BONNER ANALYTICAL TESTING COMPANY

DIOXATHION SPECIAL STUDY

FOR HERCULES, INC.

ORIGINAL STUDY

ORIGINAL MDL STUDY ON GC/FPD OF DIOXATHION SHOWED 2 SEPARATE PEAKS

USING THE OLD STANDARD & TEMPERATURES OF 140°C, 190°C, & 220°C TO DETERMINE THE CORRECT PEAK TO QUANTITATE

DIOXATHION ANALYTICAL METHOD
SW846 METHOD 8141

FOR

HERCULES, INCORPORATED
HATTIESBURG, MS

BY

BONNER ANALYTICAL TESTING COMPANY
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"Testing Your World for a Safer Tomorrow"

August 3, 1999

MS State Hand Chemistry Lab
112 Hand Lab
Morillo Road
MS State, MS 39762
Attn: Dr. Larry G. Lane
Director IAS Division

Dear Dr. Lane:

I am submitting some data that we have developed at our laboratory in Hattiesburg while attempting to run a dioxathion MDL study for Hercules, Incorporated. After you have had an opportunity to look at this data, please let me know what you think. The following is an overview of our findings:

1. We used Method 8141 with an FPD detector and an RTX-5 Megabore column. I believe that is the same column you are using. Our carrier gas flow rates probably are not exactly the same as yours so we will rely on RRTs to make comparisons.

2. What we have found is this. We are getting 2 peaks in the standard that you supplied to us.

3. The first peak (RRT = 0.670) appears to be the same as the peak that your lab found (see chromatogram supplied by your lab).

4. The second peak (RRT = 2.47) did not show up in your chromatogram, but did in our analysis. We found the second peak at a retention time at 32 minutes.

5. We have analyzed the standard a number of times and find that both peaks are reproducible.

6. We have submitted some mass spec data for both peaks. We did this on an HP quadrupole. The NBS Library is calling peak #2 dioxathion. Note that both the Library mass spectrum and the standard mass spectrum do not exhibit a molecular weight ion.

7. We also ran some experiments at injection port temperatures of 140°C, 190°C, and 220°C. What we see is that peak #1 is about 1.5% of peak #2 at 140°C, but at 220°C, peak #1 is about 15% of peak #2. It looks like peak #1 may be degradation product.

8. We have also analyzed a new sample from MW4. We ran at an injection port temperature of 140°C and 220°C. Interestingly, we found peak #1 but not peak #2 in the well sample. We also spiked a MW4 sample with standard dioxathion. We found peak #2 in the spiked sample

9. We have previously reported dioxathion, or an organo-phosphate compound that matches it, in MW4 at levels around 30 ppb. We have quantitated on peak #2 in our work.

Our next step is to perform an MDL study. At this point it appears that peak #2 is the appropriate peak to evaluate. Before we go forward with our study, I would like to know what your thoughts are. If you have any questions regarding the data I have sent, please give me a call.

Sincerely,

Michael S. Bonner, Ph.D.

cc: Tony Russell, MDEQ
Charlie Jordan, Hercules – Hattiesburg, MS
Frank Carlin, Consultant
Tim Hassett, Hercules

A REVIEW

of the

Dioxathion Analytical Method
SW846 Method 8141

utilized
in the

Analysis of Groundwater Samples

at

Hercules, Incorporated
Hattiesburg, MS

July 30, 1999

by

Michael S. Bonner, Ph.D.
Bonner Analytical Testing Company

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INTRODUCTION

During a routine investigation of monitoring wells at the Hercules, Incorporated Hattiesburg, MS facility, in 1998, Bonner Analytical Testing Co. tentatively identified dioxathion at trace levels utilizing SW846 Method 8270 (GC/MS). Estimated concentrations of 19.4, 37.3, and 29.5 ug/L of dioxathion were reported in Monitoring Well #4 (MW4) using this technique.

A split sample analyzed by the Mississippi State University Hand Chemistry Laboratory utilizing SW846 Method 8141 (GC/FPD) confirmed the presence of dioxathion, but the reported level was 805 ug/L in MW4 (see Appendix 1). After a meeting among MDEQ, MS State, and Hercules, it was agreed that both laboratories would utilize the SW846 Method 8141, a GC/FPD method for quantitation, and that GC/MS techniques would be used for confirmation. Additionally, prior to analysis, dioxathion standards would be provided by the MS State laboratory.

Bonner Analytical Testing Co. was retained by Hercules to perform the necessary method detection limit studies on both soil and water and to verify acceptable precision, accuracy and recoveries utilizing both Method 8141 (GC/FPD) and Method 8270 (GC/MS). Prior to performing the necessary MDL studies, Bonner Analytical Testing Co. consulted with MS State to obtain details relating to GC conditions used by MS State for the dioxathion analysis (via Method 8141). Generally, the MS State protocol mirrored that of the published method. GC conditions were as follows

- GC Detector: Flame Photometric (FPD) Phosphorus Mode
- Column: RTX-5 30M x 0.53mm cat# 10255 serial # 165689

- Carrier/flow rate*: He/9.27 ml/min
- Fuel/flow rate: H_2/111 ml/min
- Oxident/flow rate: Air/83.1 ml/min
- Injection port temperature: 220°C
- Detector temperature: 250°C
- Oven initial temperature/time: 165°C T1 = 5 min
- Program rate 1: 5°C/min to 215°C T2 = 10 min
- Program rate 2: 10°C/min to 250°C T3 = 31.50 min
- Extraction method: 3510C (water), 3540C (soil)
- Surrogate: Chlorpyrifos - Ultra Cat# DST4805 Lot# M1607
- Standard: MS State lab

*Carrier gas flow rate use by MS State was not provided. The flow rate recommended in Method 8141 was used for this study.

RESULTS

A review of chromatograms supplied by MS State (see Appendix 1) shows that the dioxathion standard contains a Peak #1 that elutes at 11.36 min. The surrogate marker Chlorpyrifos elutes at 15.66 min. The relative retention time (RRT) for Peak #1 was 0.725.

Bonner Analytical Testing Co. analyzed the dioxathion standard supplied by MS State (see Appendix 2). The standard was spiked with chlorpyrifos in order to confirm the relative retention time of the Peak #1 identified by MS State. Peak #1 was found to have an RRT of 0.670 using the same conditions reported by MS State (column flow rates were not available from MS State). Suffice it to say that both laboratories have identified an early eluting peak that has a RRT to chlorpyrifos of 0.670 – 0.725.

A review of the dioxathion standard chromatogram (see Appendix 2) by Bonner Analytical Testing Co. revealed the presence of a second peak with a RRT of 2.47 (absolute RT of 32.7 min). Repeated analysis confirmed that two peaks in addition to the surrogate were indeed present in the standard. Subsequently, the standard was analyzed by GC/MS (see Appendix 3), and the late eluting Peak #2 was found to be a library match for dioxathion. The early eluting Peak #1 did not give a library match for dioxathion.

Experiments in this laboratory have shown that there is a direct correlation between the early peak (Peak #1) intensity and the GC injection port temperature (see Table 1). In these experiments, 3 – 7 replicates of a 100ppm dioxathion standard were analyzed at injection port temperatures

ranging from 140°C to 220°C. Note that the early eluting peak increased in area by a factor of ten as the injection temperature was increased from 140°C to 220°C. In fact, at an injection port temperature of 140°C, the ratio of Peak #1 to Peak #2 is 1.5%, but at 220°C, the ratio is 14.8%. This data suggests that Peak #1 is a degradation product that can result from thermal decomposition of Peak #2 in the injection port.

Further study was conducted by evaluating samples collected from the Hercules MW4 (see Table 2). In this study, a method blank, a trip blank (BT55741), a field blank (BT55742), a clean water matrix spike, a matrix spike duplicate, MW4 water and MW4 water spiked at 10ppb with standard dioxathion were evaluated at injection port temperatures of 140°C and 220°C

These data demonstrate the following:

1. Excellent recoveries of the surrogate chlorpyrifos (ranging between 68% and 90%)

2. The method blank, trip blank, and field blank produced no interfering peak at either injection port temperature.

3. The matrix spike and matrix spike duplicate spiked at 10 ppb produced no early peak at 140°C, but gave an 85% and 91% recovery of the second peak When analyzed at 220°C, both Peak #1 and Peak #2 were present and recoveries ranged from 85% to 99% for Peak #2

4. Sample BT55743 from MW4 produced a chromatogram with a significant early peak, but the second peak was absent. The chromatograms were the same for both injection port temperatures. This data indicates that Peak #1 is present in the MW4 and is not the result of a hot injection port.

5. Interestingly, the concentration of Peak #1, found in the most recent MW4 sample (collected July 22, 1999), was found to be 751 ug/L, which is in good agreement with Peak #1 levels reported by MS State.

6. BT55743 was also spiked with dioxathion prior to extraction. The recovery of Peak #2 was found to range from 87% to 91.1%. This data indicates that if Peak #2 were present in the Hercules MW4, it would be detected by the method.

SUMMARY

A dioxathion standard supplied by MS State was found to contain two constituents when analyzed by GC/FPDs in accordance with SW846 Method 8141. One of those peaks eluted well before the surrogate marking compound, chlorpyrifos (Peak #1 RRT 0.670). This peak was quantitated as dioxathion by MS State. A second peak (Peak #2) that eluted at a RRT of 2.47 (absolute RT 32.6 min) was the major peak present in the standard detected by Bonner Analytical Testing Co. It appears that early work by MS State did not detect the major peak because their analytical run was terminated before this peak eluted. The available data suggests that Peak #1 is a degradation product that can be formed in a hot injection port. Secondly, GC/MS analysis of this standard under the same chromatography conditions shows that Peak #2 (RT ~ 32 min) is an excellent library match for dioxathion. Additionally, Peak #1 does not produce a library match for dioxathion. Additional experiments in this laboratory suggest that Peak #1 is present in MW4. While Peak #2 was detected by Bonner Analytical Testing Co. in MW4 on three previous occasions in 1998, this peak was not detected in MW4 samples collected on July 22, 1999.

TABLES

TABLE 1

DIOXATHION STANDARD PEAK AREA AS A FUNCTION OF INJECTION PORT TEMPERATURE USING A 100PPM STANDARD

	8:86 min Peak Area Injtemp (140°)	8:86 min Peak Area State (190°)	8:86 min Peak Area StateB (220°)	32:75 min Peak Area Injtemp (140°)	32:75 min Peak Area State (190°)	32:75 min Peak Area StateB (220°)
Run						
A	3,661,503	26,835,902	38,271,410	211,997,570	223,039,399	251,926,013
B	3,309,698	26,943,578	36,997,275	220,528,545	223,581,528	248,093,036
C	3,596,355	24,704,408	35,980,523	224,749,442	227,063,957	252,268,801
D	3,400,280	25,851,068		229,224,594	228,092,692	
E	3,179,532	24,262,766		231,234,855	230,778,415	
F	3,472,164	25,228,732		233,495,837	226,470,178	
G	3,519,926	24,982,250		235,144,114	232,932,804	
AVERAGE	3,448,494	25,544,101	37,083,069	226,624,994	228,745,461	250,762,617
STDDEV	166747.44953	1039515.296	1147850.766	8189816.301	4575538.254	2318269.097

Chlorpyrifos Surrogate Peak Areas

	Chlorpyrifos Peak Area Injtemp (140°)	Chlorpyrifos Peak Area State (190°)	Chlorpyrifos Peak Area StateB (220°)
	4,328,251	4,748,962	5,005,377
	4,455,198	4,662,562	5,221,548
	4,524,343	4,667,440	5,181,867
	4,457,611	4,921,065	
	4,517,534	4,927,239	
	4,633,435	4,740,706	
	4,258,214	4,773,053	
AVERAGE	4,452,512	4,777,290	5,136,264
STDDEV	127236.6969	108431.4734	115074.7606

TABLE 2

HERCULES MW#4 WATER ANALYSIS AT INJECTION PORT TEMPERATURES OF 140° AND 220°

							Dioxathion							
SAMPLE	Recovery 140°	Recovery 220°	Chlorpyrifos Area 140°	PPB 140°	Chlorpyrifos Area 220°	PPB 220°	8.71 Peak Area 140°	PPB 140°	8:71 Peak Area 220°	PPB 220°	32.66 Peak Area 140°	PPB 140°	32.66 Peak Area 220°	PPB 220°
Method Blank	76.22	74.15	[illegible]	[illegible]	3,780,227	741	0	0	0	0	0	0	0	0
Matrix Spike	81.95	87.23	[illegible]	[illegible]	4,447,060	872	0	0	290,229	8.51	2509675	8.50	2,334,860	9.16
Matrix Spike Dup.	80.56	81.16	[illegible]	[illegible]	4,137,747	812	0	0	287,473	8.43	2679758	9.07	2,530,033	9.92
Trip Blank	85.04	89.87	[illegible]	[illegible]	4, 582,008	899	0	0	0	0	0	0	0	0
Equipment Blank	82.27	86.04	[illegible]	[illegible]	4,386,597	860	0	0	0	0	0	0	0	0
BT55743	68.71	71.38	[illegible]	[illegible]	3,639,118	714	[illegible]	[illegible]	25,612,299	751	0	0	0	0
BT55743Spike	75.91	77.62	[illegible]	[illegible]	3,957,297	776	[illegible]	[illegible]	29,148,836	855	[illegible]	[illegible]	2,322,462	9.11

TABLE 3

A Comparison of Dioxathion Standard Curve Peak Areas at Injecton port Temperatures of 140°C and 220°C

CONCENTRATION OF DIOXATHION (PPB)	AREA Peak #1 8:17 @ 220°	AREA Peak #2 32:67 @ 220°	AREA Peak #1 8:17 @ 140°	AREA Peak #2 32:67 @ 140°
10	0	0	0	0
100	48,744	211,555	0	317,593
500	172,273	1,342,216	0	1,525,991
1000	325,255	2,818,252	0	3,022,982
10000	3,354,621	30,357,694	271,286	35,727,623
100000	28,060,253	260,319,076	1,823,342	287,369,745
LINEARITY	0 999816732	0 999859964	0 99854932	0 999699157

APPENDIX 1

1A – Chromatogram Supplied by MS State
Run #1171 – 3.22 ng of Dioxathion
RT = 11.36 min

1B – Chromatogram Supplied by MS State
Run #1169 – 0.432 ng Chlorpyrifos
RT = 15.66 min
RRT = 0.723

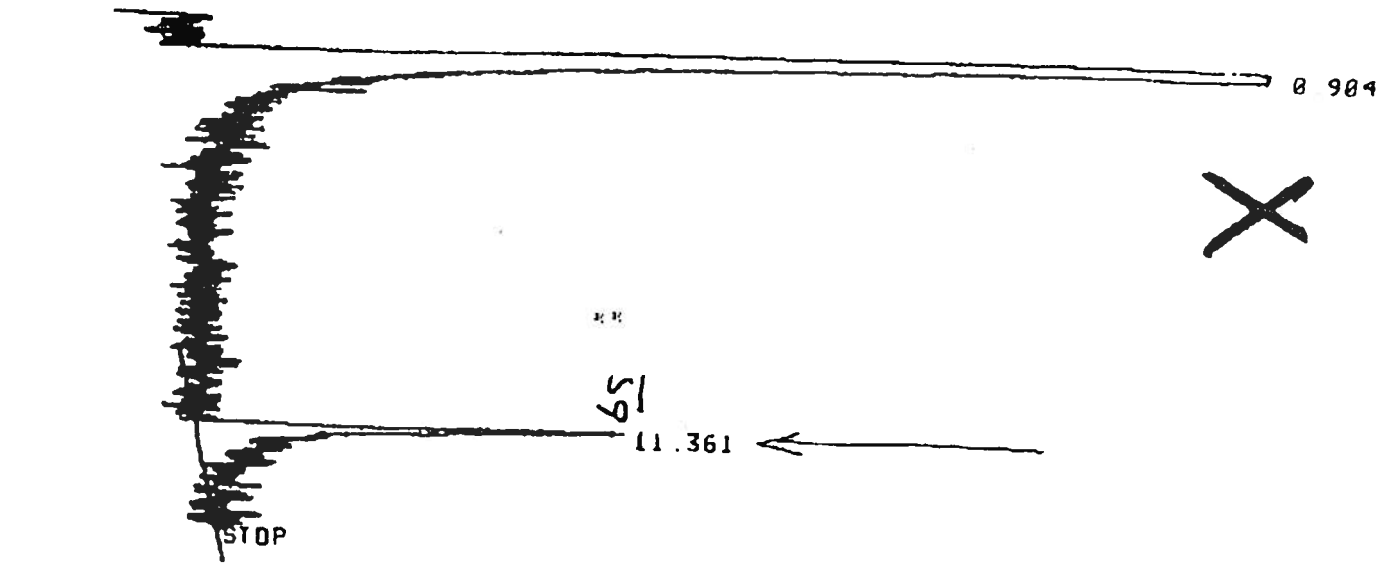
1C – Chromatogram Supplied by MS State
Run #1180
Hercules MW4
Dioxathion = 805ppb

MUL FACTOR=1.0000E+00

Miss State Chromatogram #1
Dioxathion AD.

*NOTEPAD

(USE BREAK OR CONTROL-Y TO END)
3.22 NG DIOXATHION
* RUN # 1171 SEP 30, 1998 04:10:37
START



RUN# 1171 SEP 30, 1998 04:10:37

AREAX

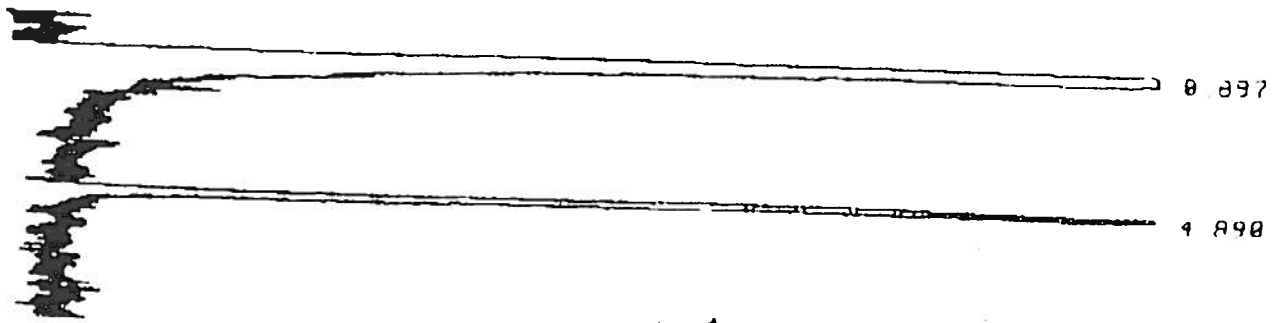
RT	AREA	TYPE	WIDTH	AREAX
.904	1496460	PB	.119	96.03987
11.361	61705	BV	.164	3.96011

TOTAL AREA=1558165
MUL FACTOR=1.0000E+00

Varian 3600-FPD/P
DB-5 Megabore

*NOTEPAD

(USE BREAK OR CONTROL-Y TO END)
100 NG SP WATER
* RUN # 1172 SEP 30, 1998 04:31:14
START



Appendix I-A

THRSH = 1
PK WD = 0.04

Miss State Chromatogram Chlorpyrifos #2 FPD/P

*NOTEPAD

(USE BREAK OR CONTROL-Y TO END)
0.432 NG CHLORPYRIFOS
* RUN # 1169 SEP 30, 1998 03:33:40
START

0.898

15.657

STOP

RUN# 1169 SEP 30, 1998 03:33:40

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.898	1589077	PB	.125	96.52694
15.657	57175	PU	.149	3.47304

TOTAL AREA=1646252
MUL FACTOR=1.0000E+00

Varian 3600-FPD/P
DW-V Workstation

*NOTEPAD

(USE BREAK OR CONTROL-Y TO END)
0.393 NG CHLORMEPHOS
* RUN # 1170 SEP 30, 1998 04:00:03
START

0.905

0.982

STOP

Appendix I-B

TOTAL AREA=2585925
MUL FACTOR=1.0000E+00

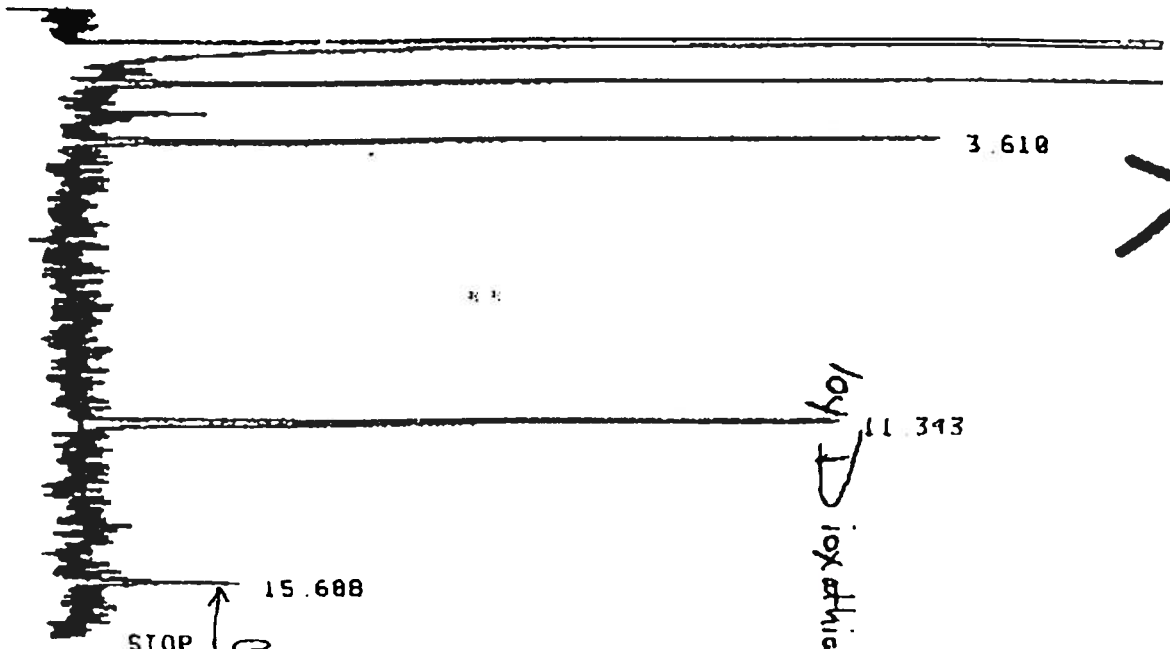
Miss State Chromatogram #3

*NOTEPAD

(USE BREAK OR CONTROL-Y TO END) Groundwater from Hercules MW-7.7 MG 10896

* ~~RUN # 1180~~ OCT 1, 1998 01:18:12

START



Dioxathion

Chlorpyrifos

RUN# 1180 OCT 1, 1998 01:18:12

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.934	710178	PB	.073	77.16717
2.001	56676	PV	.055	6.15835
3.610	59877	VP	.077	6.50617
11.343	73219	PV	.108	7.95590
15.688	20361	VP	.130	2.21240

TOTAL AREA= 920311
MUL FACTOR=1.0000E+00

Varian 2600 - FPD/P
DB-5 Megabore

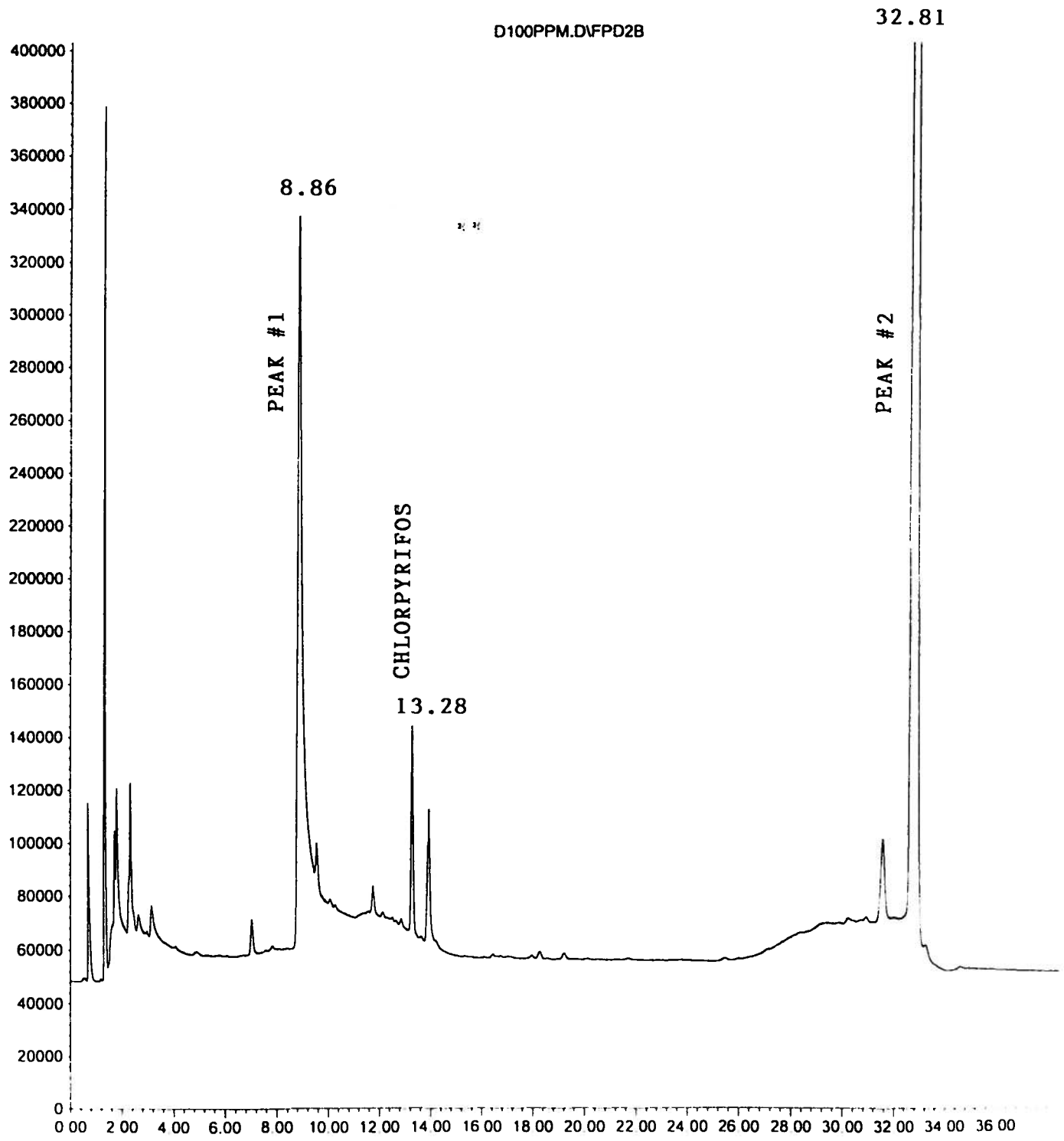
Appendix 1c

APPENDIX 2

2A – Chromatogram of Dioxathion Standard
Supplied by MS State
Peak #1 – RRT = 0.667
Peak #2 – RRT = 2.471

BONNER CHROMATOGRAM
MS STATE STANDARD
DIOXATHION @ 100ppm
GC/FPD

File : C:\HPCHEM\1\DATA\0699\062899\D100PPM.D
Operator : jps
Acquired : 30 Jun 99 12:16 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion @ 100ppm
Misc Info : injection 220
Vial Number: 17



APPENDIX 2A

APPENDIX 3

3A – GC/MS Chromatogram of Dioxathion Standard

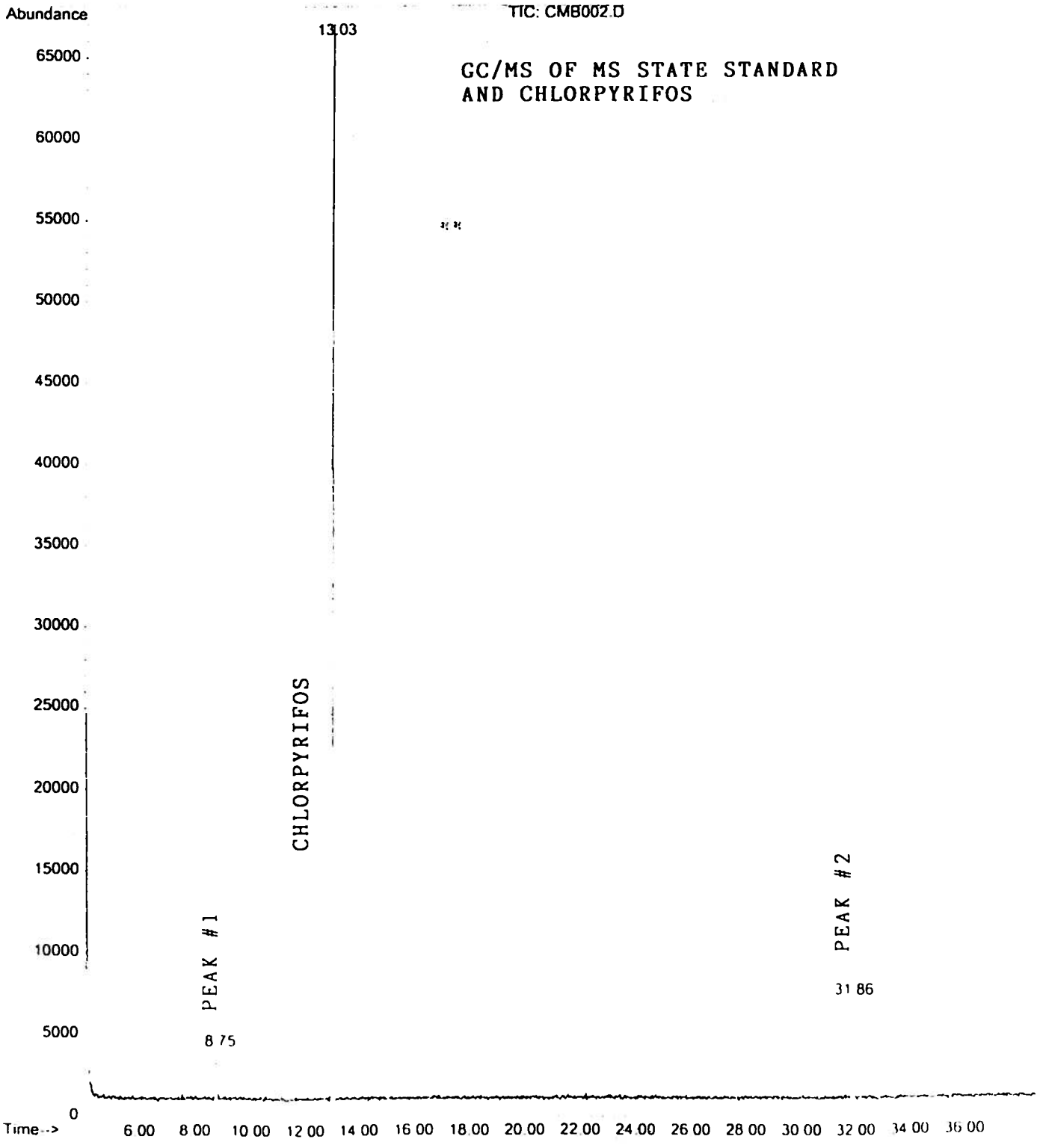
3B – Mass Spectrum Profile of Dioxathion from NBS Library

3C – Mass Spectrum Profile of Peak #2
RRT = 2.445 & RT = 31.9 min
NBS Library Match

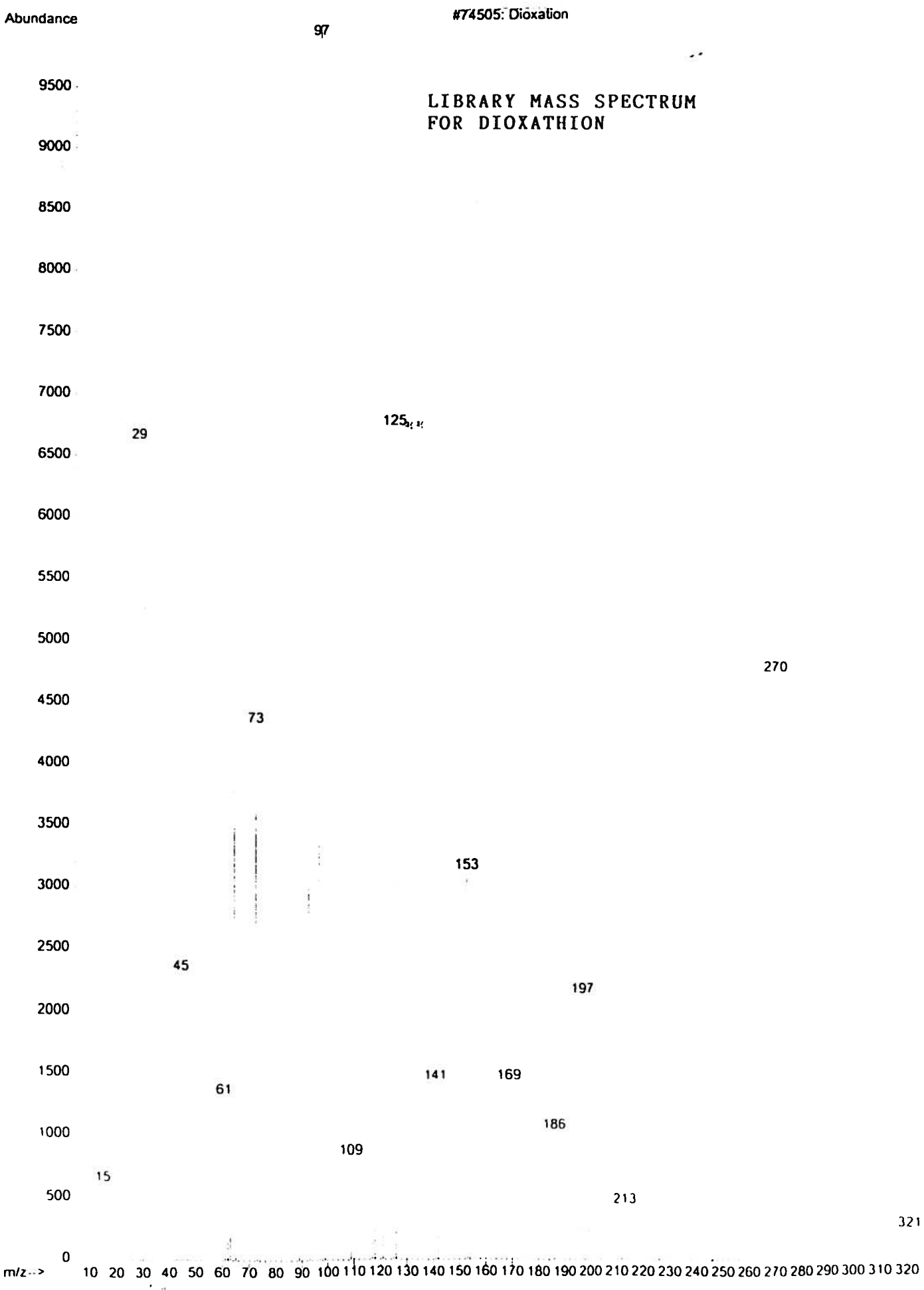
3D – Mass Spectrum Profile of Peak #1
RRT = 0.671 & RT = 8.75 min
Does Not Match NBS Library

File : C:\HPCHEM\1\DATA\MISC\CMB002.D
Operator : JMR
Acquired : 30 Jun 99 11:09 am using AcqMethod CHRIS
Instrument : GC/MS Ins
Sample Name: Dioxathion/Chlorpyrifos
Misc Info : Det. 200/ Inj 220
Vial Number: 1

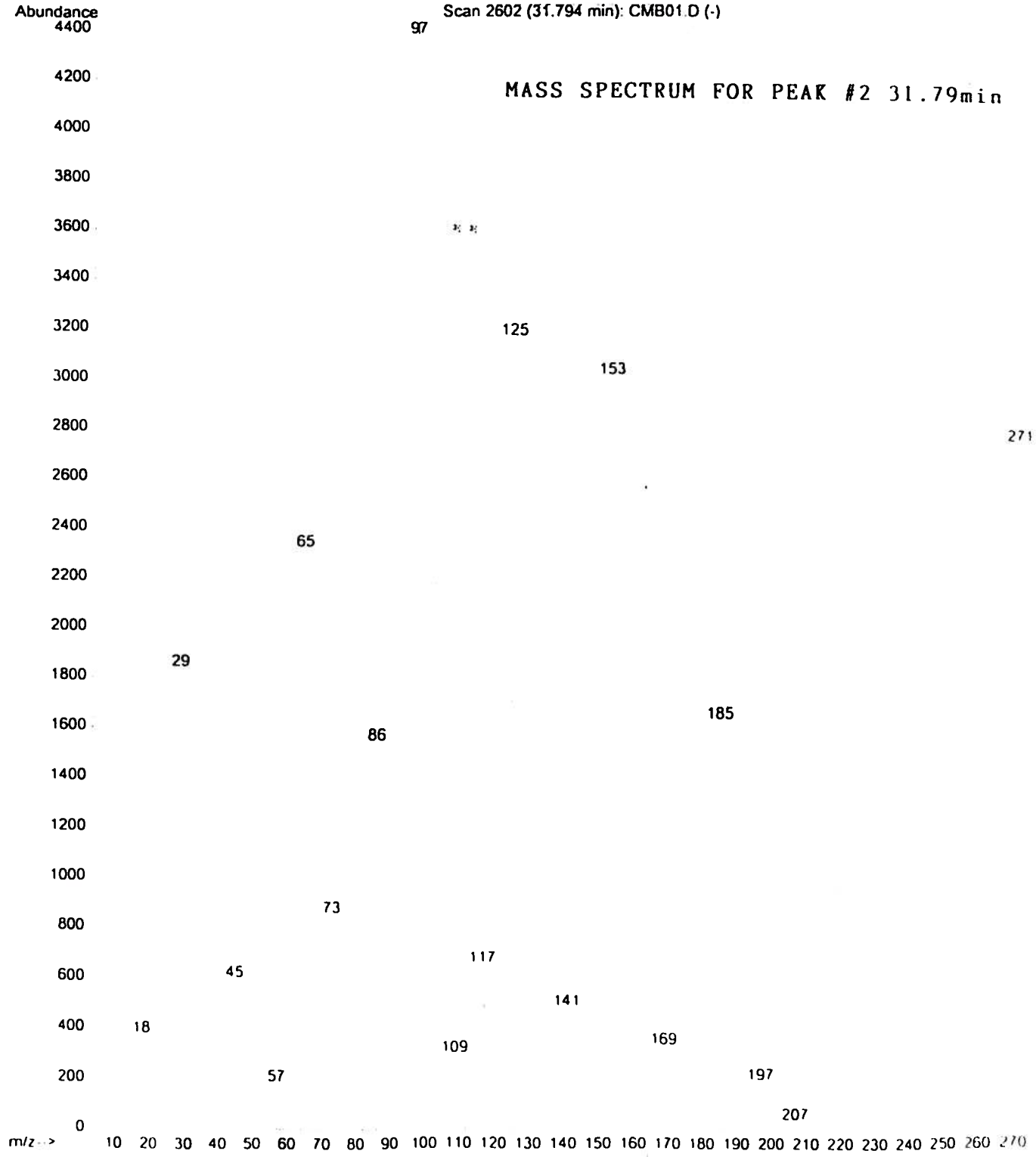
NOTE THIS CHROMATOGRAM IS FROM THE GC/MS



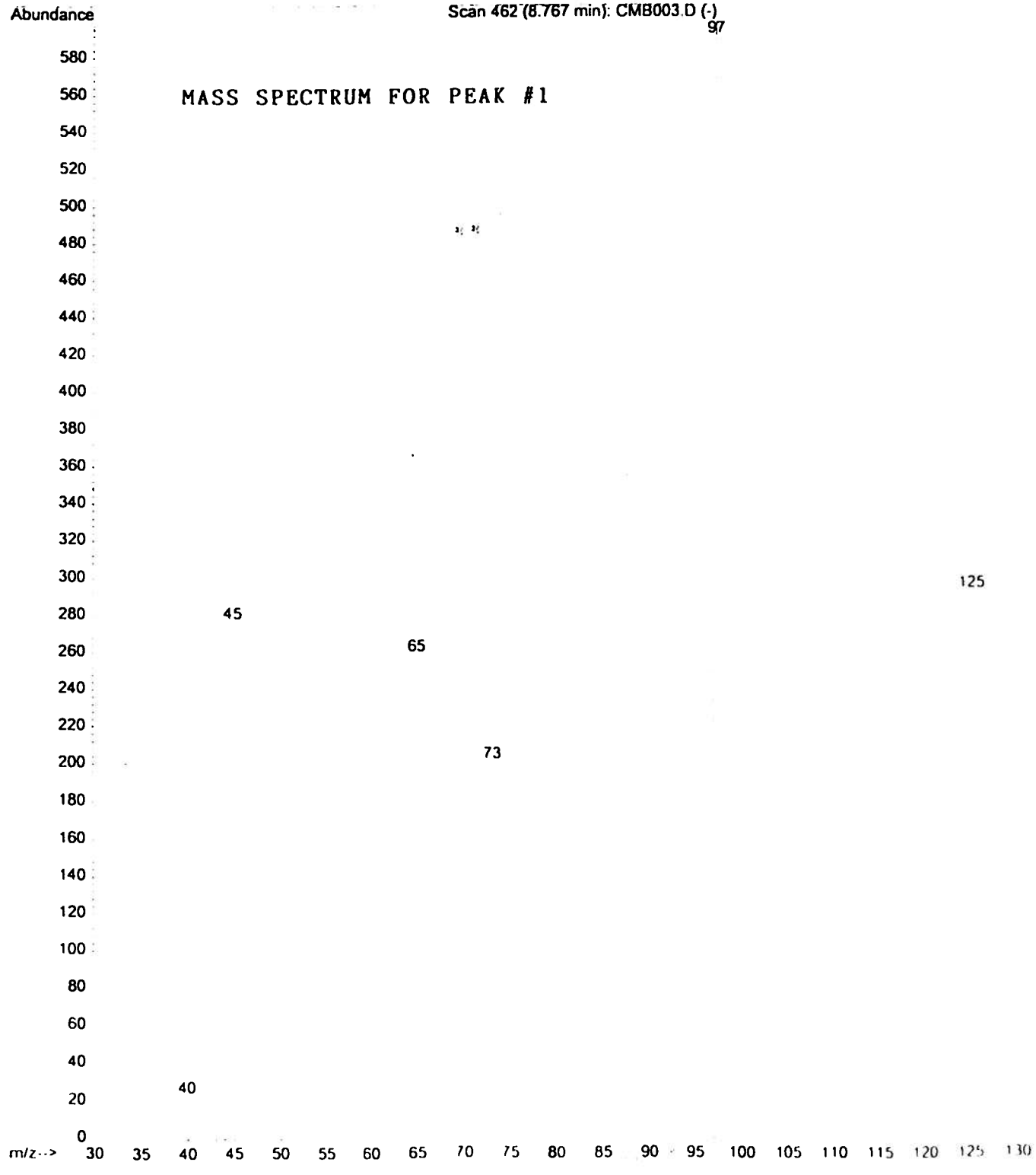
Library : C:\DATABASE\NBS75K.L



File : C:\HPCHEM\1\DATA\MISC\CMB01.D
Operator : cmb
Acquired : 22 Jun 99 11:50 am using AcqMethod CHRIS
Instrument : GC/MS Ins
Sample Name: dioxathion 100ppm
Misc Info : Injector 220 Detector 280
Vial Number: 1



File : C:\HPCHEM\1\DATA\MISC\CMB003.D
Operator : cmb
Acquired : 30 Jun 99 11:57 am using AcqMethod CHRIS
Instrument : GC/MS Ins
Sample Name: Dioxathion(100) no Chloropyrfos
Misc Info : Det. 200/ Inj 220
Vial Number: 1



APPENDIX 4

Raw Data

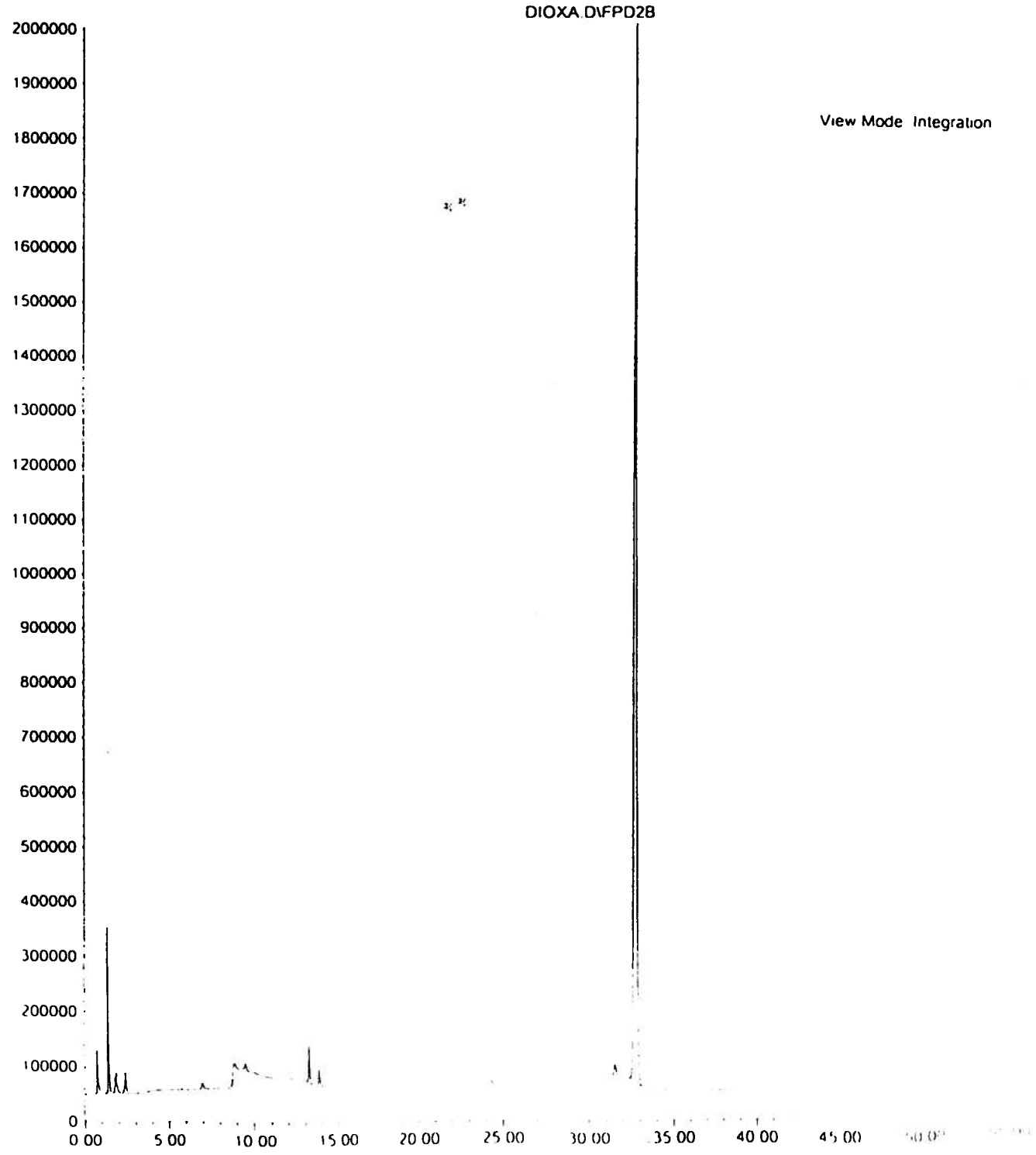
4A – Dioxathion Standard Analyzed at Injection Port of 140°C

4B – Dioxathion Standard Analyzed at Injection Port of 190°C

4C – Dioxathion Standard Analyzed at Injection Port of 220°C

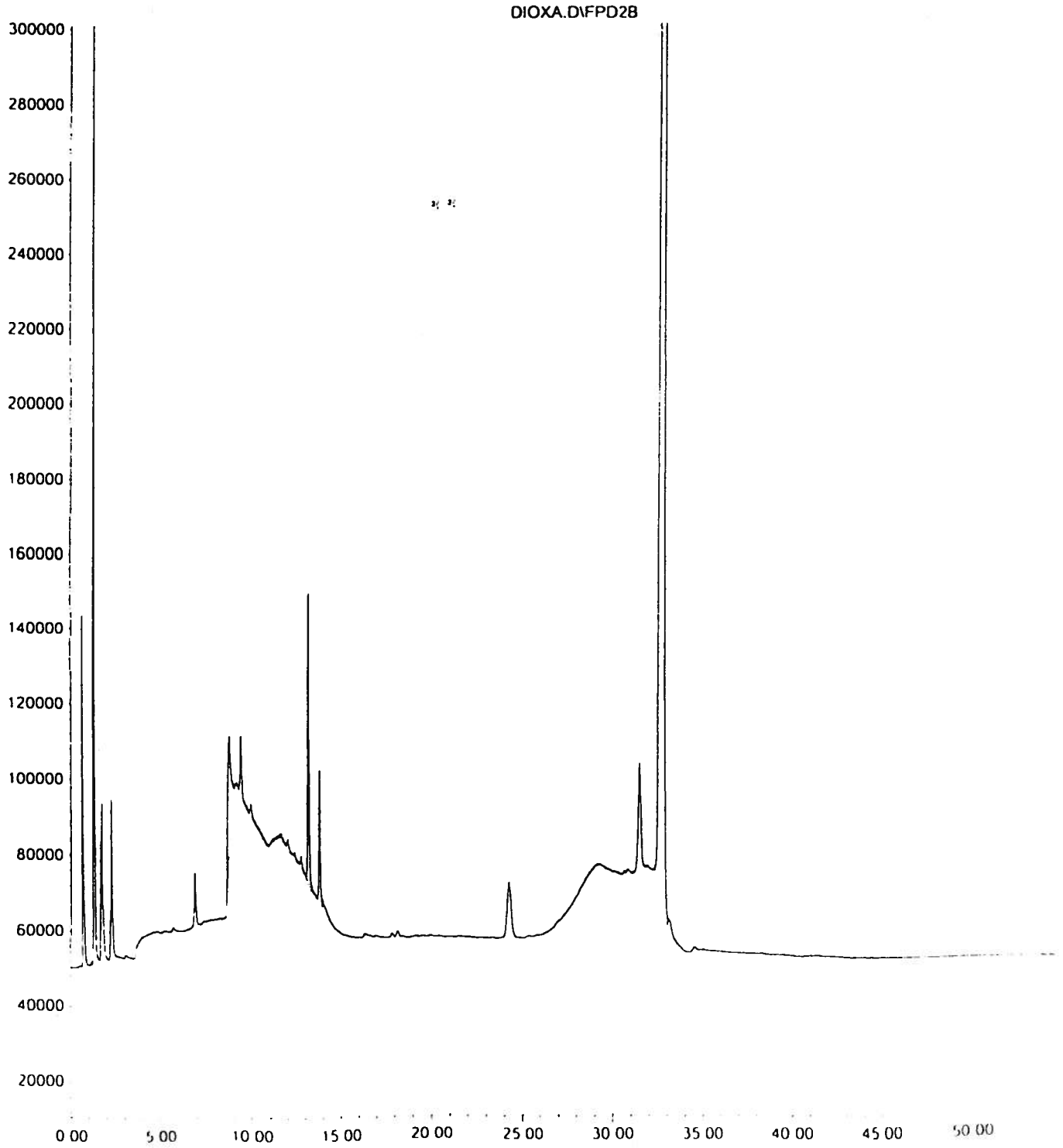
4A – Dioxathion Standard Analyzed at Injection Port of 140°C

File : C:\HPCHEM\1\DATA\072199\DIOXA.D
Operator :
Acquired : 21 Jul 99 15:12 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3

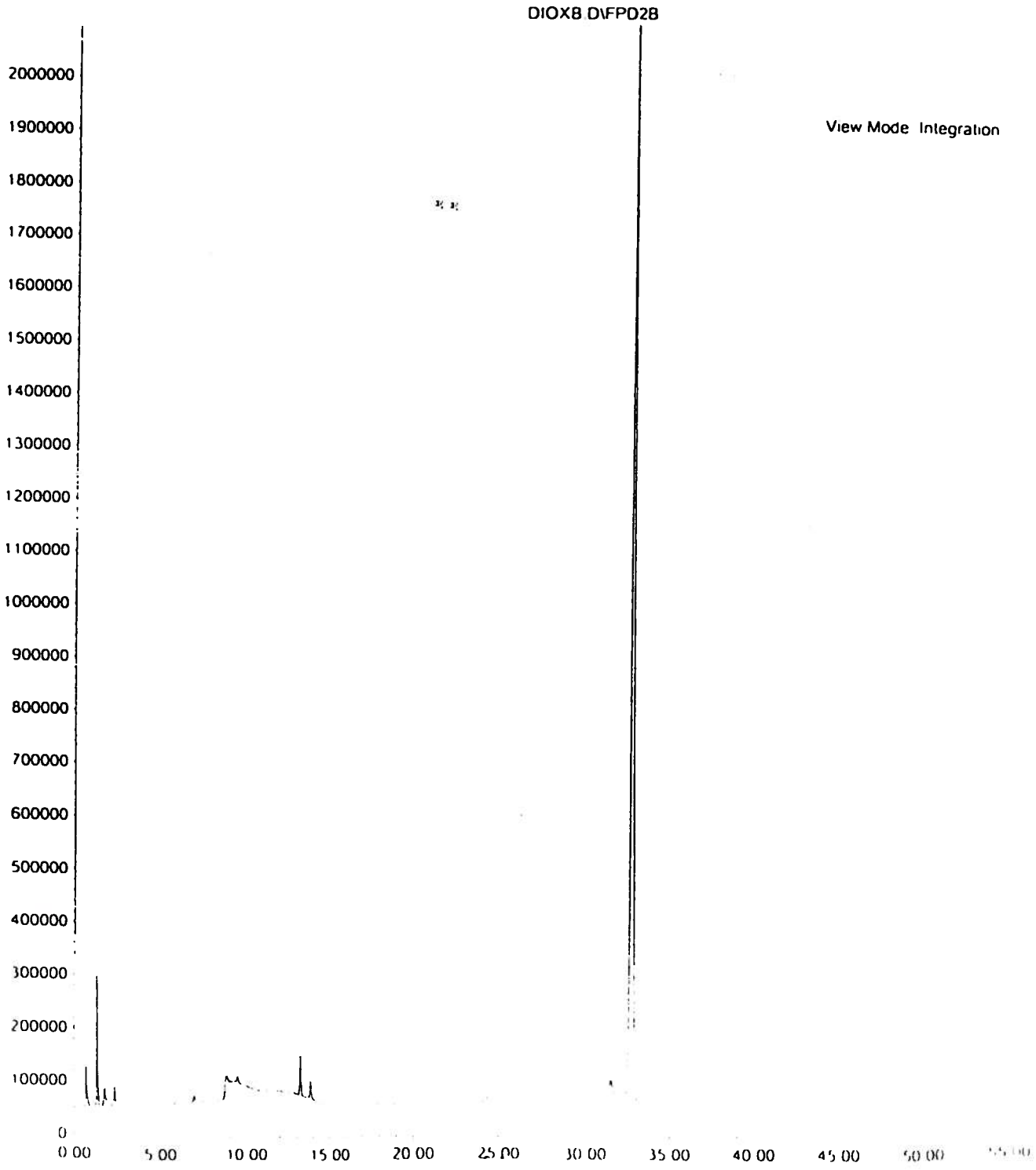


Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.860	M	0.201	3661503	8.655	9.059
2	13.227	M	0.093	4321251	13.098	13.384
3	32.747	M	0.182	211997570	32.434	33.082

File : C:\HPCHEM\1\DATA\072199\DIOXA.D
Operator :
Acquired : 21 Jul 99 15:12 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3

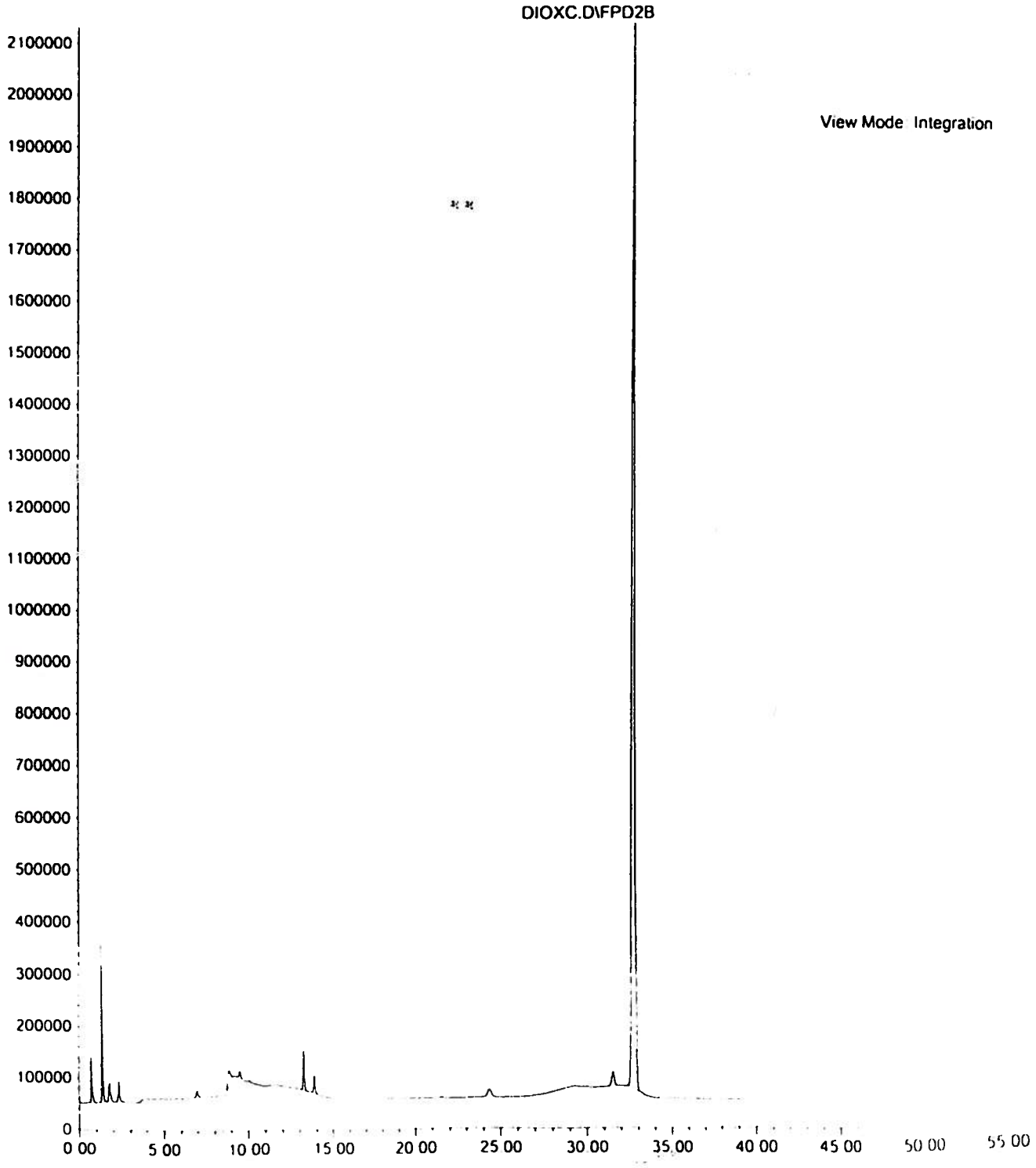


File : C:\HPCHEM\1\DATA\072199\DIOXB.D
Operator :
Acquired : 21 Jul 99 16:34 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



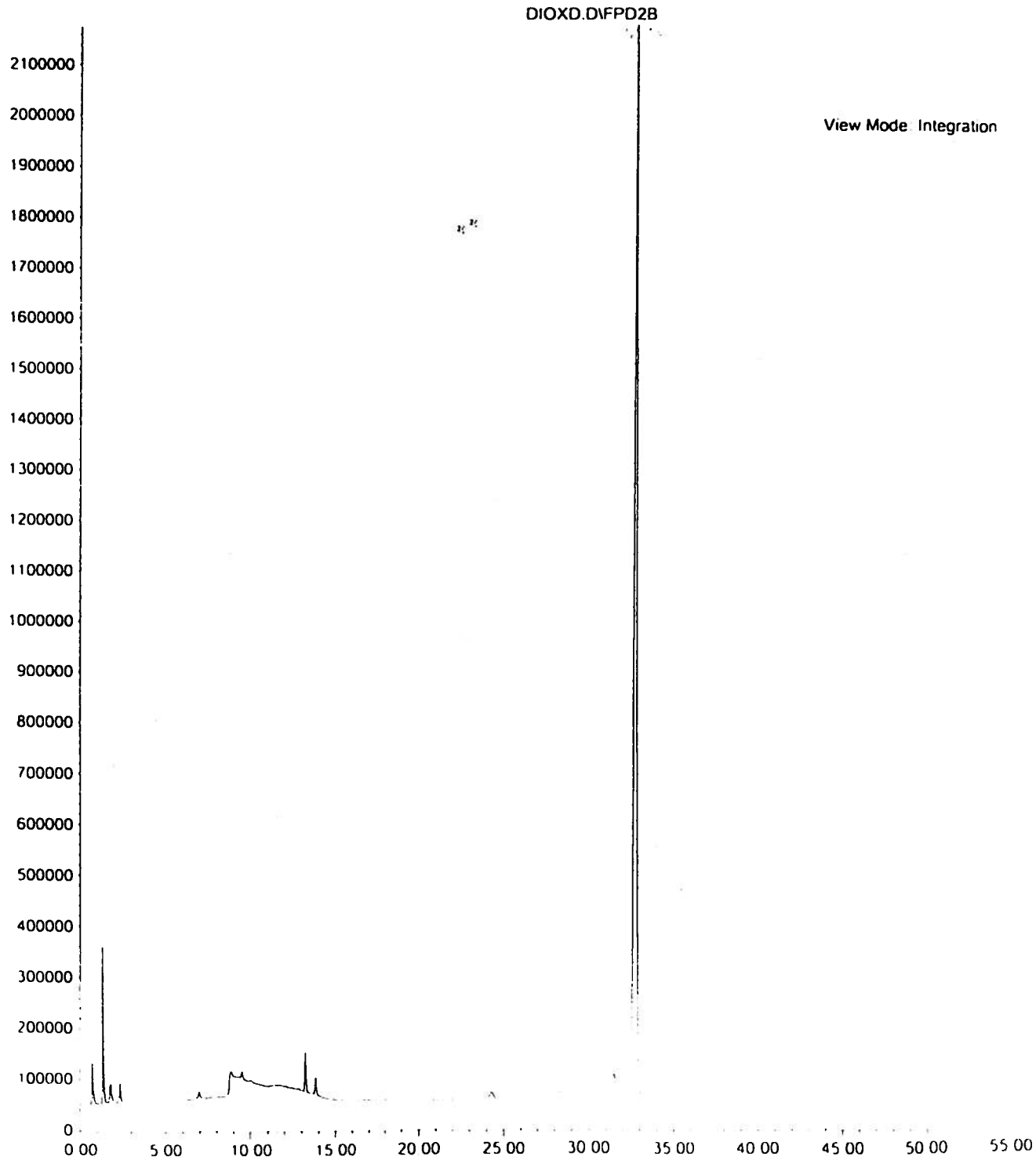
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.856	M	0.193	3309698	8.662	9.006
2	13.225	M	0.094	4455198	13.074	13.383
3	32.735	M	0.181	220528545	32.417	33.056

File : C:\HPCHEM\1\DATA\072199\DIOXC.D
Operator :
Acquired : 21 Jul 99 17:57 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



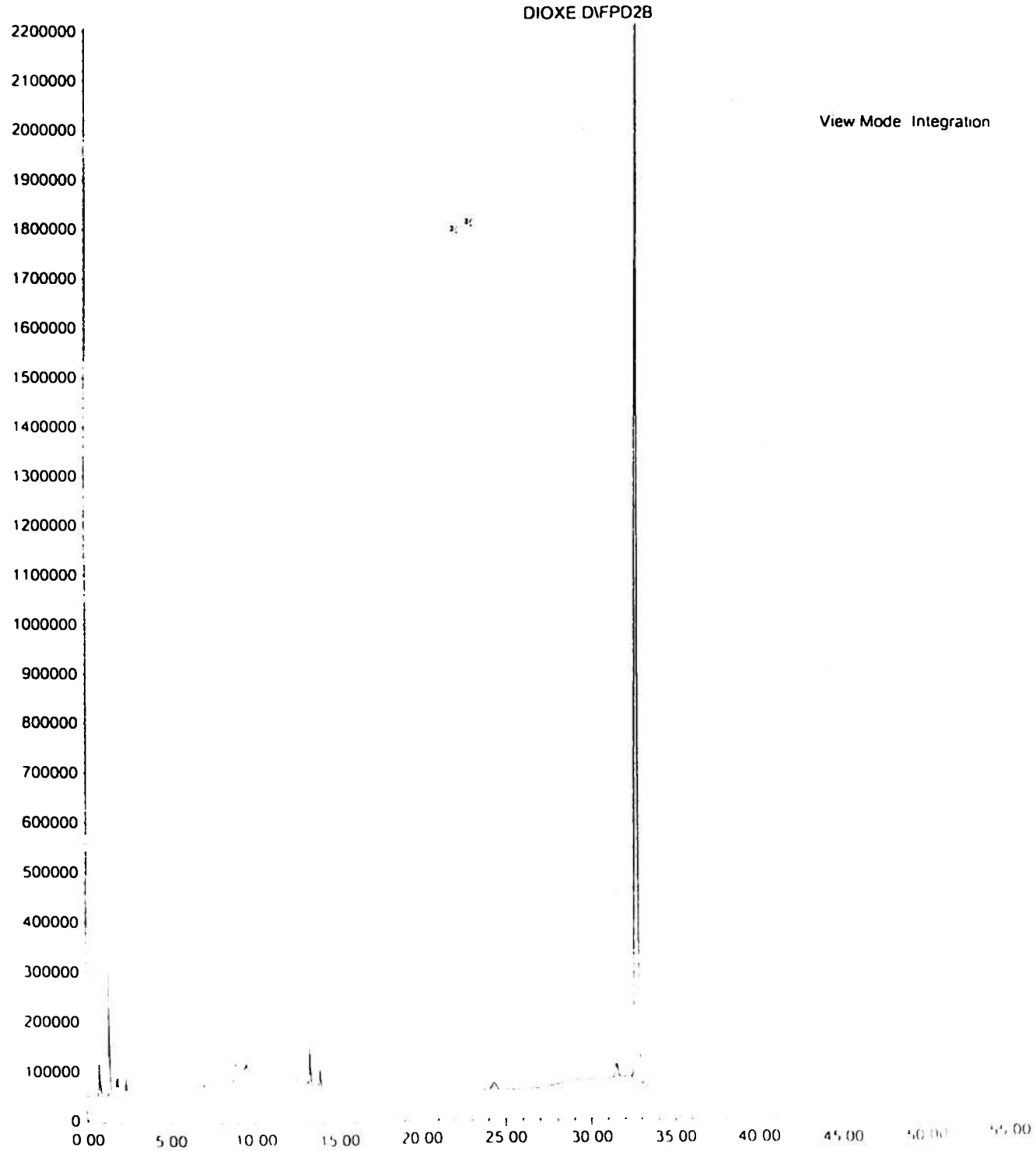
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.866	M	0.204	3596355	8.639	9.043
2	13.221	M	0.094	4524343	13.093	13.385
3	32.730	M	0.182	224749442	32.369	33.065

File : C:\HPCHEM\1\DATA\072199\DIOXD.D
Operator :
Acquired : 21 Jul 99 19:20 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



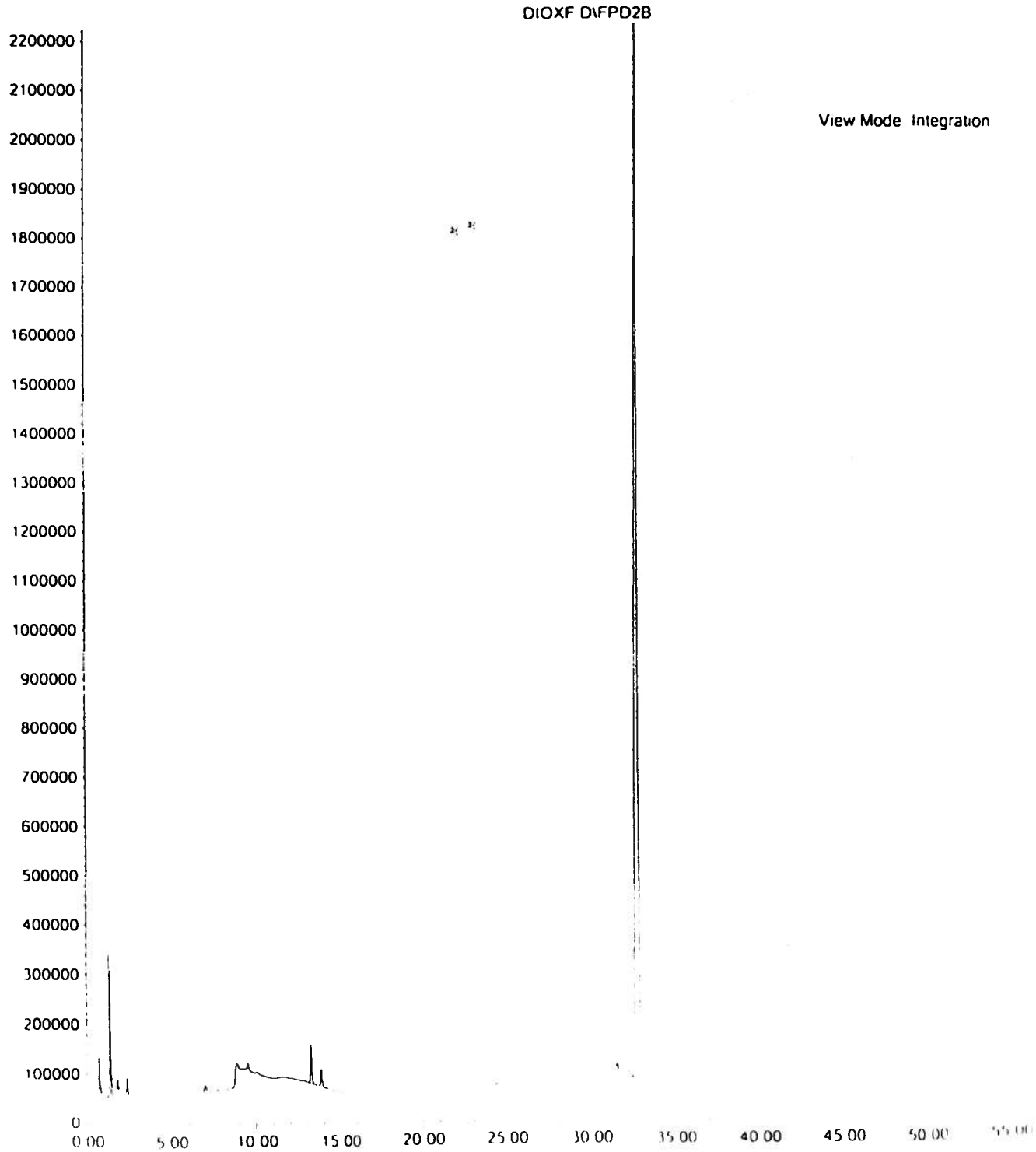
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.876	M	0.207	3400280	8.644	9.029
2	13.226	M	0.092	4457611	13.072	13.361
3	32.729	M	0.182	229224594	32.383	33.083

File : C:\HPCHEM\1\DATA\072199\DIOXE.D
Operator :
Acquired : 21 Jul 99 20:43 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



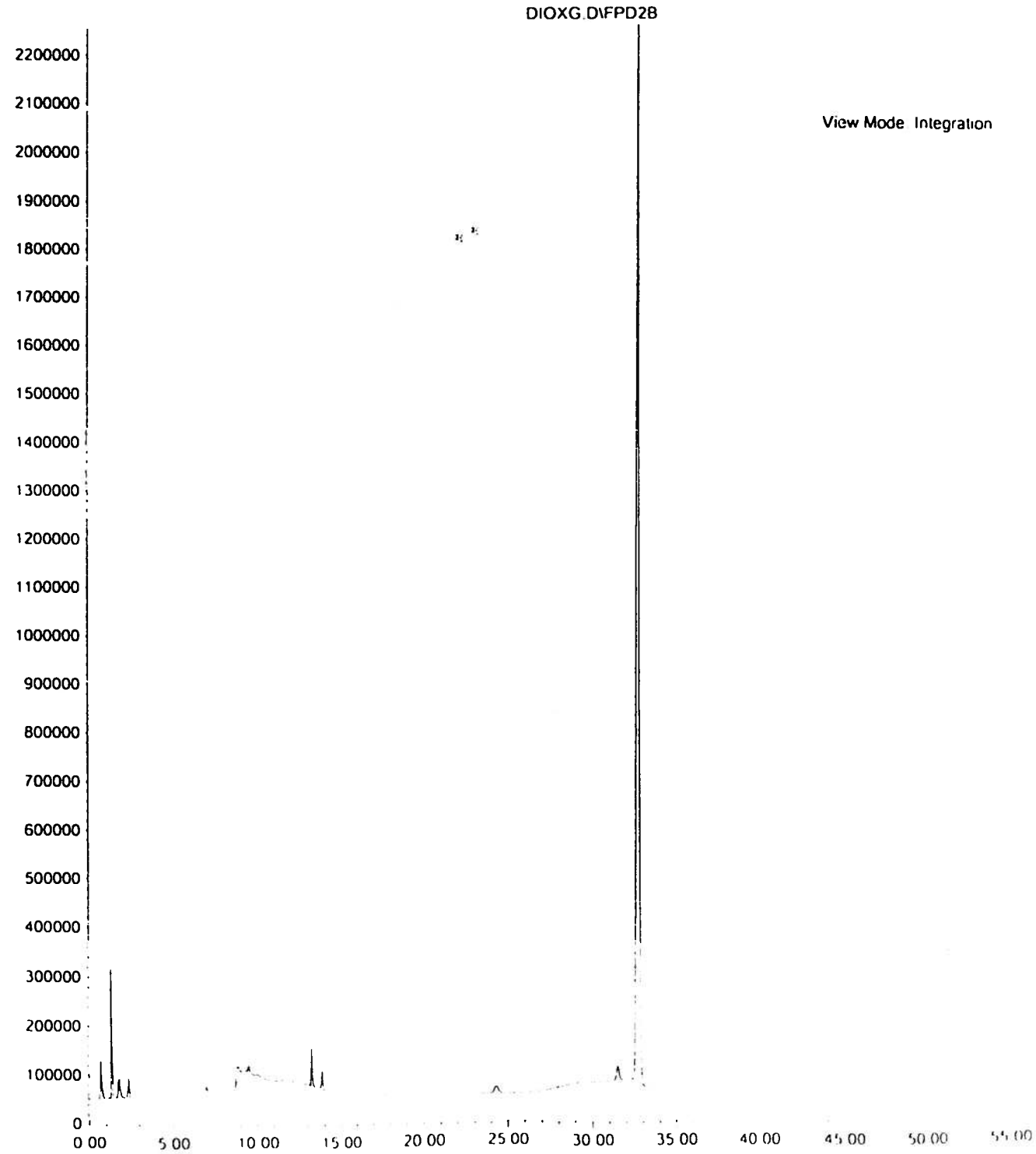
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.853	M	0.184	3179532	8.641	9.010
2	13.228	M	0.091	4517534	13.111	13.365
3	32.722	M	0.181	231234855	32.382	33.065

File : C:\HPCHEM\1\DATA\072199\DIOXF.D
Operator :
Acquired : 21 Jul 99 22:06 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.859	M	0.187	3472164	8.654	9.006
2	13.230	M	0.093	4633435	13.097	13.386
3	32.719	M	0.181	233495837	32.360	33.071

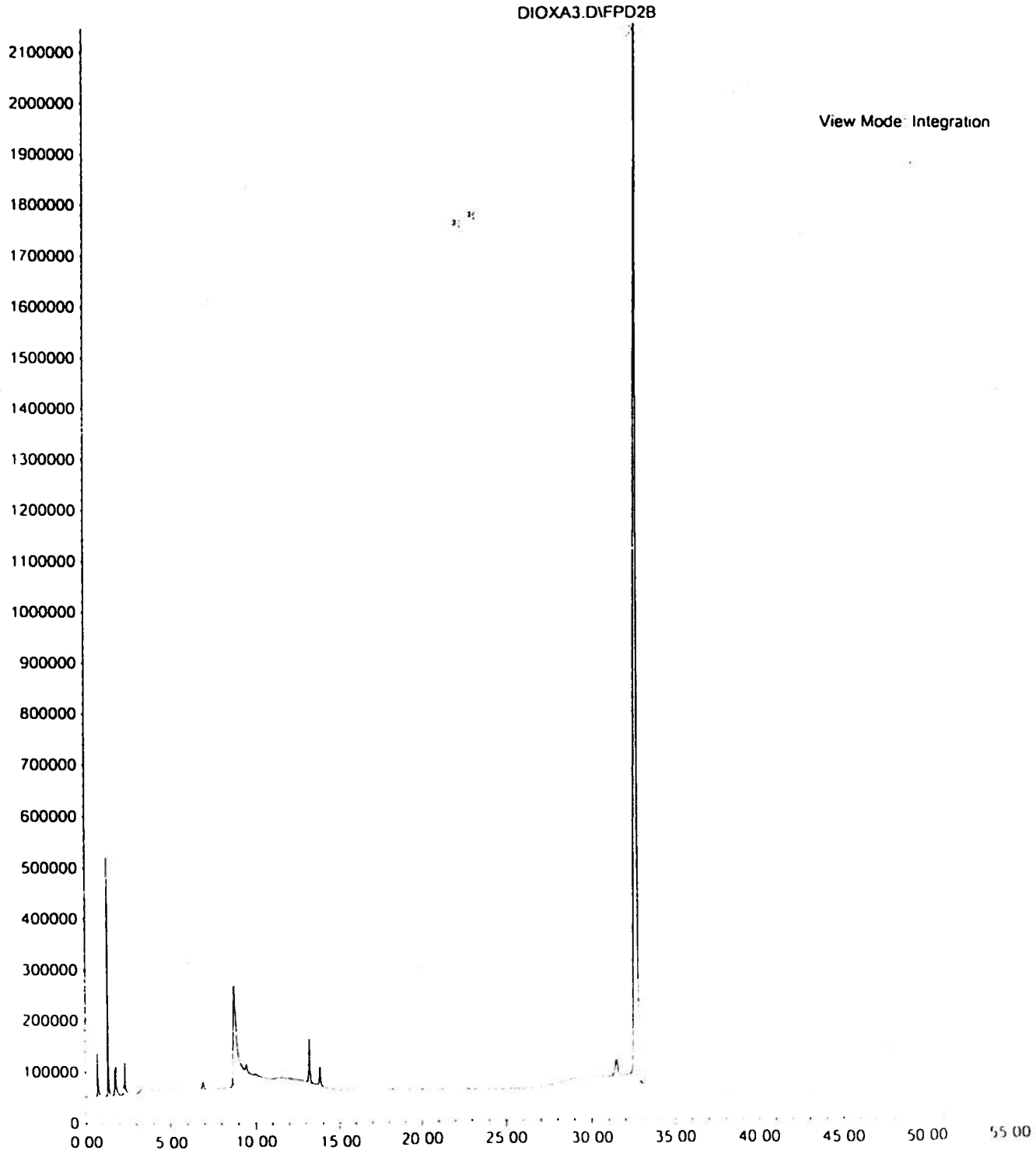
File : C:\HPCHEM\1\DATA\072199\DIOXG.D
Operator :
Acquired : 21 Jul 99 23:29 using AcqMethod INJTEMP.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 140 degrees
Vial Number: 3



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.856	M	0.187	3519928	8.686	9.006
2	13.231	M	0.087	4258214	13.120	13.384
3	32.719	M	0.180	235144114	32.403	33.038

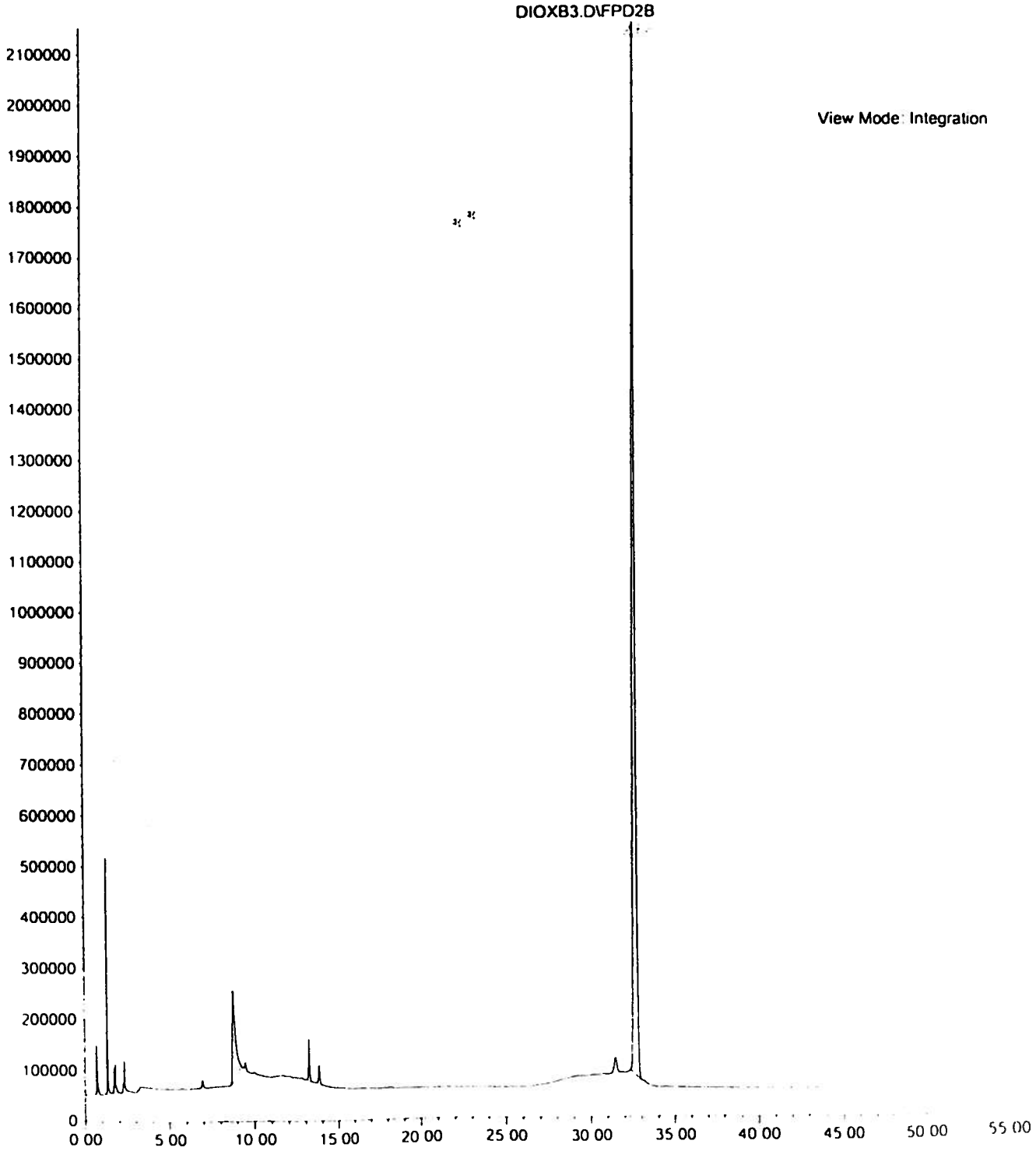
4B – Dioxathion Standard Analyzed at Injection Port of 190°C

File : C:\HPCHEM\1\DATA\072199\DIOXA3.D
Operator :
Acquired : 22 Jul 99 12:26 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



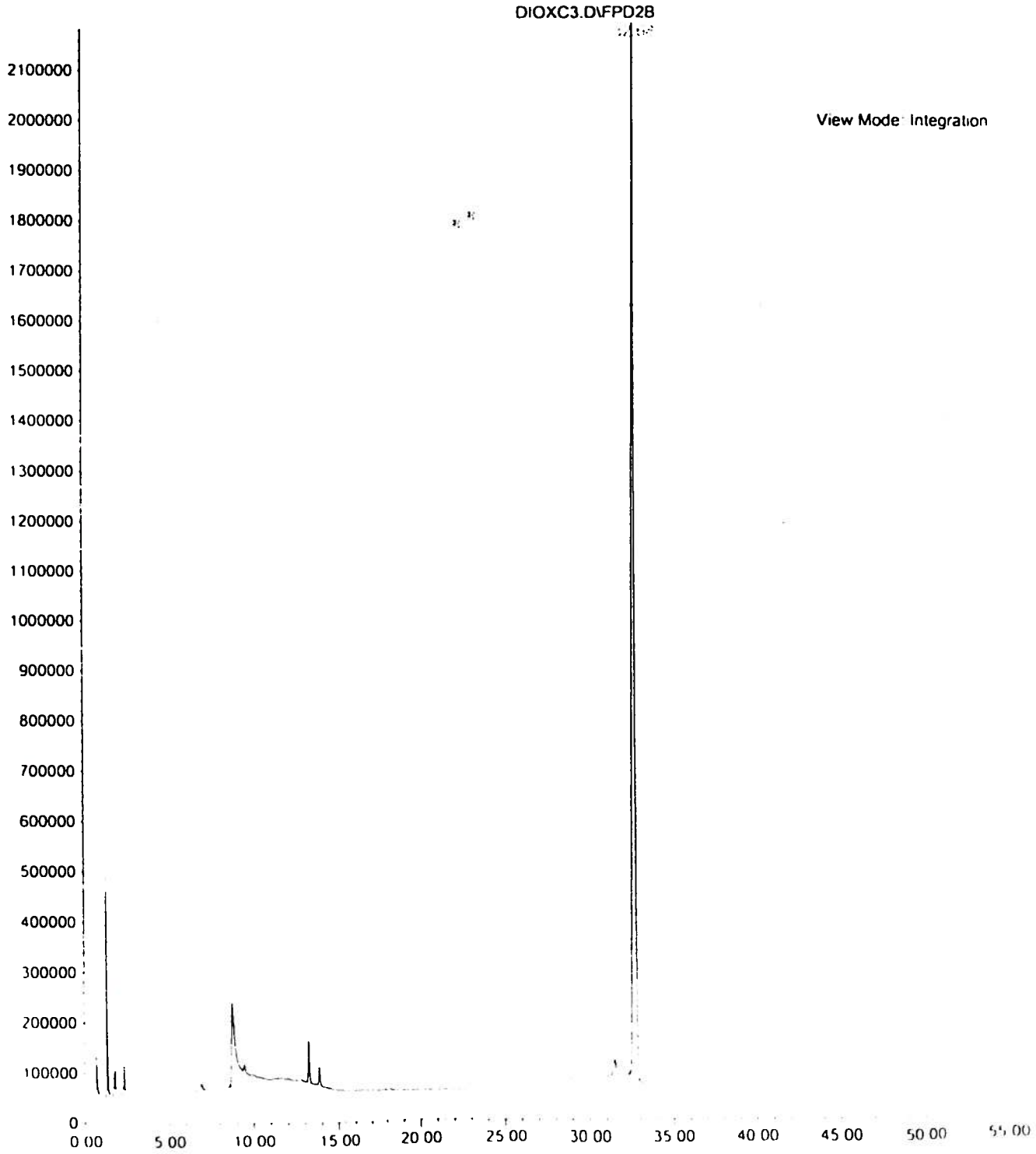
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.790	M	0.232	26835902	8.597	9.323
2	13.208	M	0.089	4748962	13.050	13.345
3	32.673	M	0.179	223039399	32.294	33.008

File : C:\HPCHEM\1\DATA\072199\DIOXB3.D
Operator :
Acquired : 22 Jul 99 13:30 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.780	M	0.245	26943578	8.632	9.386
2	13.205	M	0.090	4662562	13.064	13.349
3	32.676	M	0.180	223581528	32.394	32.979

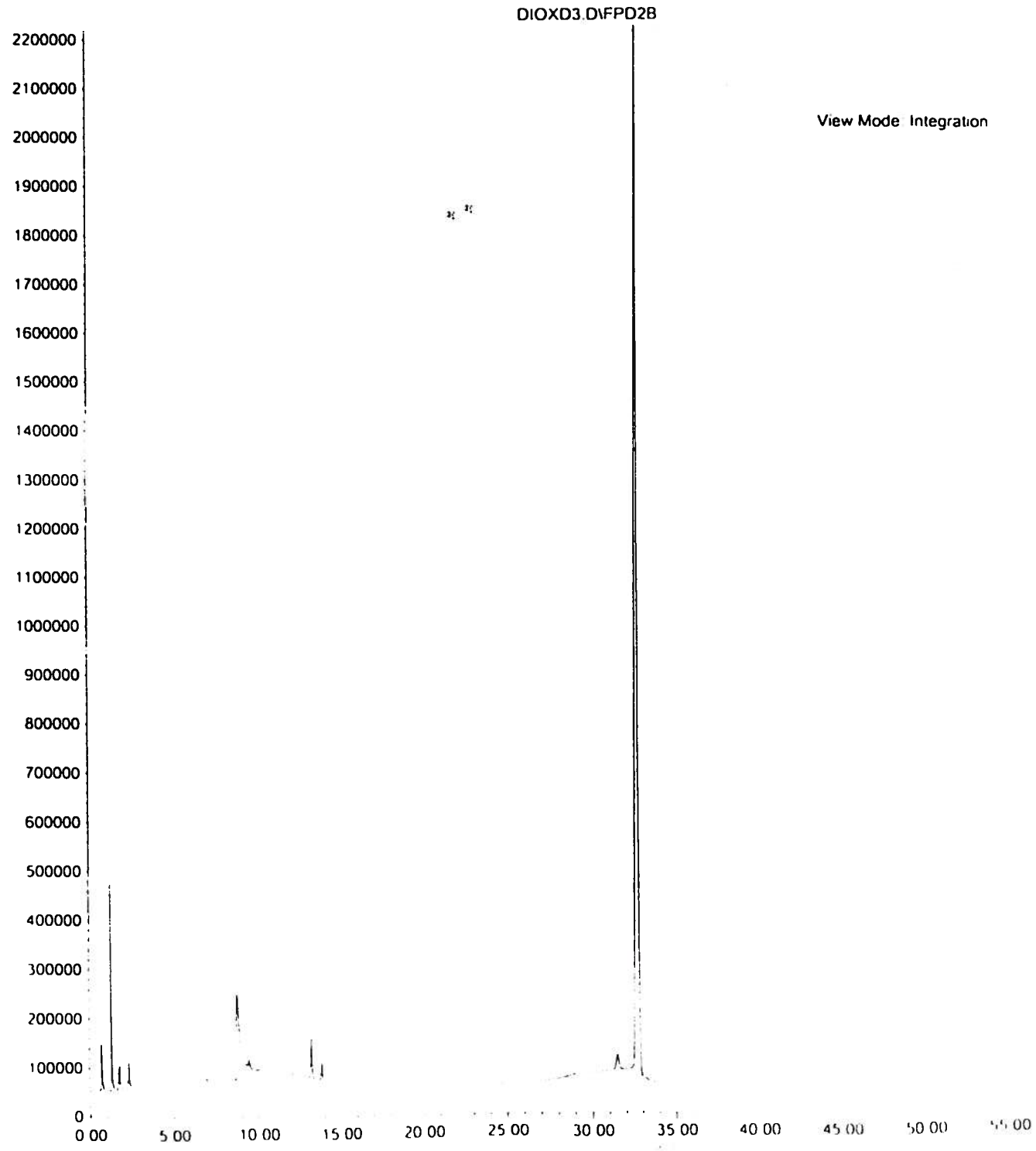
File : C:\HPCHEM\1\DATA\072199\DIOXC3.D
Operator :
Acquired : 22 Jul 99 15:39 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



D10XC3.D\FPD2B

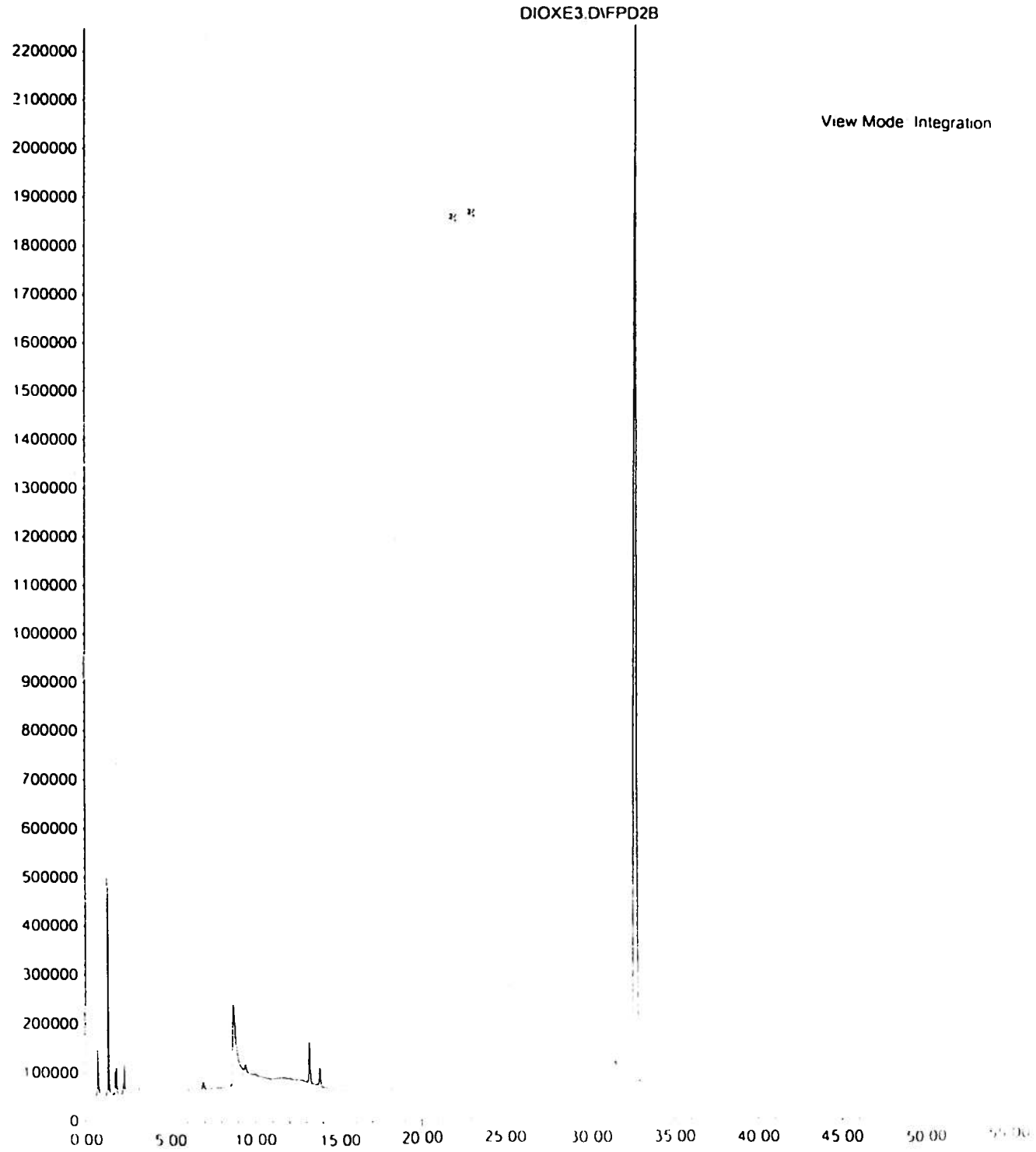
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.783	M	0.250	24704408	8.607	9.371
2	13.207	M	0.089	4667440	13.066	13.344
3	32.677	M	0.180	227063957	32.306	33.015

File : C:\HPCHEM\1\DATA\072199\DIOXD3.D
Operator :
Acquired : 22 Jul 99 16:43 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



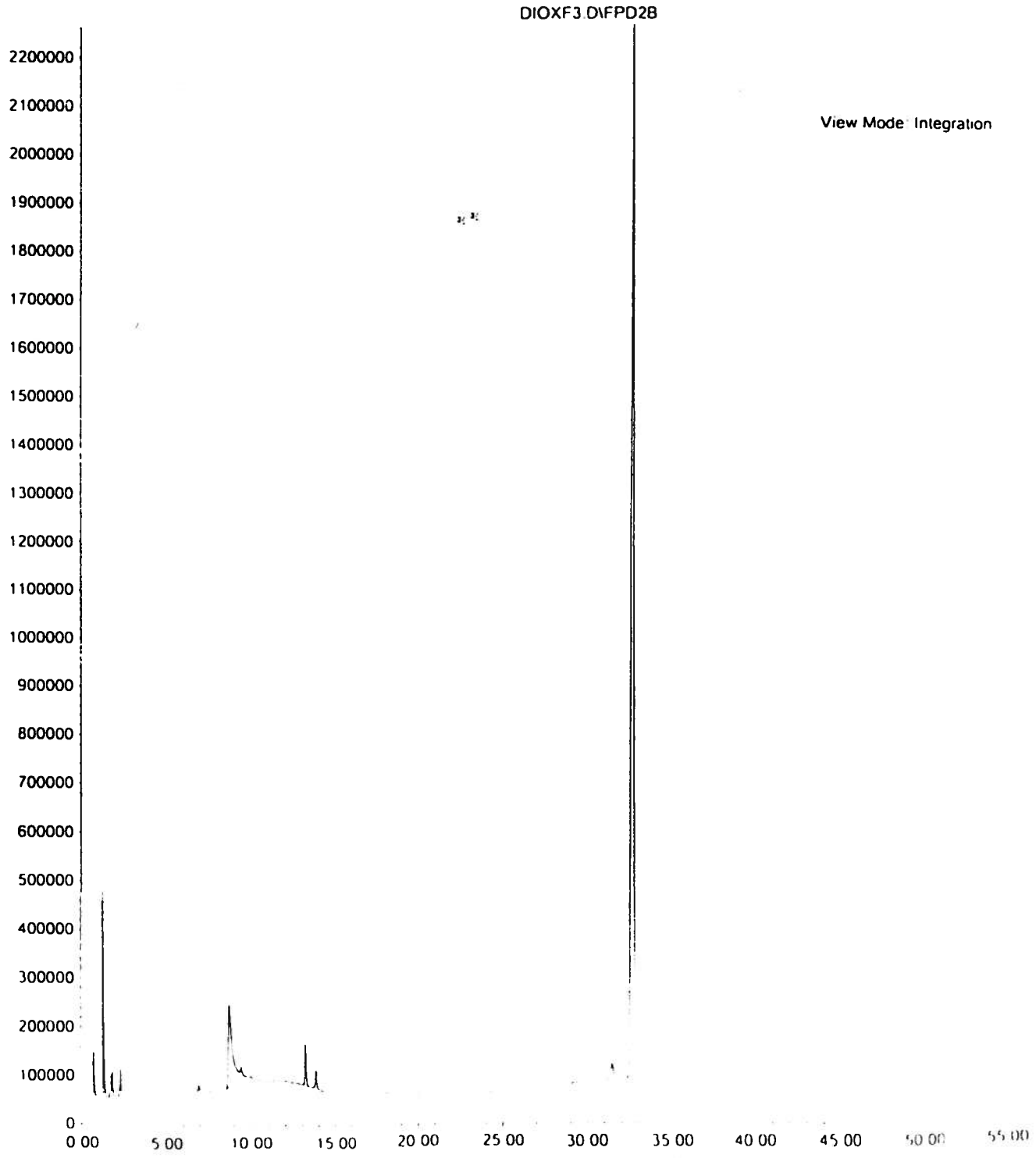
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.780	M	0.247	25851068	8.530	9.385
2	13.201	M	0.093	4921065	13.053	13.350
3	32.675	M	0.178	228092392	32.269	33.034

File : C:\HPCHEM\1\DATA\072199\DIOXE3.D
Operator :
Acquired : 22 Jul 99 17:47 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



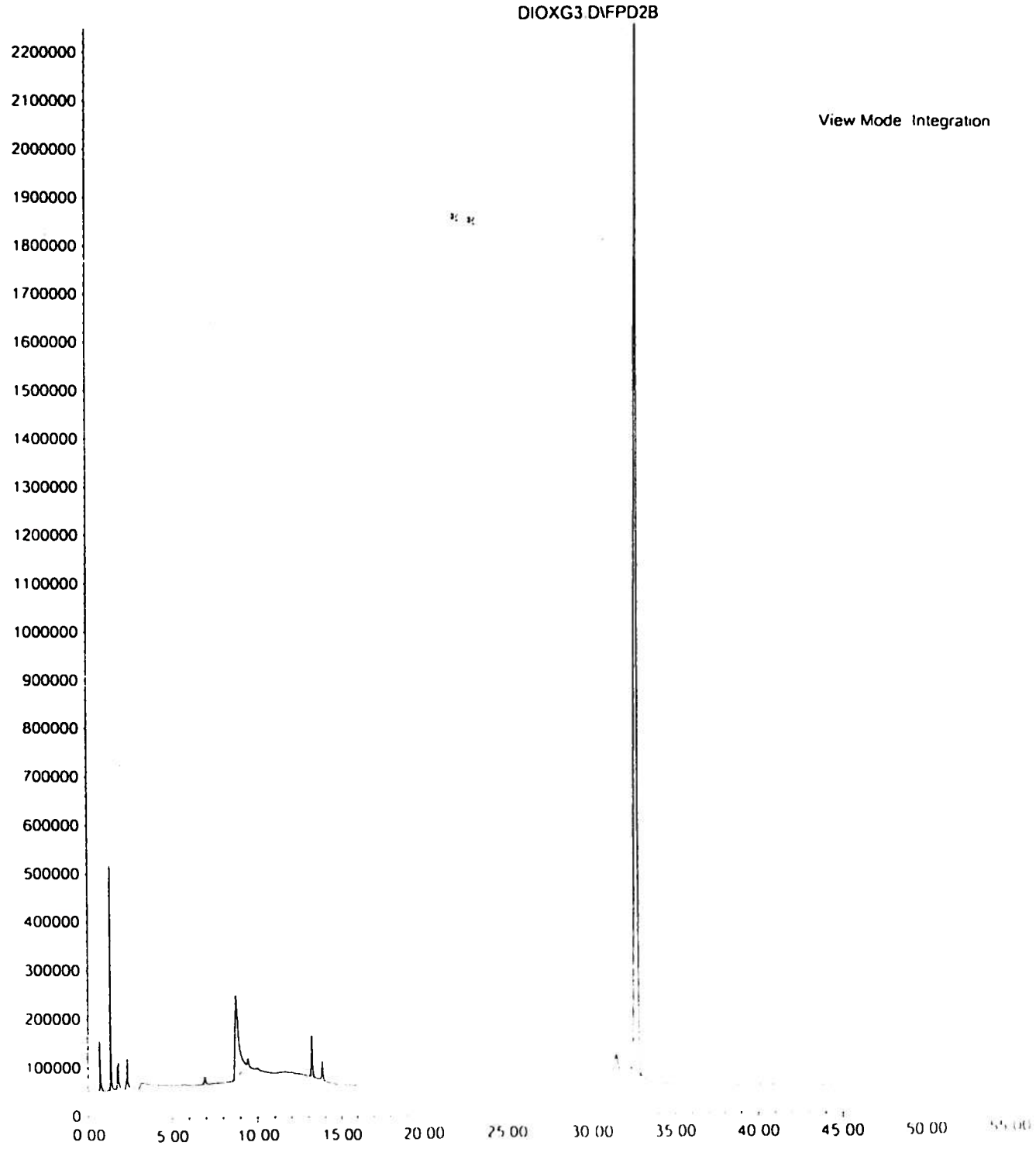
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.775	M	0.248	24262766	8.561	9.355
2	13.198	M	0.093	4927239	13.060	13.343
3	32.673	M	0.178	231778415	32.337	33.027

File : C:\HPCHEM\1\DATA\072199\DIOXF3.D
Operator :
Acquired : 22 Jul 99 18:51 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.779	M	0.248	25228732	8.615	9.368
2	13.201	M	0.090	4740706	13.078	13.320
3	32.674	M	0.178	232701733	32.332	32.981

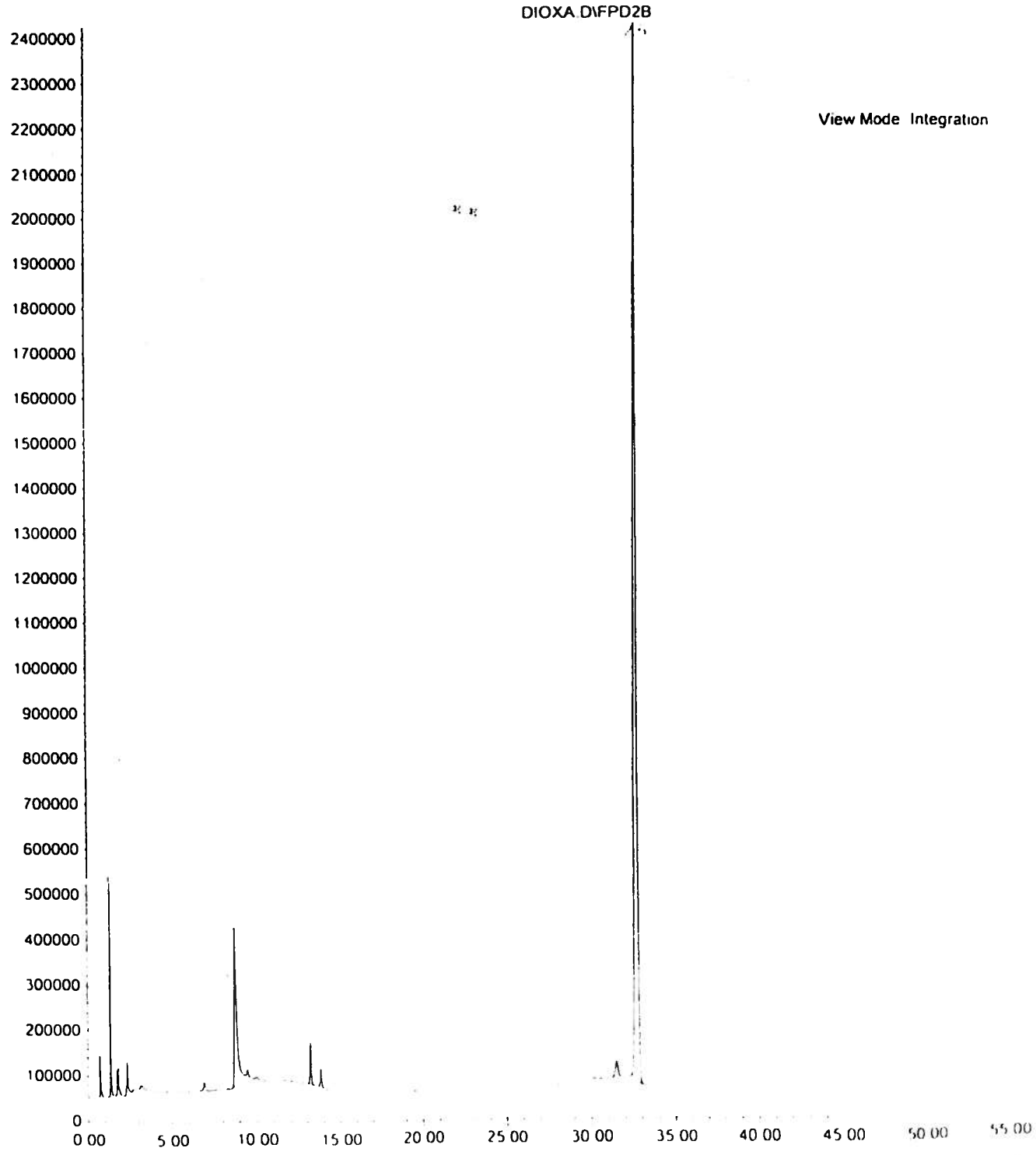
File : C:\HPCHEM\1\DATA\072199\DIOXG3.D
Operator :
Acquired : 22 Jul 99 19:55 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: Dioxathion Std. @ 100ppm
Misc Info : Spiked w chlor; Injection port @ 190 degrees
Vial Number: 4



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.777	M	0.244	24982250	8.585	9.372
2	13.201	M	0.090	4773053	13.085	13.333
3	32.672	M	0.180	234932804	32.358	33.002

4C – Dioxathion Standard Analyzed at Injection Port of 220°C

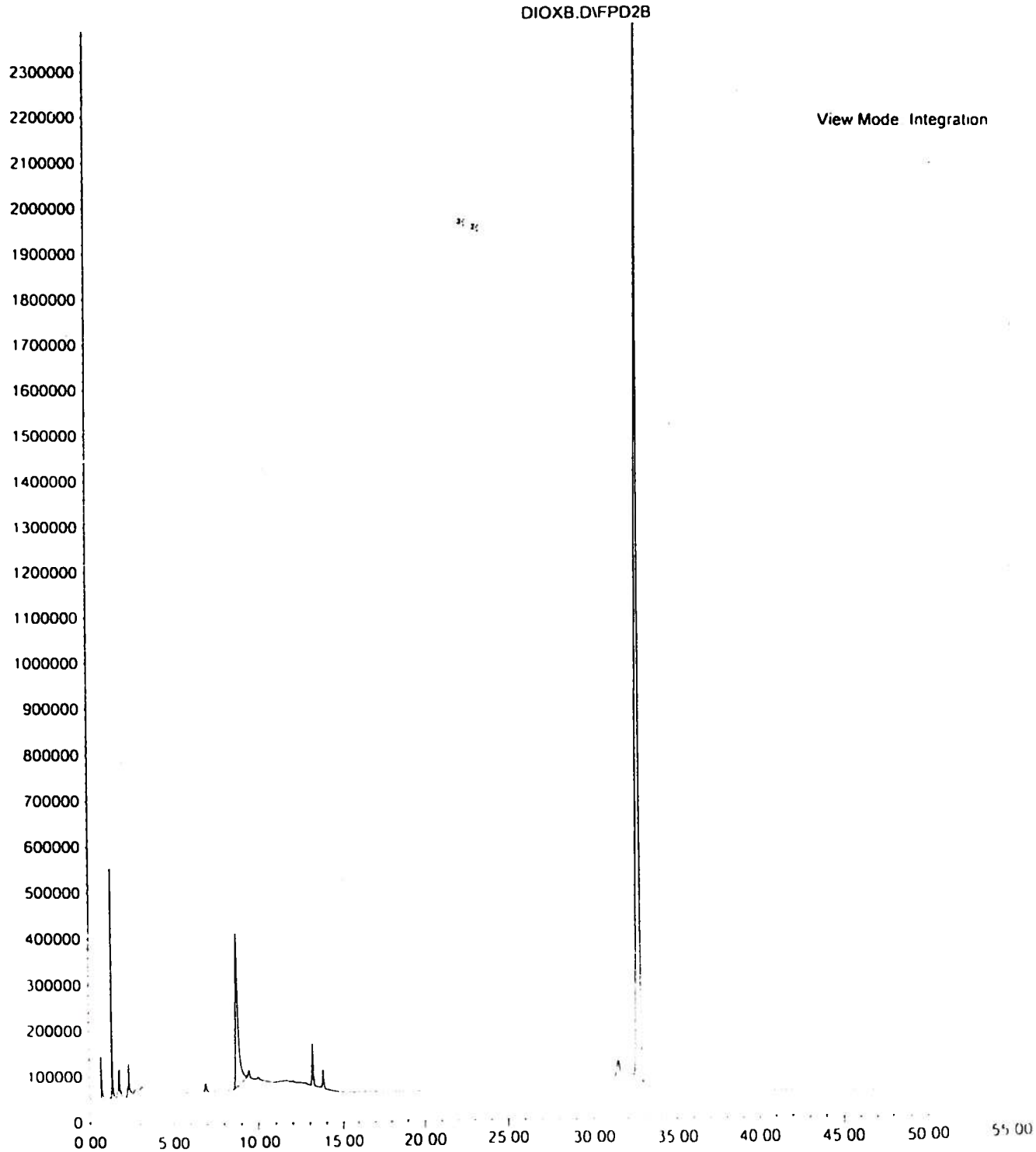
File : C:\HPCHEM\1\DATA\072699\DIOXA.D
Operator :
Acquired : 26 Jul 99 18:22 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: dioxathion std @ 100ppm
Misc Info : chlor @ 1ppm; injection port @ 220 degrees
Vial Number: 3



DIOXA.D\FPD2B

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.749	M	0.181	38271410	8.578	9.334
2	13.188	M	0.088	5005377	13.077	13.315
3	32.691	M	0.179	251926013	32.382	33.009

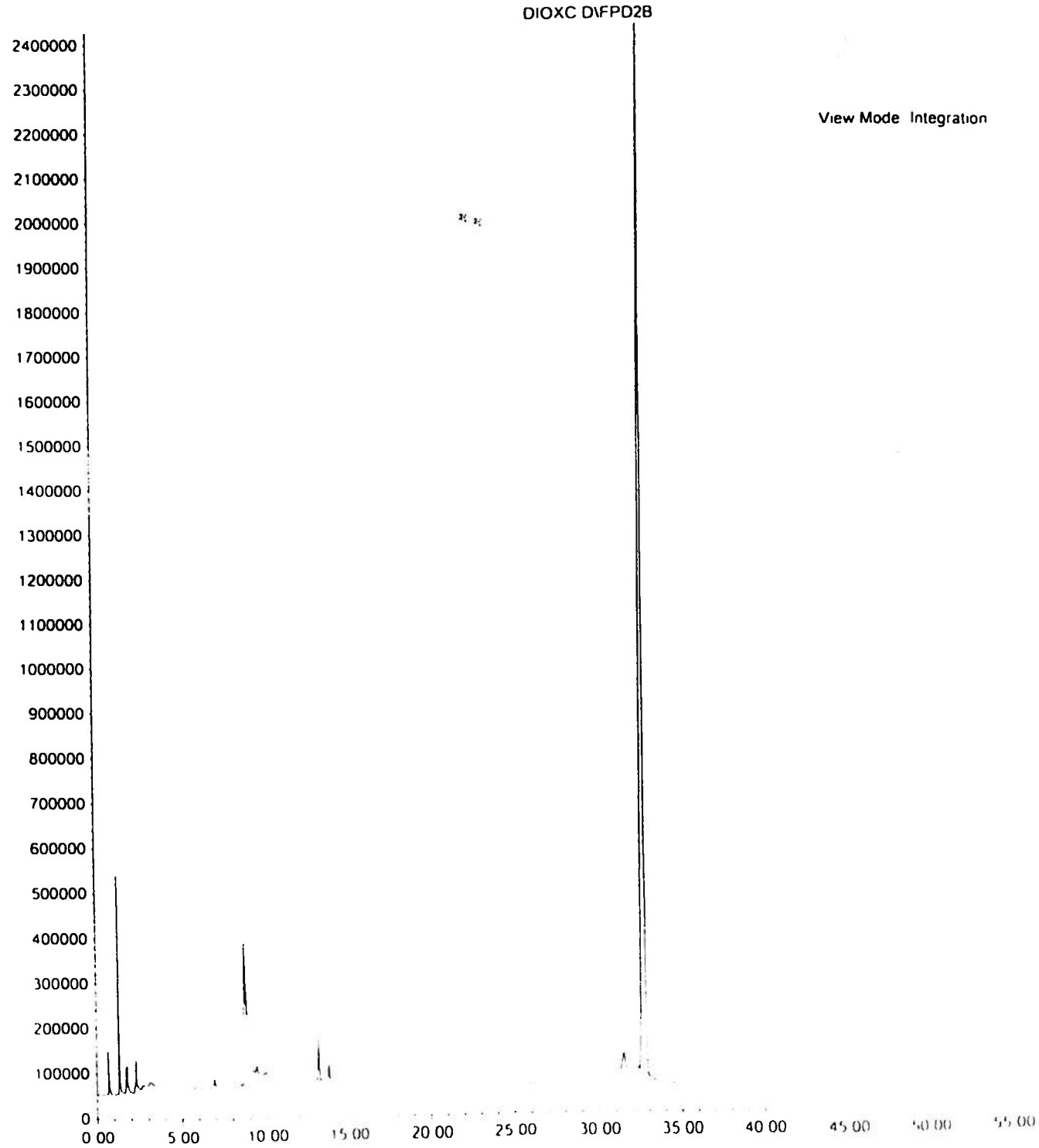
File : C:\HPCHEM\1\DATA\072699\DIOXB.D
Operator :
Acquired : 26 Jul 99 19:26 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: dioxathion std @ 100ppm
Misc Info : chlor @ 1ppm; injection port @ 220 degrees
Vial Number: 3



DIOXB.D\FPD2B

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	1.295	BB	0.034	11864006	1.250	1.420
2	8.751	BB	0.120	30928278	8.603	9.049
3	32.683	BB	0.144	247369039	32.409	33.027

File : C:\HPCHEM\1\DATA\072699\DIOXC.D
Operator :
Acquired : 26 Jul 99 20:30 using AcqMethod STATE.M
Instrument : FPD/FID I
Sample Name: dioxathion std @ 100ppm
Misc Info : chlor @ 1ppm; injection port @ 220 degrees
Vial Number: 3



Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	8.750	M	0.189	35980523	8.543	9.379
2	13.190	M	0.090	5181867	13.071	13.303
3	32.677	M	0.179	252268801	32.384	32.988

APPENDIX 5

5A – Table 3 – A Comparison of Peak #1 & Peak #2
Standard Curves at 140°C Injection & 220°C Injection

5B – Raw Data – Chromatograms for Table 3
Standard Curve at 140°C Injection

5C – Raw Data – Chromatograms for Table 3
Standard Curve at 220°C Injection

TABLE 3

A Comparison of Dioxathion Standard Curve Peak Areas at Injecton port Temperatures of 140°C and 220°C

CONCENTRATION OF DIOXATHION (PPB)	AREA Peak #1 8:17 @ 220°	AREA Peak #2 32:67 @ 220°	AREA Peak #1 8:17 @ 140°	AREA Peak #2 32:67 @ 140°
10	0	0	0	0
100	48,744	211,555	0	317,593
500	172,273	1,342,216	0	1,525,991
1000	325,255	2,818,252	0	3,022,982
10000	3,354,621	30,357,694	271,286	35,727,623
100000	28,060,253	260,319,076	1,823,342	287,369,745
LINEARITY	0.999816732	0.999859964	0.99854932	0.999699157

5B – Raw Data – Chromatograms for Table 3
Standard Curve at 140°C Injection

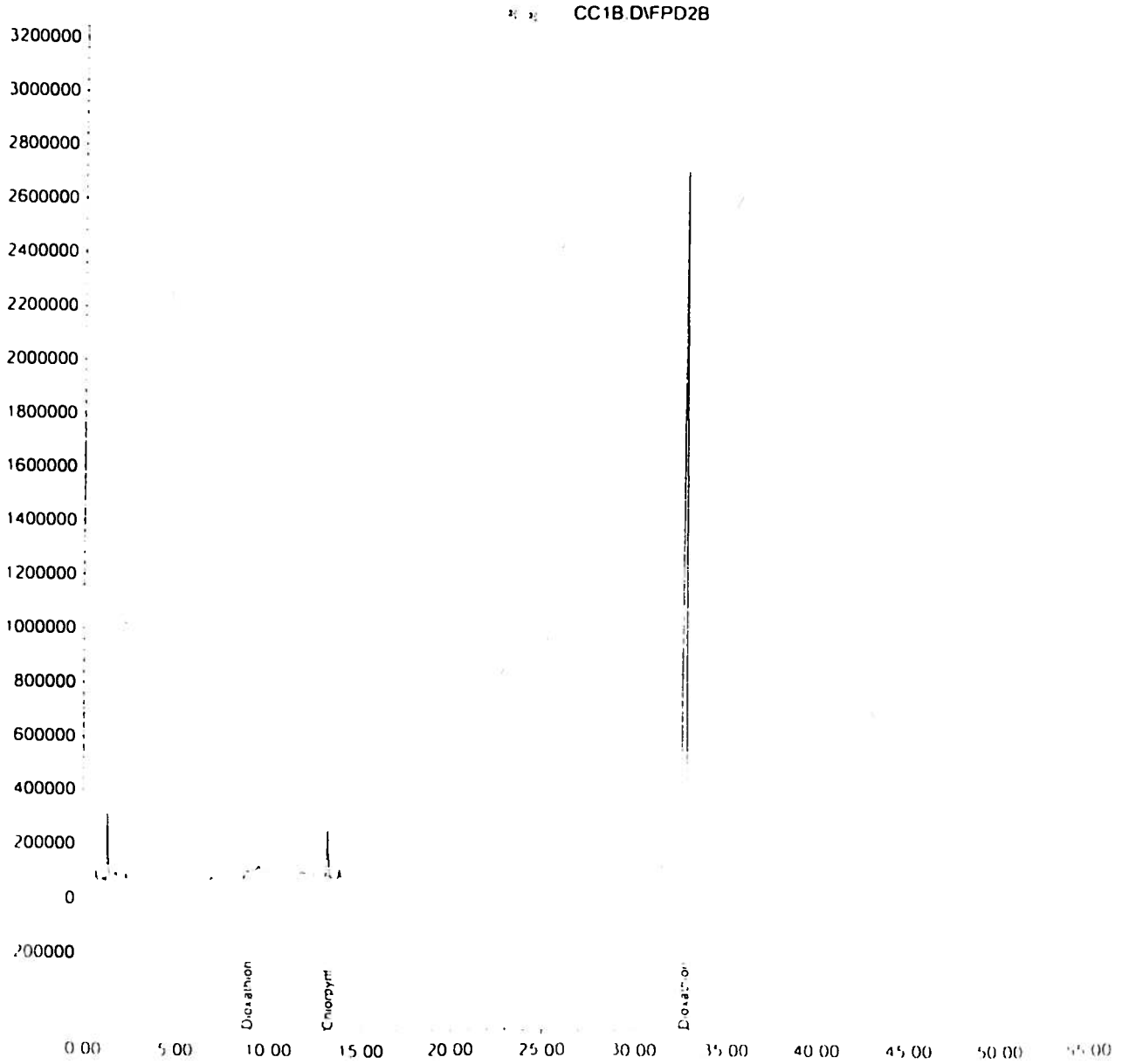
Calibration Curve

Data File : C:\HPCHEM\1\DATA\072799\CC1B.D Vial: 6
Acq On : 28 Jul 99 15:42 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FI
Misc : Diox @ 100ppm; Chlor @ 1ppm: 140 degrees Multiplr: 1.00
Sample Amount: 0.00
IntFile : events.e

Quant Time: Jul 28 16:45 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



Quantitation Report (QA Reviewed)

Data File : C:\HPCHEM\1\DATA\072799\CC1B.D Vial: 6
Acq On : 28 Jul 99 15:42 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 100ppm; Chlor @ 1ppm: 140 degrees Multiplr: 1.00
Sample Amount: 0.00
IntFile : events.e

Quant Time: Jul 28 16:45 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

	Compound	R.T.	Response	Conc	Units	
	System Monitoring Compounds					
1)	S Chlorpyrifos	13.22	10499641	1999.126	ppb	m
Spiked Amount	1000.000		Recovery =	199.91%		
	Target Compounds					
2)	T Dioxathion Peak #1	8.89	1823342	5141.081	ppb	m
3)	T Dioxathion Peak #2	32.73	287369745	96930.270	ppb	m

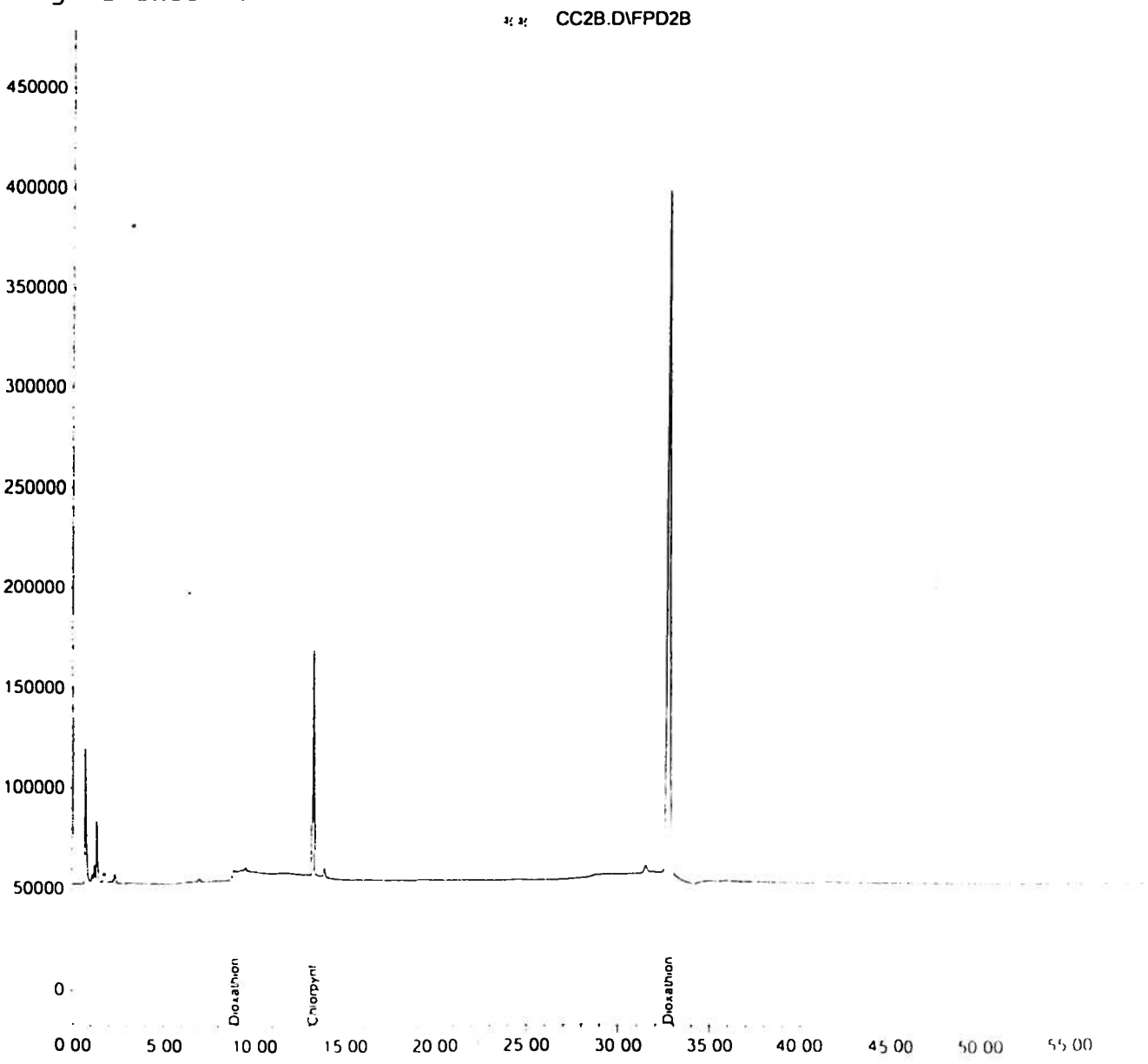
(f)=RT Delta > 1/2 Window (m)=manual int.

Data File : C:\HPCHEM\1\DATA\072799\CC2B.D Vial: 5
Acq On : 28 Jul 99 14:19 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 10ppm; Chlor @ 1ppm; 140 degrees Multiplr: 1.00
Sample Amount: 0.00
IntFile : events.e

Quant Time: Jul 28 15:27 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



Data File : C:\HPCHEM\1\DATA\072799\CC2B.D Vial: 5
Acq On : 28 Jul 99 14:19 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 10ppm; Chlor @ 1ppm; 140 degrees Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 15:27 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc	Units	
System Monitoring Compounds					
1) S Chlorpyrifos	13.21	6234166	1186.982	ppb	m
Spiked Amount 1000.000		Recovery = 118.70%			
Target Compounds					
2) T Dioxathion Peak #1	8.89	271286	764.915	ppb	m
3) T Dioxathion Peak #2	32.71	35727623	12050.984	ppb	m

(f)=RT Delta > 1/2 Window (m)=manual int.
CC2B.D 8141A.M Wed Jul 28 15:27:36 1999 RHONDA Page

Quantitation Report

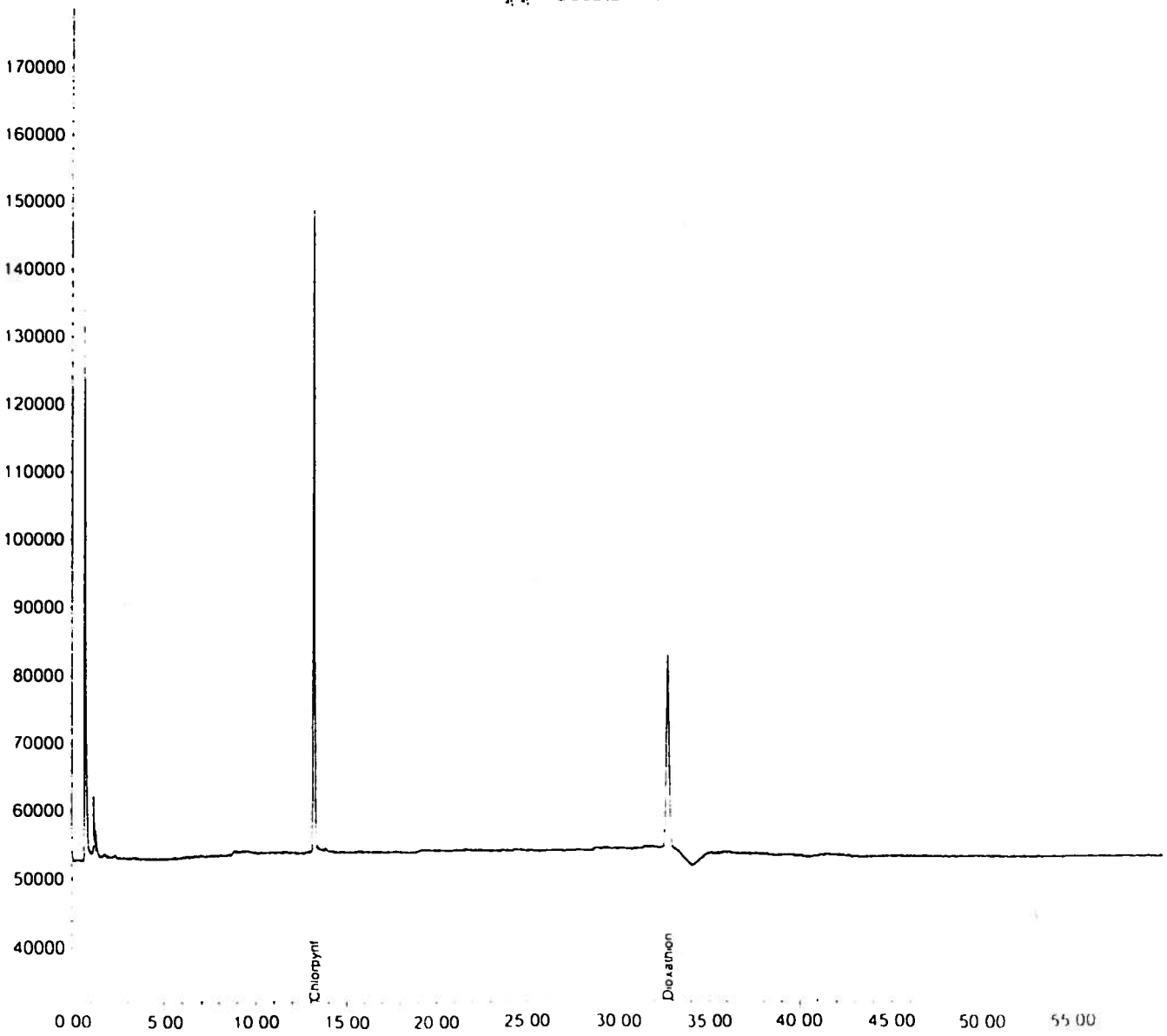
Data File : C:\HPCHEM\1\DATA\072799\CC3B.D Vial: 4
Acq On : 28 Jul 99 12:56 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 1ppm; Chlor @ 1ppm; 140 degrees Multiplr: 1.00
Sample Amount: 0.00
IntFile : events.e

Quant Time: Jul 28 13:58 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

CC3B.D\FPD2B



Quantitation Report (QT Reviewed)

Data File : C:\HPCHEM\1\DATA\072799\CC3B.D Vial: 4
Acq On : 28 Jul 99 12:56 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 1ppm; Chlor @ 1ppm; 140 degrees Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 13:58 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc	Units	
System Monitoring Compounds					
1) S Chlorpyrifos	13.22	5262139	1001.909	ppb	m
Spiked Amount 1000.000		Recovery = 100.19%			
Target Compounds					
2) T Dioxathion Peak #1	0.00	0	N.D.	ppb	
3) T Dioxathion Peak #2	32.71	3022982	1019.657	ppb	m

(f)=RT Delta > 1/2 Window (m)=manual int.

Data File : C:\HPCHEM\1\DATA\072799\CC4B.D
Acq On : 28 Jul 99 11:32
Sample : Calibration Curve 07/27/99
Misc : Diox @ 500ppb; Chlor @ 1 ppm; 140 degree

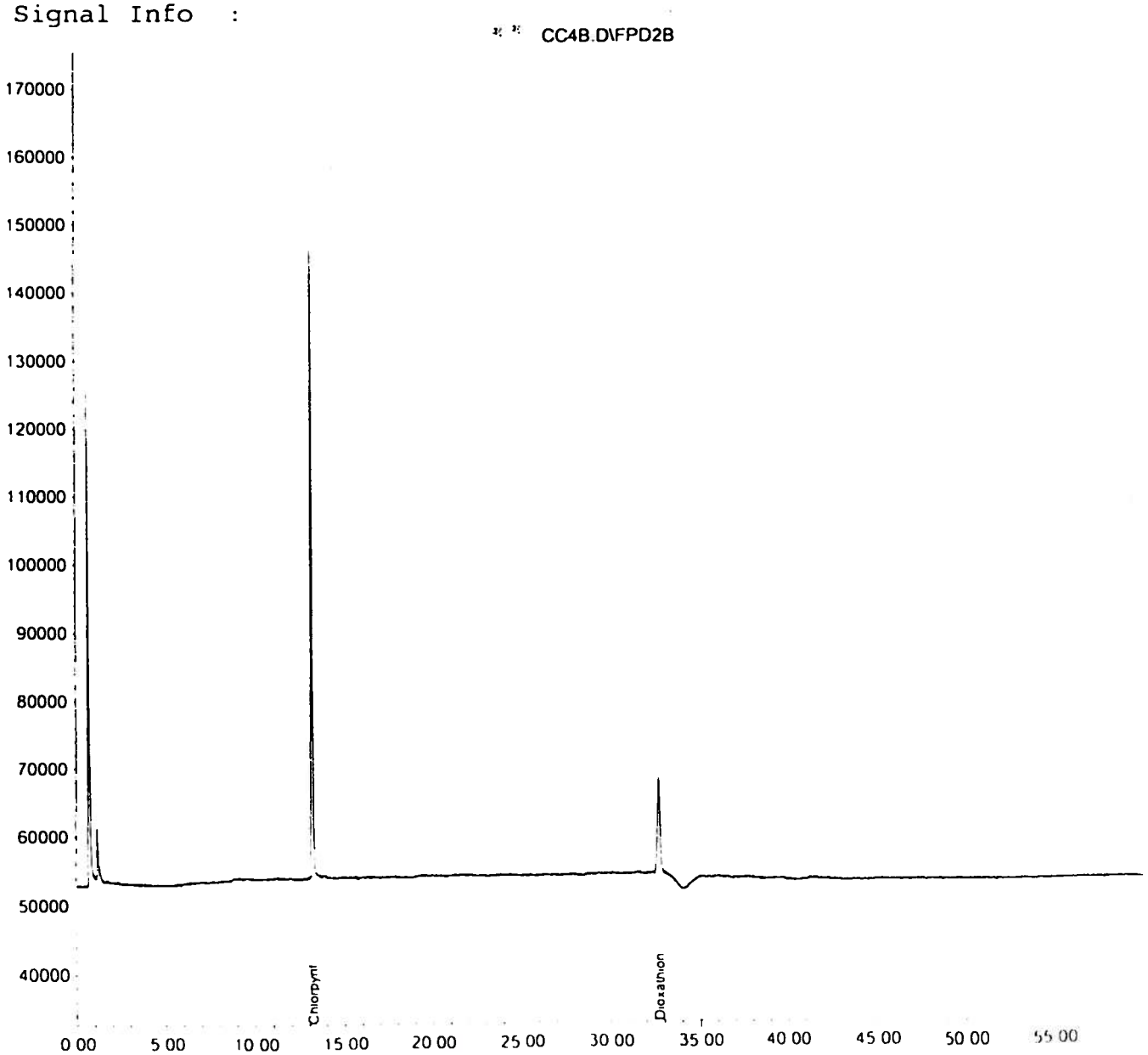
Vial: 3
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 13:32 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



Quantitation Report (QT Reviewed)

Data File : C:\HPCHEM\1\DATA\072799\CC4B.D Vial: 3
Acq On : 28 Jul 99 11:32 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 500ppb; Chlor @ 1 ppm; 140 degree Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 13:32 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc	Units	
System Monitoring Compounds					
1) S Chlorpyrifos	13.21	5161948	997.543	ppb	m
Spiked Amount 1000.000		Recovery = 99.75%			
Target Compounds					
2) T Dioxathion Peak #1	0.00	0	N.D.	ppb	
3) T Dioxathion Peak #2	32.71	1525991	527.806	ppb	m

(f)=RT Delta > 1/2 Window (m)=manual int.

Data File : C:\HPCHEM\1\DATA\072799\CC5B.D
Acq On : 28 Jul 99 10:08
Sample : Calibration Curve 07/27/99
Misc : Diox @ 100ppb; Chlor @ 1ppm; 140 degrees

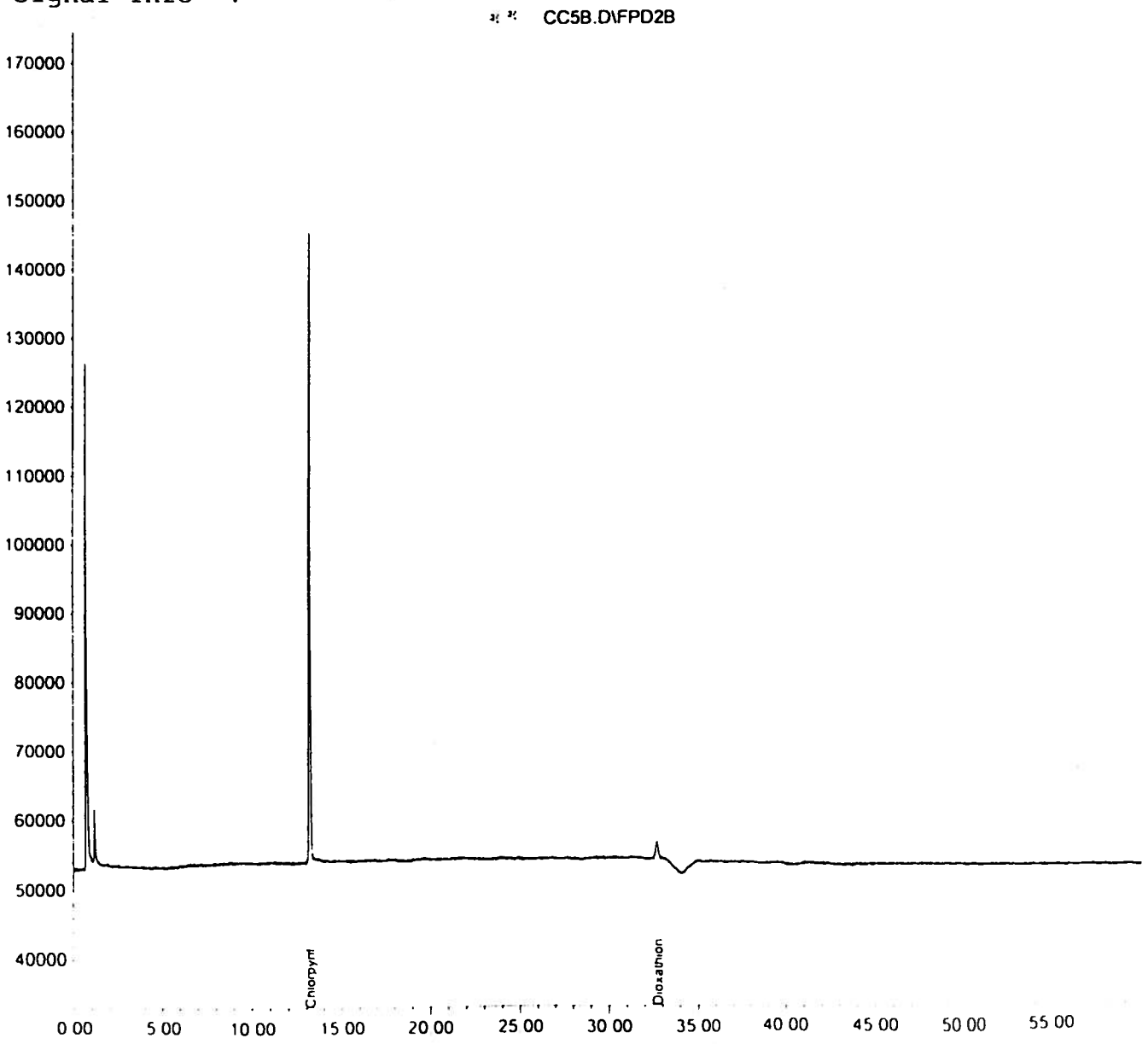
Vial: 2
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 12:00 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



Quantitation Report (QT Reviewed)

Data File : C:\HPCHEM\1\DATA\072799\CC5B.D
Acq On : 28 Jul 99 10:08
Sample : Calibration Curve 07/27/99
Misc : Diox @ 100ppb; Chlor @ 1ppm; 140 degrees

Vial: 2
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 12:00 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc Units
System Monitoring Compounds			
1) S Chlorpyrifos	13.22	5091860	998.753 ppb m
Spiked Amount 1000.000		Recovery =	99.88%
Target Compounds			
2) T Dioxathion Peak #1	0.00	0	N.D. ppb
3) T Dioxathion Peak #2	32.72	317593	119.781 ppb m

(f)=RT Delta > 1/2 Window (m)=manual int.

Data File : C:\HPCHEM\1\DATA\072799\CC6B.D
Acq On : 28 Jul 99 8:46
Sample : Calibration Curve 07/27/99
Misc : Diox @ 10ppb; Chlor @ 1ppm; 140 degrees

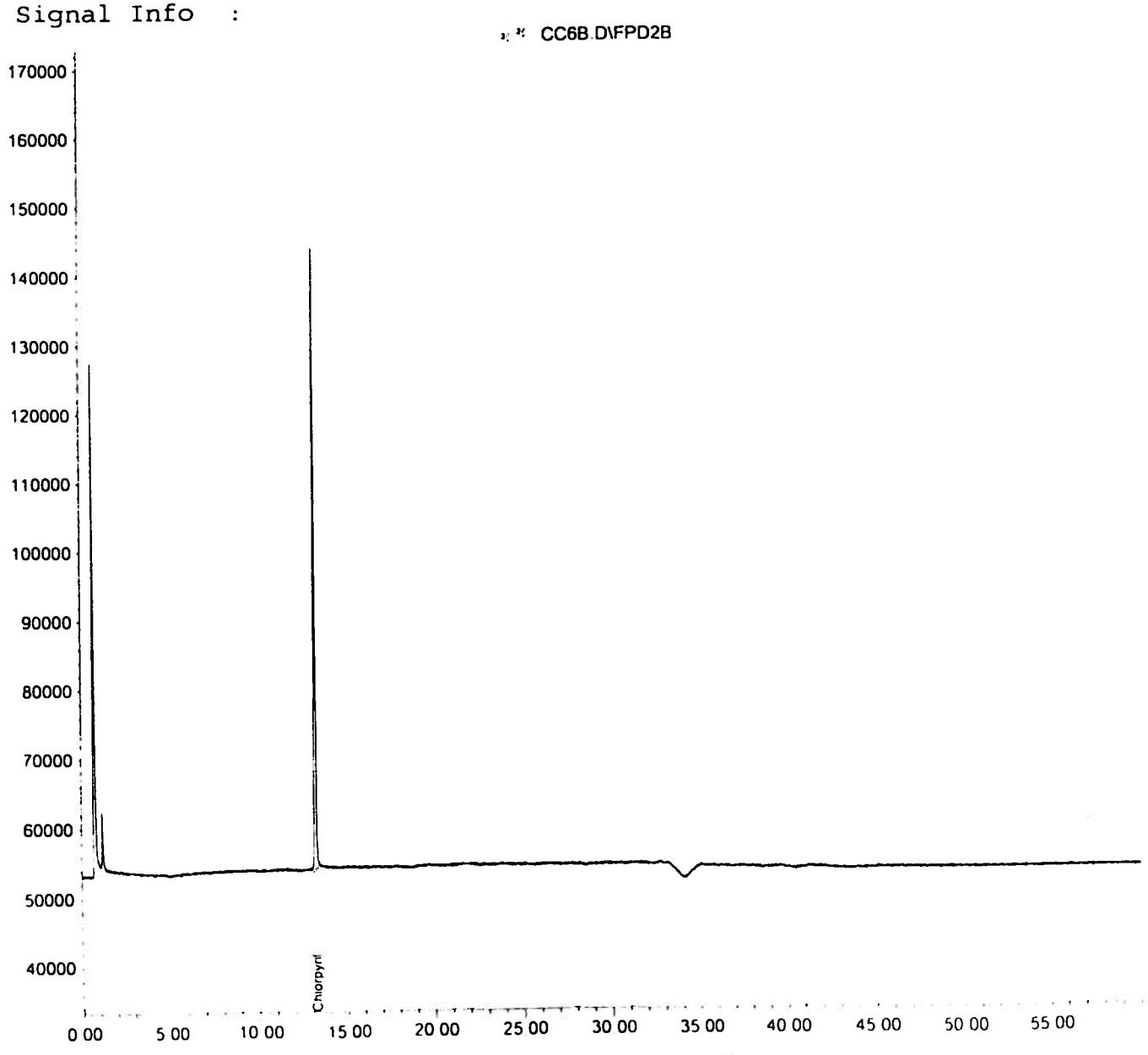
Vial: 1
Operator:
Inst : FPD/FID I
Multiplr: 0.01
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 11:53 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



Data File : C:\HPCHEM\1\DATA\072799\CC6B.D Vial: 1
Acq On : 28 Jul 99 8:46 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID I
Misc : Diox @ 10ppb; Chlor @ 1ppm; 140 degrees Multiplr: 0.01
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 11:53 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 11:23:33 1999
Response via : Initial Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc	Units	
System Monitoring Compounds					
1) S Chlorpyrifos	13.22	5129388	1006.114	ppb	m
Spiked Amount 1000.000		Recovery = 100.61%			
Target Compounds					
2) T Dioxathion Peak #1	0.00	0	N.D.	ppb	
3) T Dioxathion Peak #2	0.00	0	N.D.	ppb	

(f)=RT Delta > 1/2 Window (m)=manual int.

5B – Raw Data – Chromatograms for Table 3
Standard Curve at 140°C Injection

Midpoint

Data File : C:\HPCHEM\1\DATA\072799\MDPT2.D
Acq On : 29 Jul 99 4:21
Sample : Calibration Curve 07/27/99
Misc : Diox @ 1ppm; Chlor @ 1ppm; 140 degrees

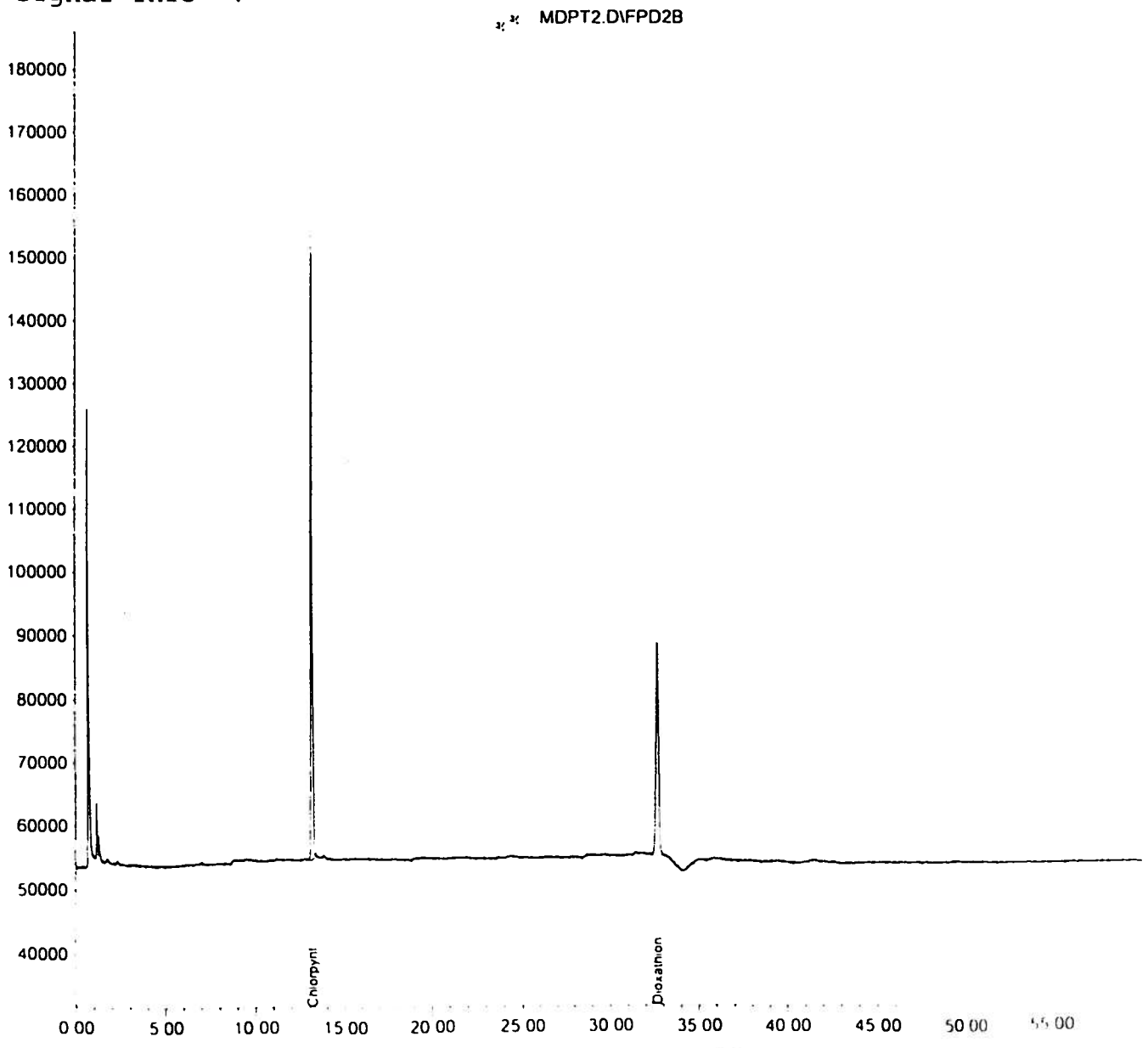
Vial: 4
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 29 8:48 1999 Quant Results File: 8141AB.RES

Quant Method : C:\HPCHEM\1\METHODS\8141AB.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 09:17:49 1999
Response via : Multiple Level Calibration
DataAcq Meth : INJTEMP.M

Volume Inj. :
Signal Phase :
Signal Info :



5C – Raw Data – Chromatograms for Table 3
Standard Curve at 220°C Injection

Calibration Curve

Quantitation Report

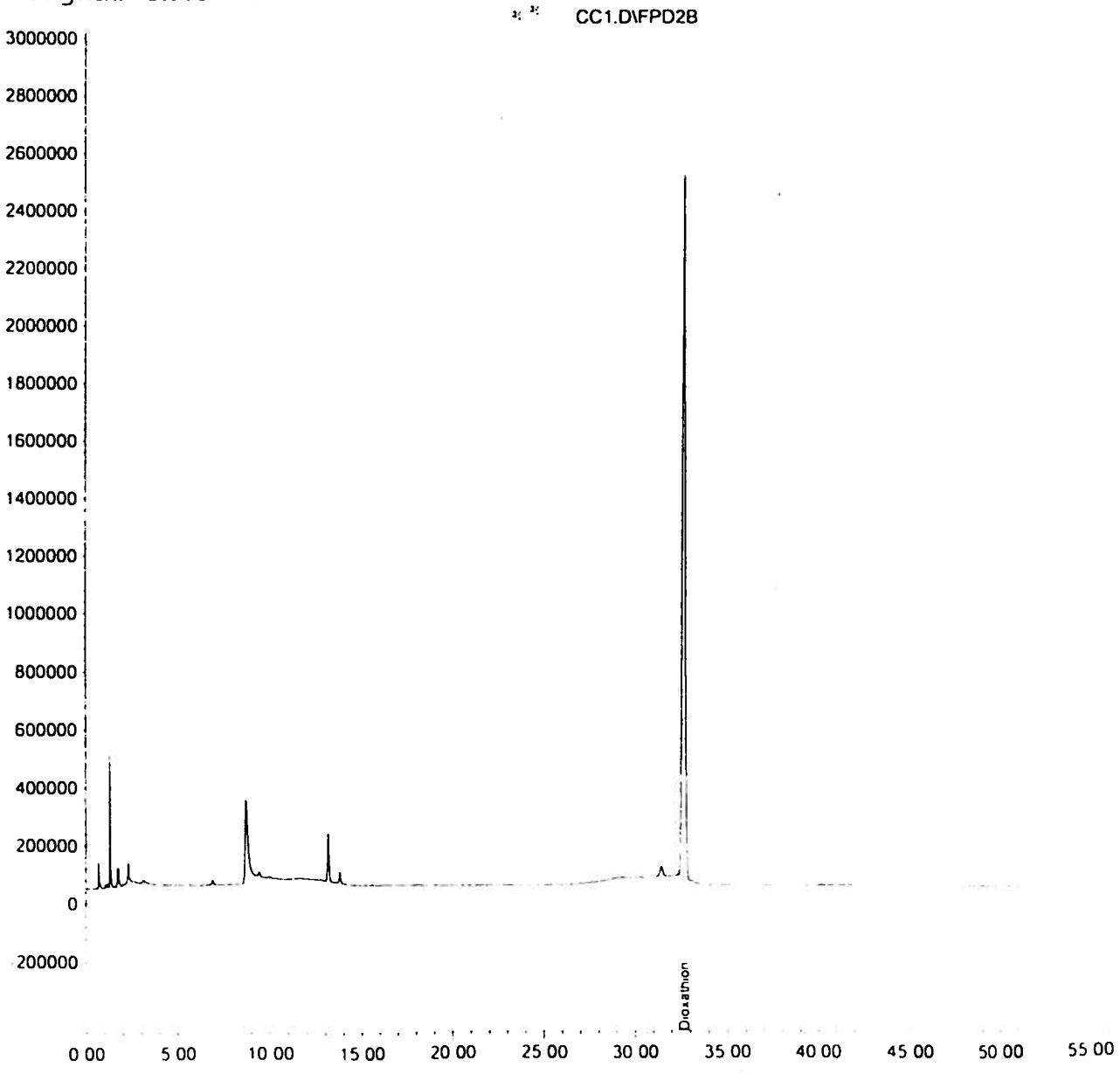
Data File : C:\HPCHEM\1\DATA\072799\CC1.D
Acq On : 27 Jul 99 22:46
Sample : Calibration Curve 07/27/99
Misc : Diox @ 100ppm; Chlor @ 1ppm; 220 degrees
IntFile : events.e

Vial: 6
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

Quant Time: Jul 28 9:01 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 08:58:17 1999
Response via : Single Level Calibration
DataAcq Meth : STATE.M

Volume Inj. :
Signal Phase :
Signal Info :



Quantitation Report (QT Reviewed)

Data File : C:\HPCHEM\1\DATA\072799\CC1.D Vial: 6
Acq On : 27 Jul 99 22:46 Operator:
Sample : Calibration Curve 07/27/99 Inst : FPD/FID
Misc : Diox @ 100ppm; Chlor @ 1ppm; 220 degreés Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 9:01 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 08:58:17 1999
Response via : Initial Calibration
DataAcq Meth : STATE.M

Volume Inj. :
Signal Phase :
Signal Info :

Compound	R.T.	Response	Conc Units
System Monitoring Compounds			
1) S Chlorpyrifos	13.18	10148126	NoCal ppb
Spiked Amount 1000.000		Recovery =	0.00%
Target Compounds			
2) T Dioxathion Peak #1	8.77	28060253	NoCal ppb
3) T Dioxathion Peak #2	32.67	260319076	125303.733 PPB

(f)=RT Delta > 1/2 Window (m)=manual int.

Data File : C:\HPCHEM\1\DATA\072799\CC2.D
Acq On : 27 Jul 99 21:42
Sample : Calibration Curve 07/27/99
Misc : Diox @ 10ppm; Chlor @ 1ppm; 220 degrees

Vial: 5
Operator:
Inst : FPD/FID
Multiplr: 1.00
Sample Amount: 0.00

IntFile : events.e

Quant Time: Jul 28 9:06 1999 Quant Results File: 8141A.RES

Quant Method : C:\HPCHEM\1\METHODS\8141A.M (Chemstation Integrator)
Title :
Last Update : Wed Jul 28 09:04:13 1999
Response via : Single Level Calibration
DataAcq Meth : STATE.M

Volume Inj. :
Signal Phase :
Signal Info :