *              2              205
125.982          306 BV    0.150   2      0.215   206
130.854           92 BV    0.091   2      0.0140  209

      Time Reference Peak      Expected RT     Actual RT      Difference
                10                39.556         39.522          -0.1%
                95               102.477        102.442          -0.0%

Not all calibrated peaks were found

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

Chart 6 (Cont'd)

```
22 Jun 95  01:41 PM
Method: C:\HPCHEM\1\METHODS\IBPCBN.MTH

                              Integration Events

         Events:              Value:   Time:
Initial Area Reject              50   INITIAL
Initial Peak Width            0.040   INITIAL
Shoulder Detection              OFF   INITIAL
Initial Threshold                -4   INITIAL
Negative Peak ON                       0.000
Baseline Now                          18.230
Baseline Now                          18.483
Baseline Now                          19.250
Baseline Now                          20.610
Baseline Now                          31.027
Negative Peak OFF                     31.886
Area Sum ON                           32.158
Area Sum OFF                          32.279
Negative Peak ON                      32.302
Baseline Now                          33.447
Baseline Now                          43.747
Baseline Now                          45.733
Negative Peak OFF                     50.081
Area Sum ON                           50.092
Area Sum OFF                          50.224
Negative Peak ON                      50.558
Negative Peak OFF                     60.026
Area Sum ON                           60.479
Area Sum OFF                          60.687
Area Sum ON                           60.885
Area Sum OFF                          61.037
Negative Peak ON                      61.386
Negative Peak OFF                     62.010
Area Sum ON                           62.492
Area Sum OFF                          62.643
Negative Peak ON                      62.803
Negative Peak OFF                     67.328
Area Sum ON                           67.663
Area Sum OFF                          67.850
Negative Peak ON                      68.652
Negative Peak OFF                     71.281
Area Sum ON                           71.318
Area Sum OFF                          71.523
Area Sum ON                           72.138
Area Sum OFF                          72.325
Negative Peak ON                      73.705
Baseline Now                          92.057
Baseline Now                          92.080
Baseline Now                         110.463
Baseline Now                         111.157

                          Calibration Settings

Title:
```

```
22 Jun 95  01:41 PM
Method: C:\HPCHEM\1\METHODS\IBPCBN.MTH


Reference window:                    0.500 %
Non-reference window:                0.500 %
Units of amount:                        ng
Multiplier:                            1.0
RF uncal peaks:                        0.0
Sample Amount:                         0.0

                          Sample ISTD Information

          I#        Amount
           1            8.0
           2            6.0

                           Multilevel Information

          Fit:     Linear
          Origin:  Force
```

Chart 7

Chart 7 (Cont'd)

7.0 Creating Excel File for PCB Analysis

7.1 Call *.txt files

7.2 Select "fixed width" and click on "next"

7.3 Click on line and "finish"

7.4 Delete a1..a5

7.5 Insert six rows

7.6 Delete Column C

7.7 Change column width b to 10

7.8 Edit and replace *not found* to 0

7.9 Copy 0's to C

7.10 Type at A1

- Subdirectory

- Sample name
- Vial id
- Date

7.11 Delete rows A124 to I131

7.12 Delete negative peaks amount

7.13 At 124 type "total pcbs"

7.14 At C124 put formula =((sum(c9..c121))-(c15+c16+c43+c69+c90)

7.15 At A126 type % recovery of 14 = c16/20*100

7.16 At A127 type % recovery of 65 = c43/5*100

7.17 At 128 type % recovery of 166 = c90/5*100

7.18 At 130 type ratio of 30:204 = B18/B103

7.19 At A132 type HCB = c15

7.20 At A133 type DDE = c69

Write comments

Save as .xls files

subdirectory		on94ch			
sample name		th02c941024,h			
vial id		14			
date		22-Jun-95			
ret time	Area	f# ng	Name		
19.225	50	0.496	1		
24.692	0	0	3		
27.971	868	3.027	4+10		
32.111	4297	3.839	7+9		
33.752	2840	4.459	6		
34.697	6752	19.228	5+8		
34.958	235283	80.586	HCB		
37.071	9754	20.72	14		
37.95	398	0.327	19		
39.522	10418	-IR 8.000	30		
41.195	250	0.393	12		
41.464	1313	0.835	13		
42.141	9515	7.438	18		
42.378	5080	8.112	17+15		
43.807	0	0	24		
43.922	662	0.272	27		
45.191	3020	3.319	16		
45.367	2080	2.622	32		
47.763	0	0	29		
48.833	1350	1.39	26		
49.142	890	0.804	25		
50.16	3441	3.219	31		
50.304	11483	11.717	28		
51.964	12077	11.951	33		
52.238	1138	0.779	53		
53.02	710	0.446	51		
53.236	3123	4.763	22		
54.087	1466	1.178	45		
55.485	625	0.555	46		
56.91	9430	8.396	52		
57.204	908	0.866	43		
57.566	4527	2.889	49		
57.941	1458	1.044	47		
58.058	1937	1.181	48		
58.291	7669	5.113	65		
60.3	4996	4.236	44		
60.588	1785	2.658	37		
60.756	2317	1.532	42		
62.234	1399	1.401	41+71		
62.393	2446	1.405	64		
63.56	0	0	40		
65.155	0	0	100		
65.997	0	0	63		

Chart 8

66.702	1344	0.977	74		
67.507	3229	2.106	70+76		
67.986	1823	2.236	66		
68.207	6262	5.29	95		
69.243	1042	0.924	91		
70.796	1228	1.131	56+60		
71.659	2814	3.904	84+92		
72.058	2434	2.361	89		
72.621	5809	4.21	101		
73.438	2358	1.45	99		
74.559	74	0.0427	119		
75.229	205	0.205	83		
76.122	1239	0.708	97		
76.759	111	0.191	81		
77.05	1895	1.371	87		
77.661	730	0.658	85		
78.056	507	0.572	136		
78.257	10906	11.329	DDE		
78.605	0	0	77		
78.786	3549	2.766	110		
80.467	300	0.224	82		
81.131	679	0.42	151		
81.906	252	0.219	135+144		
82.216	0	0	124+147		
82.713	94	0.0876	107		
83.246	2093	1.156	123+149		
83.522	1475	0.924	118		
84.792	152	0.0981	134		
85.586	1290	2.22	114+131		
86.783	480	0.333	146		
87.809	2728	1.495	105+132+153		
89.85	723	0.303	141		
91.13	0	0	137+176		
91.387	109	0.117	130		
92.311	1195	0.944	163+138		
92.655	157	0.117	158		
93.488	72	0.0122	129		
94.054	111	0.108	178		
94.404	7584	4.854	166		
94.96	0	0	175		
95.468	562	0.294	187+182		
96.299	202	0.141	183		
96.698	170	0.0841	128		
97.488	0	0	167		
97.911	0	0	185		
99.281	305	0.202	174		
100.114	123	0.0918	177		
100.853	112	0.0569	202+171		
101.108	0	0	156		
101.644	0	0	173		
102.226	0	0	157+200		

Chart 8 (Cont'd)

102.442	8529	IR 6	204		
103.27	0	0	172		
103.458	0	0	197		
104.311	361	0.242	180		
104.764	0	0	193		
105.299	106	0.0882	191		
105.932	0	0	199		
108.986	225	0.139	170+190		
110.27	1318	0.924	198		
110.709	105	0.107	201		
111.539	0	0	203		
111.712	0	0	196		
114.085	0	0	189		
116.256	0	0	208+195		
117.576	0	0	207		
120.031	0	0	194		
120.791	0	0	205		
125.982	0	0	206		
130.854	0	0	209		
total PCBs		159.0276			
%rec 14		103.6			
%rec 65		102.26			
%rec 166		97.08			
30:204		1.22148			
hcb		80.586			
dde		11.329			
comments		sample saver worked properly			

Chart 8 (Cont'd)

8.0 Data Storage and Data Retrieval

8.1 Copy the complete files (*.d, *.txt, *.evt,*.cal, and *.xls) on to the floppy disks. Leave one copy on hard disks too.

8.2 Loading Old Chromatogram

Same as Loading chromatogram in Section 4.3

8.3 Loading Old Calibration

Program Manager | File Manager

Click on
c:\hpchem\1\subdirectory\0*
*R0101.cal

File | Copy

To C:\hpchem\1\methods\appropriate method.cal

Replace Calib | Yes

Close File manager and Return to TOP
Click on Method and Load proper method.
Make sure that you got correct calibration table by clicking on

Report | Edit Calibration Table

8.4 Loading Old Integration Event

Program Manager | File Manager

Click on c:\hpchem\1\data\subdirectory\0**R0101.evt

File | Copy

8.5 Data Storage and Data Retrieval

To C:\hpchem\1\methods\appropriate method.evt

Replace Event | Yes

Close File manager and Return to TOP
Click on Method and Load proper method.
Make sure that you got correct integration event by clicking on

Integration | Event

Close File manager and Return to TOP
Click on Method and Load proper method.
Make sure that you got correct calibration table by clicking on

Report | Edit Calibration Table