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**Author** Smith, R.M.

**Corporate Author**

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**Description Notes**

Determination of Tetra-Hexa CDFs and Tetra-CDDs in  
Air Samples from the 11, 14, 16 and 17th Floors  
of the Binghamton State Office Building

R. M. Smith, D. Hilker, P. O'Keefe and K. Aldous  
Laboratory for Organic Analytical Chemistry  
New York State Department of Health

5/16/83

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## INTRODUCTION

Analysis of air samples taken from the 16th floor (NE, SE, NW, and SW Corners) of the Binghamton State Office Building (March 16, 1983 NYS report) showed concentrations of from 7 to 16 pg/m<sup>3</sup> 2,3,7,8 TCDF and 52-102 pg/m<sup>3</sup> total TCDF. Penta-CDF appeared to be lower in concentration, detected in only one sample out of four at a total concentration of 22 pg/m<sup>3</sup>. 2,3,7,8 TCDD, found at 0.26-2.2 ppm levels in soot samples analyzed after the transformer fire, was not confirmed in air samples from the 16th floor at concentrations of <1.3 pg/m<sup>3</sup>, although a signal at m/e 322 was detected.

In the present study it was necessary to analyze additional air samples to:

1. determine the floor-to-floor variation of PCDFs in air.
2. re-analyze samples to determine the precision of sampling and analysis.
3. separately analyze the particulate and adsorbed gaseous PCDFs.
4. combine samples for greater sensitivity for both PCDFs and TCDDs.

Using silica gel adsorbent cartridges containing labelled <sup>13</sup>C 2,3,7,8 TCDD, <sup>13</sup>C1 OCDD, <sup>13</sup>C1 2,3,7,8 TCDF and <sup>13</sup>C1 penta-CDF (3 isomers), 50 m<sup>3</sup> air were collected. They were then combined as necessary prior to clean-up and analysis by SP 2330 capillary GC/SIM high resolution mass spectrometry. It is important to note that while the analysis has been validated and is quantitative for 2,3,7,8 TCDD, 2,3,7,8 TCDF, OCDD and OCDF, certain non-2,3,7,8

substituted TCDD (and presumably PCDF) isomers have been found to be removed in the clean-up process.

## EXPERIMENTAL

### Samples for analysis

#### PCDFs

Floor 16, analyzed in our preliminary study of PCDFs in air, was sampled as a repeat analysis. Duplicate samples were taken on the 14th and 17th floor for which previously obtained full-scanning HRMS PCDF in soot (dust) data is available. For 17th floor samples, the glass fiber particulate filter ( $<.3\mu$ ) was analyzed separately from the silica gel gaseous adsorbent cartridge. To obtain greater sensitivity, particularly for the higher chlorinated PCDFs, a combined sample from the 11th floor was analyzed. Duplicate 10  $\text{pg}/\text{m}^3$  2,3,7,8 TCDF-fortified background air samples (NSA 6S, DOH 1) along with a background air sample (DOH 2) and a solvent blank were also analyzed as part of normal quality control procedures.

#### TCDDs

To obtain adequate sensitivity, four 50  $\text{m}^3$  samples were extracted, combined and analyzed (in duplicate) for TCDDs, injecting nearly the entire sample in a single injection. A combined solvent blank and a 1  $\text{pg}/\text{m}^3$  2,3,7,8 TCDD-fortified background air sample were also analyzed.

### Sampling Procedures

Generally, each BSOB sample taken on a given date was identified by a number specifying the floor, a letter specifying the corner of the building, another number indicating replicates and finally a letter P for particulate or G for gaseous, e.g. 17C2G.

In addition to the internal standards used in our earlier study (60  $\text{pg}$   $^{13}\text{C}$  2,3,7,8 TCDD, 5000  $\text{pg}$   $^{13}\text{C}$  OCDD and 1200  $\text{pg}$   $^{13}\text{C}$  2,3,7,8 TCDF), 24000  $\text{pg}$  of  $^{13}\text{C}$  penta-CDF (3 unspiked

isomers) in benzene was deposited directly onto the silica gel (activated at 140<sup>o</sup>C) trapping adsorbent prior to sampling. The 2 stage sampling apparatus, assembly, transport and sampling has been described in the March 16, 1983 NYS report.

#### Extraction

(See March 16, 1983 NYS report) 16 hr benzene soxhlet extraction. Internal standards were added to 17C1P and 17C2P prior to extraction. Native 2,3,7,8 TCDD and 2,3,7,8 TCDF were added to fortified samples prior to extraction.

#### Sample Clean-up

(See March 16, 1983 NYS report) The microprocessor controlled clean-up was a sequence of basic alumina, PX-21 carbon, and neutral alumina followed by a single concentration to 5-10  $\mu$ L for GC/MS injection.

#### Capillary GC/HRMS

In summary: 2  $\mu$ L of sample extract was injected onto a 0.3 mm x 60 m SP 2330 GC capillary interfaced directly into MS-50 via a fused silica SP 2330 butt-joint transfer line. The GC program was 70<sup>o</sup> for 3 min, 60<sup>o</sup>/min to 190<sup>o</sup>, 2.5<sup>o</sup>C/min to 240<sup>o</sup> hold. Kratos MS-50 mass spectrometer was used in the EI mode (70 ev), approx. 10,000 resolution (10% valley), source 250<sup>o</sup>C, DS-55 data system, with multiple peak monitoring mode as follows:

#### For PCDFs (m/e)

1st Injection: 304, 306 - M, M+2 of Tetra CDF  
312 - TCDF internal std  
340 - M+2 of penta CDF

2nd Injection: 338, 340 - M, M+2 of penta CDF  
 (optional)  
 348 - penta CDF internal std  
 374 - hexa CDF

For TCDDs

1st Injection: 320, 322 - M, M+2 of TCDD  
 334 - TCDD internal std.

Calculations

(See March 16, 1983 NYS report for details) Internal standard method  
 of calculation  $C_1 = X C_2 A_1/A_2$ .

TCDDs: individual scans at m/e 322 are summed across a single  
 peak (for 2,3,7,8 TCDD) or a range of observed peaks (for  
 total TCDDs). The area under the resulting mass profile  
 is then ratioed to that of the  $^{13}C$  2,3,7,8 TCDD  
 internal standard at m/e 334.

TCDFs: individual scans at m/e 306 are summed across a single  
 peak (for 2,3,7,8 TCDF) or a range of observed peaks (for  
 total TCDFs). The area under the resulting mass profile  
 is then ratioed to that of the  $^{37}Cl$  2,3,7,8 TCDF  
 internal standard at m/e 312.

penta-CDFs: individual scans at m/e 340 are summed across a range of  
 observed peaks for total penta CDFs. The area under the  
 resulting mass profile is then ratioed to that of the  
 $^{37}Cl$  2,3,7,8 TCDF internal standard. A response  
 factor of 1 is assumed. The  $^{37}Cl$  penta CDF internal  
 standards were monitored in a 2nd injection of several  
 samples and not used for quantitation.

hexa-CDFs: individual scans at  $m/e$  374 are summed a range of observed peaks for total hexa CDF. The area under the resulting mass profile is then ratioed to that of the  $^{37}\text{Cl}$  2,3,7,8 TCDF internal standard. A response factor of 1 is assumed.

## RESULTS AND DISCUSSION

In general, air samples taken from the 11, 14, 16 and 17th floors of the Binghamton State Office Building were found to contain complex mixtures of tetra-, penta and possibly hexa CDF isomers as shown by the high resolution (HR) single ion chromatograms in figure 1 for floor 11. Tetra-CDFs were predominant on all floors, having concentrations of from 76 to 185  $\text{pg}/\text{m}^3$  total TCDF as given in Table 1. Figures 5-12 are HR single ion chromatograms showing the presence of a mixture of TCDF isomers in all samples (except particulate) from floors 11, 14, 16 and 17. Figures 13 and 14 show a 2378-TCDF-fortified sample and solvent blank. A more complete set of data including HR mass profiles is shown in figs. 17-26 for samples 11C (combined) and 16C1. When interpreting these chromatograms it is important to note that isomer patterns may be distorted as all isomers are not recovered through the sample clean up. The 2,3,7,8 isomer was found in all samples at concentrations of from 0.8 to 23  $\text{pg}/\text{m}^3$ . For floor 17, the glass fiber particulate filter (17C1P, 17C2P) was analyzed separately from the silica gel adsorbent cartridge (17C1G, 17C2G). The entire mixture of TCDFs was found on the adsorbent and little or none in the particulate section ( $>.3 \mu$ ) of the sample trap (figs. 8-10, 15-16).

Penta CDFs, which were more abundant than tetra CDF in the original soot, were found in all non-particulate air samples however at lower concentrations of from 6.6 to 24.4  $\text{pg}/\text{m}^3$  (Table 2). A possible explanation for this is the lower volatility of penta compared to tetra CDFs. High resolution single ion chromatograms and mass profile data for penta CDFs in samples 11B3 + 11B4 + 11C1, 17C1G, 17C1P, and a solvent blank are shown in figures 27-30. A typical 2nd injection of a sample to acquire data on hexa (and penta) CDFs is shown for sample 11B3 + 11B4 + 11C1 in figures 31-34. No hexa CDF could be confirmed at a detection limit of approximately 5  $\text{pg}/\text{m}^3$ .

To obtain adequate sensitivity for TCDDs, four 50  $\text{m}^3$  BSOB air samples were extracted and combined to form a single sample prior to clean up and analysis. The SP2330 capillary GC/HRMS results for a combined 11th floor air sample (analyzed in duplicate), a solvent blank, and a background air sample fortified to 0.82  $\text{pg}/\text{m}^3$  with native 2,3,7,8 TCDD are given in Table 3. Because only a small amount of sample 11C6 + 11C7 + 11C8 + 11C10 was injected, the signal/noise for that sample was weak (a wide peak was also observed, hence insignificant concentration). The HR single ion chromatograms and mass profiles for a combined BSOB air sample 11C1 + 11C2 + 11C4 + 11C5 (figs. 35-37) showed 226  $\text{pg}$  2,3,7,8 TCDD equivalent to 0.94  $\text{pg}/\text{m}^3$  air (also possible traces of other TCDDs). A quality control (120  $\text{m}^3$  Albany air) sample fortified with 98  $\text{pg}$  native 2,3,7,8 TCDD to 0.82  $\text{pg}/\text{m}^3$  air was analyzed and found to have a concentration of 1.3  $\text{pg}/\text{m}^3$ . A solvent blank was similarly analyzed and found to contain a rather high 47  $\text{pg}$  2,3,7,8 TCDD equivalent to 0.39  $\text{pg}/\text{m}^3$  air.



## CONCLUSIONS

Analysis of air samples for a complex mixture of PCDDs and PCDFs at concentrations in the parts per quadrillion range had never been attempted prior to these studies. Although considerable progress has been made in the development and application of the necessary analytical techniques, several problems remain unresolved. The concentration of 2,3,7,8-TCDD in the air of the BSOB could not be accurately determined in this group of samples due to the presence of very low concentrations of interfering compounds in the blank. It is clear, however, that the concentration of 2,3,7,8-TCDD is below  $1.5 \text{ pg/m}^3$ , consistent with an earlier report from the laboratory. A second problem relates to the lack of labelled quantitative PCDF standards (for congeners other than 2,3,7,8-TCDF). Quantitative analysis of a congener without an internal standard is difficult, since recoveries are not accurately known. In the present instance, when even unlabelled standards of the range of compounds sought are unavailable, and certain congeners are demonstrably poorly recovered, the difficulty is even more acute. Thus, calculations of "total TCDF" and "total PCDF" may underestimate actual concentrations of these mixtures. Attempts will be made to further address these problems in future analyses.

Nevertheless, these data permit some important conclusions regarding the PCDD/PCDF concentrations in the BSOB. The concentration of 2,3,7,8-TCDF measured in six locations varied from  $9.9$  to  $23 \text{ pg/m}^3$ . The ratio of tetra CDF to penta and hexa CDF is clearly higher in these air samples than

in the soot samples analyzed earlier. Coupled with the observations that the bulk of both 2,3,7,8-TCDF and "total TCDF" was found in the silica gel cartridge rather than in the particulate material, this result suggests that these compounds are predominantly in the gas phase. Finally, these experiments confirm that 2,3,7,8-TCDD is present at concentrations well below those of 2,3,7,8-TCDF in these air samples.

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Table 1. TETRA-CDFs

Floor/sample <sup>①</sup>	Volume m <sup>3</sup>	Concentration of 2378 TCDF (pg/m <sup>3</sup> )	Detection limit (pg/m <sup>3</sup> )	Ion <sup>②</sup> ratio(%) 304/306	Relative retention time	<sup>37</sup> Cl TCDF recovery (%)	Total TCDF (pg/m <sup>3</sup> )	Detection limit (pg/m <sup>3</sup> )	Ion <sup>②</sup> ratio(%) 304/306	Ratio 2378/total (%)
14 C1	61.5	11	0.4	78	1.0	97	92	0.8	83	12
14 A3	63.0	14	0.3	73	1.0	157	185	0.3	72	8
16 C1	62.4	16	0.4	74	1.0	88	118	2.7	77	14
17 C1G	62.1	12	1.7	84	1.0	26	79	5	89	15
17 C1P	62.1	0.8	0.7	70	1.0	78	3.9	2.7	89	21
17 C2G	59.8	9	0.3	78	1.0	71	59	4	73	15
17 C2P	59.8	0.9	0.4	126	1.0	74	ND	5	92	—
11 B3 + 11 B4 + 11 C1	184.5	16	0.5	84	1.0	41	133	3.7	78	12
11 C1 + 11 C2 + 11 C4 + 11 C5	240.8	23	0.3	80	1.0	42	76	4	73	30
Solvent blank	0	ND	0.2	—	—	110	ND	1.9	—	—
DOH2 background air	49.1	1.9	0.6	68	1.0	74	9.5	2.5	110	20
NSA 6S 10.0 pg/m <sup>3</sup> 2378 TCDF Fortified back- ground air	41.8	9.9	0.4	79	1.0	120	10.2	2.1	—	97
DOH1 8.1 pg/m <sup>3</sup> 2378 TCDF Fortified back- ground air	51.6	10.0	1.2	80	1.0	75	16.8	2.0	90	60

① e.g. 14A3 is 14th floor, N East building corner, 3rd sample taken on that date.

② Theoretical ion ratio = 78%.

③ Recovery variation (vs. external standard) is probably due to the volume measurement and manipulation of small (5-10  $\mu$ L) volumes prior to injection.

Table 2 PENTA CDFs

<u>Floor/Sample</u>	<u>Total Penta-CDF (pg/m<sup>3</sup>)</u>	<u>Detection Limit (pg/m<sup>3</sup>)</u>	<u>Ion<sup>②</sup> ratio (%) 338/340</u>
14C1	20.6	2.4	
14A3	12.7	1.6	
16C1	21.2	1.2	
17C1G	24.4	7.4	
17C1P	ND	2.0	
17C2G <sup>①</sup>	6.6	1.3	0.78
17C2P	2.9	2.1	0.23
11B3 + 11B4 +11C1	19.2	1.5	0.62
11C1 + 11C2 +11C4 + 11C5	16.4	2.3	
Solvent blank	ND	1.3	
DOH2 background	ND	2.0	

① Recovery of three <sup>37</sup>Cl Penta CDF internal standard isomers was calculated to be 80%, 91% and <10% for sample 17C2G (other samples were similar).

② Multiple ions were checked on a 2nd injection for several samples.

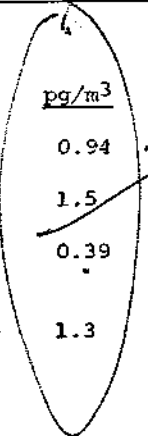
*Penta*  
13 pg/m<sup>3</sup>

Table 3

Floor/sample	Air volume (m <sup>3</sup> )	m/e 322 area (counts)	pg found	pg/m <sup>3</sup>	Detection limit (pg)	Ratio <sup>②</sup> 320/322	<sup>13</sup> C TCDD recovery (%)	GC relative retention time
11C1 + 11C2 + 11C4 + 11C5	241	1040165	226	0.94	12	0.66	90	1.00
11C6 + 11C7 + 11C8 + 11C10	241	529026	372	1.5	29	0.84	58	1.00
Solvent blank 2 + 3	0 (120 for calcs.)	296261	47	0.39	17	0.60	112	1.00
DOH 3 + 4 background air fortified with 98 pg native TCDD (=0.82 pg/m <sup>3</sup> )	120	706423	150	1.3	13	0.89	78	1.00

~~0.9~~  
 0.9  
 $\frac{48}{1.2}$   
 $1.22 \times \frac{48}{1.2}$   
 $= 48 \text{ pg/m}^3$   
 $33 \text{ pg/m}^3$  corrected

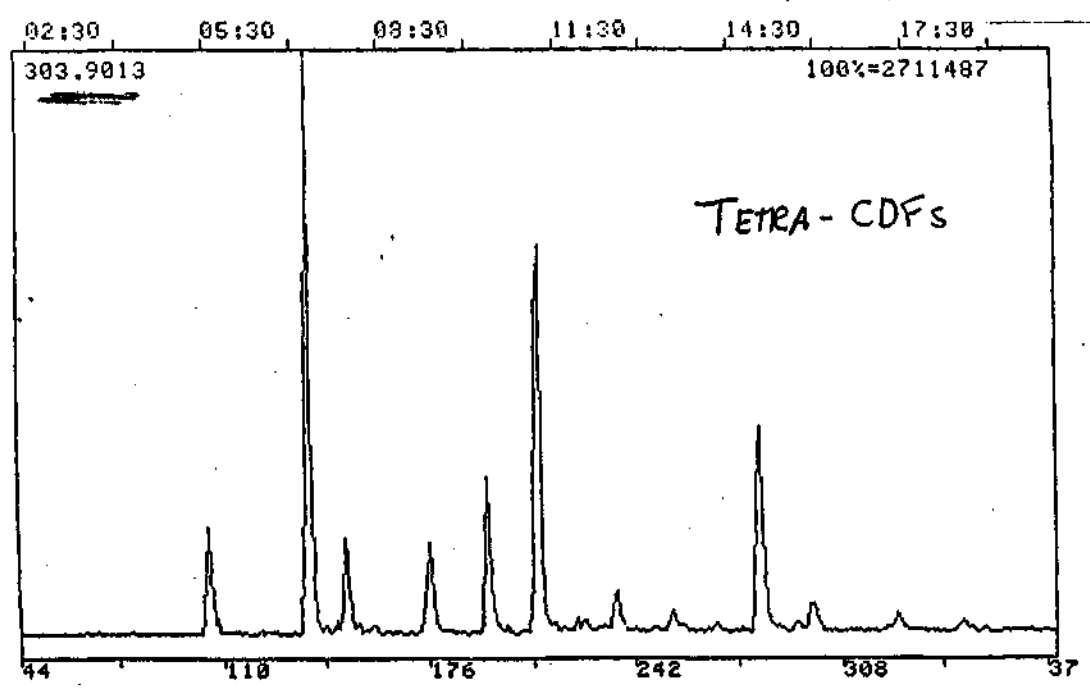
2378 TCDD<sup>①</sup>



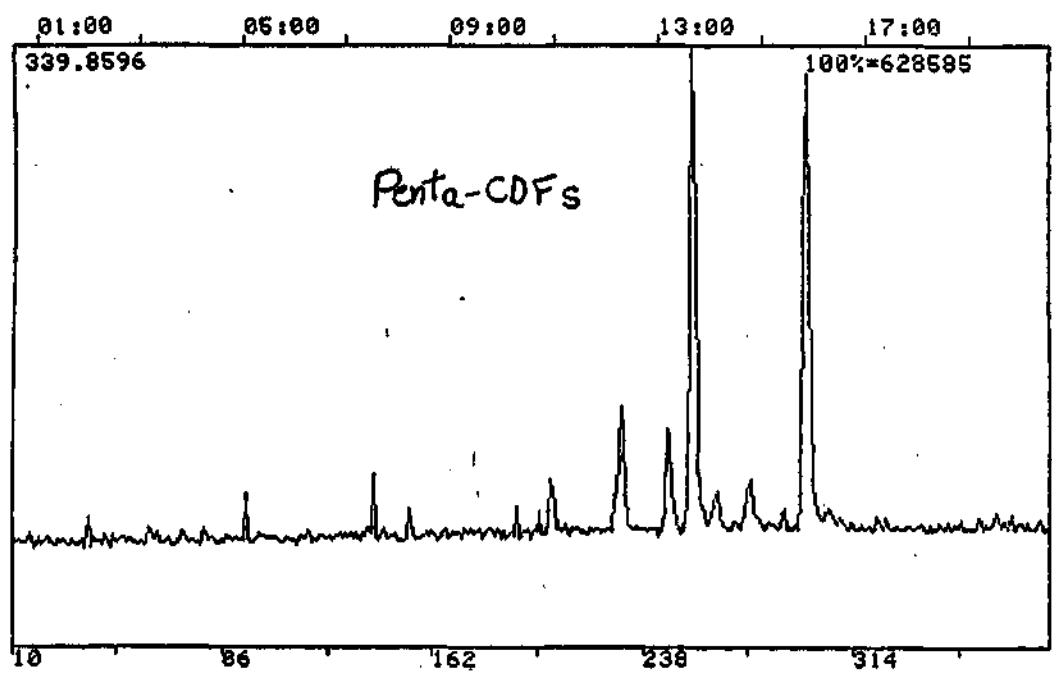
① Both BSOB air samples appeared to contain small amounts of several other TCDD isomers as evidenced by a simultaneous response at other retention times for m/e 319.8964 and 321.8935; however, additional analysis is needed for verification.

② Theoretical Ratio = 0.78

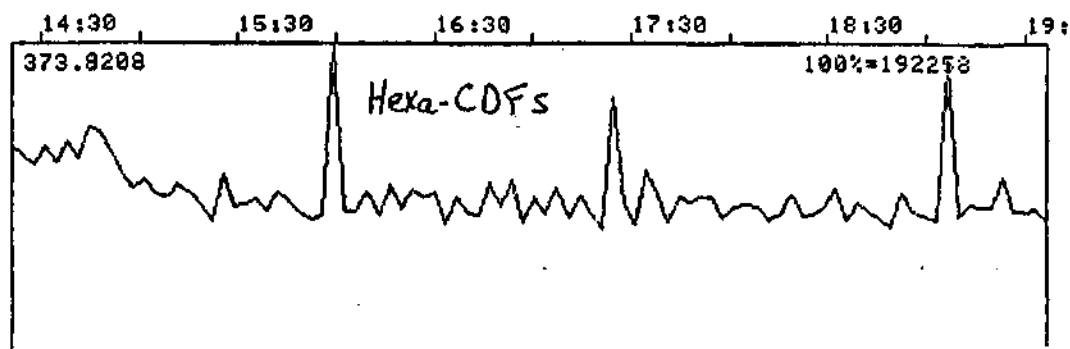
RETN  
TIME



RETN  
TIME

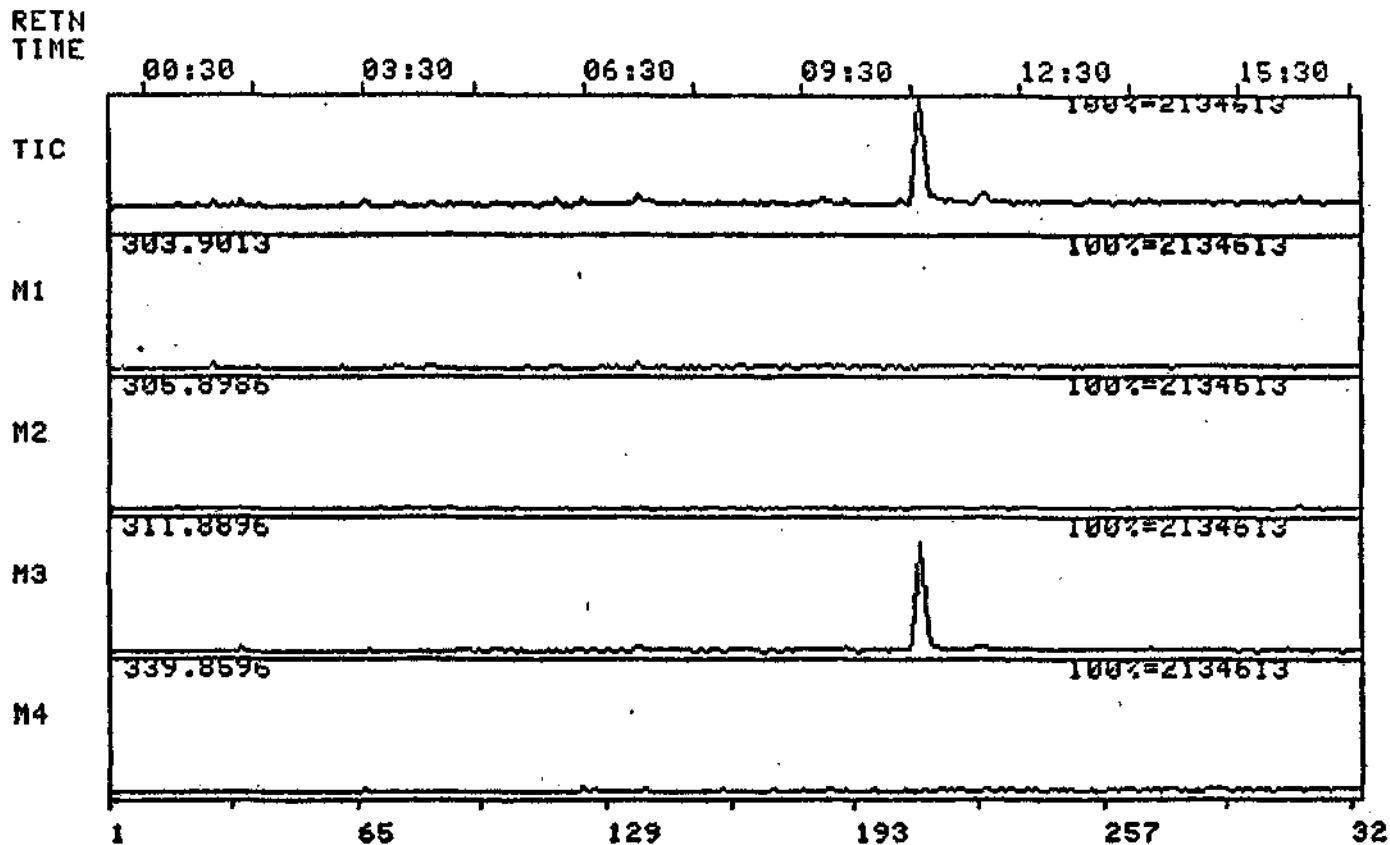


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TIME

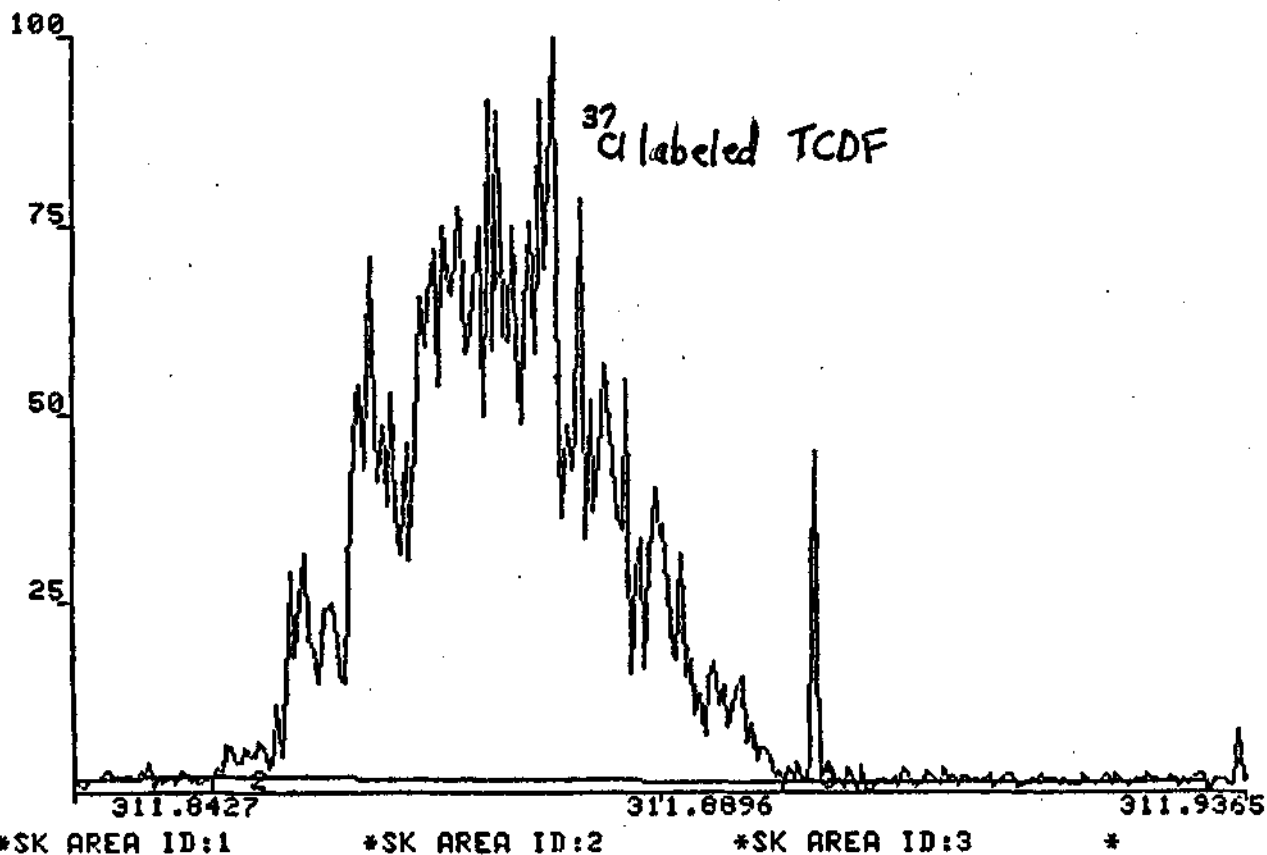


DSS5 HIGH RESOLUTION MPM  
RUNNAME MDHM4 DATE 3/28/83 TIME

Figure 2: mixed labeled standard  
ions for native tetra + penta CDF  
and labeled TCDF



RUNNAME MDHM4 DATE 3/28/83 TIME 17: 7  
MASS 311.8896 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 205-215 100% INTENSITY 103419



\*SK AREA ID:1

\*SK AREA ID:2

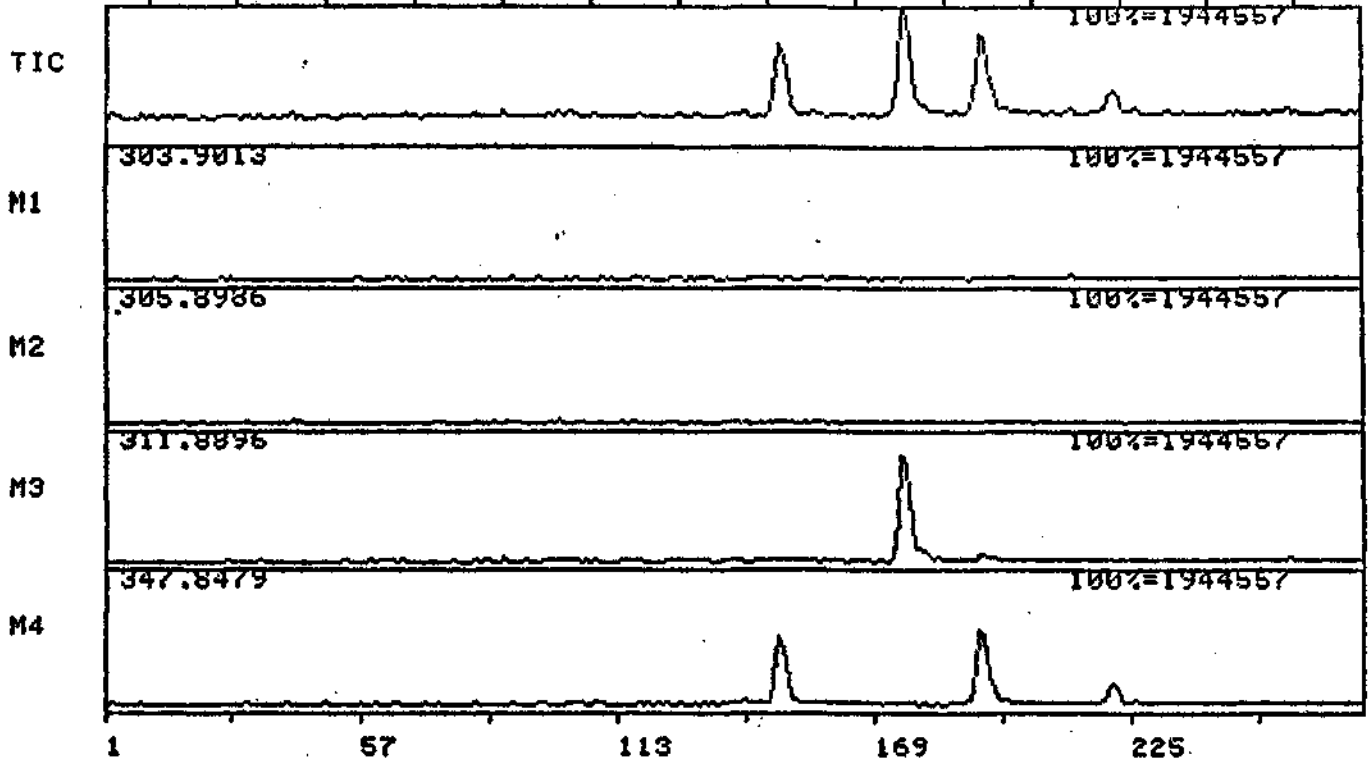
\*SK AREA ID:3

\*



RETN  
TIME

00:30 02:30 04:30 06:30 08:30 10:30 12:30



RUNNAME MDHM1 DATE 3/28/83 TIME 13: 0  
MASS 347.8479 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 94-231 100% INTENSITY 203331

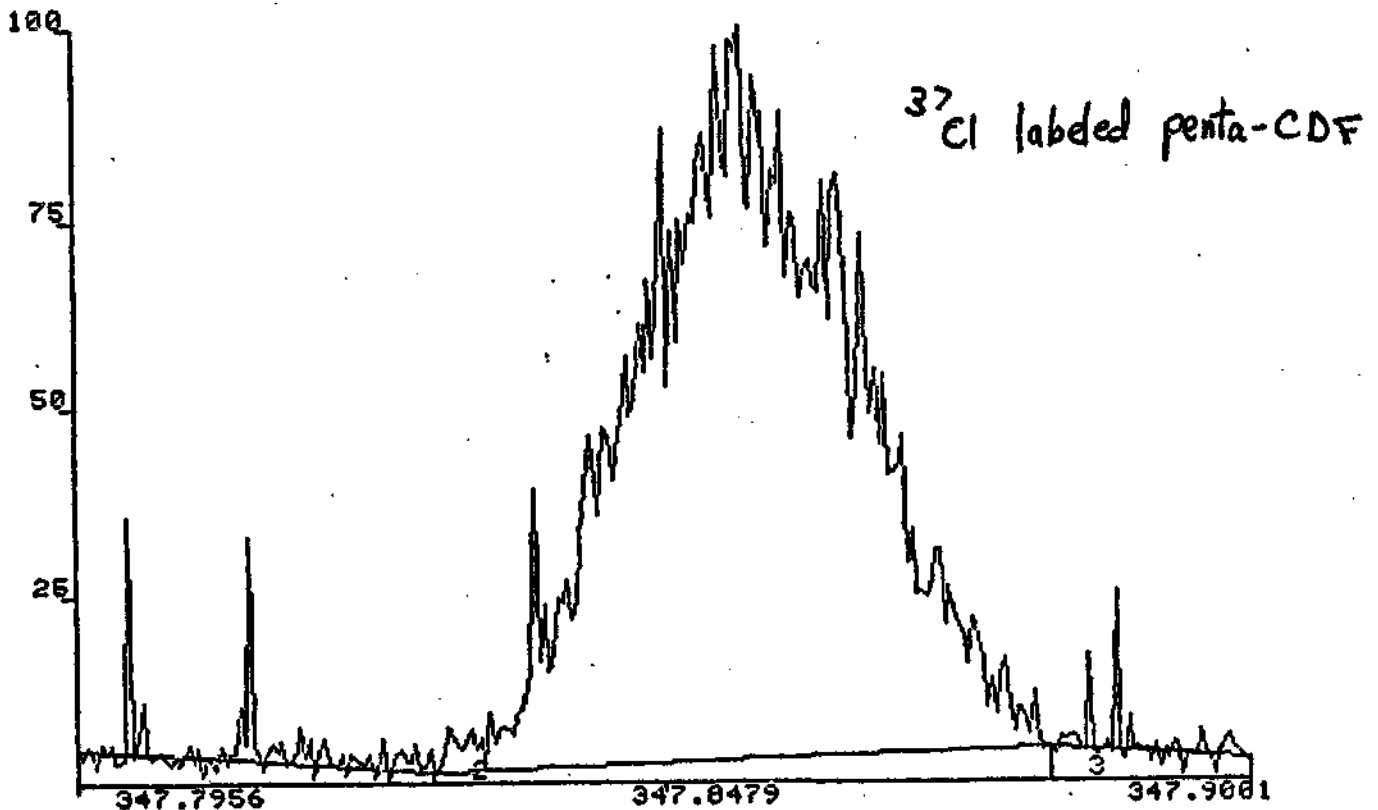
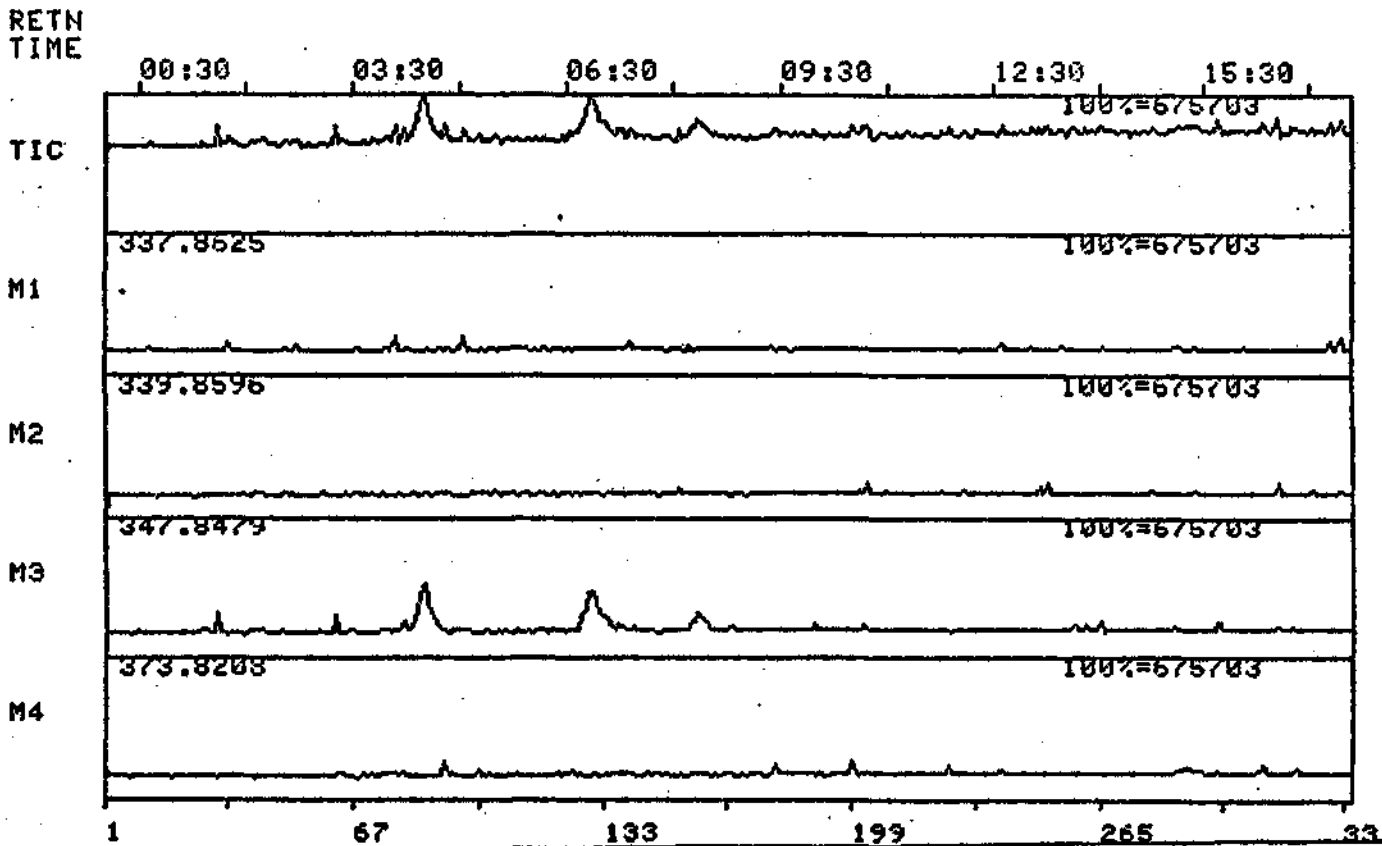
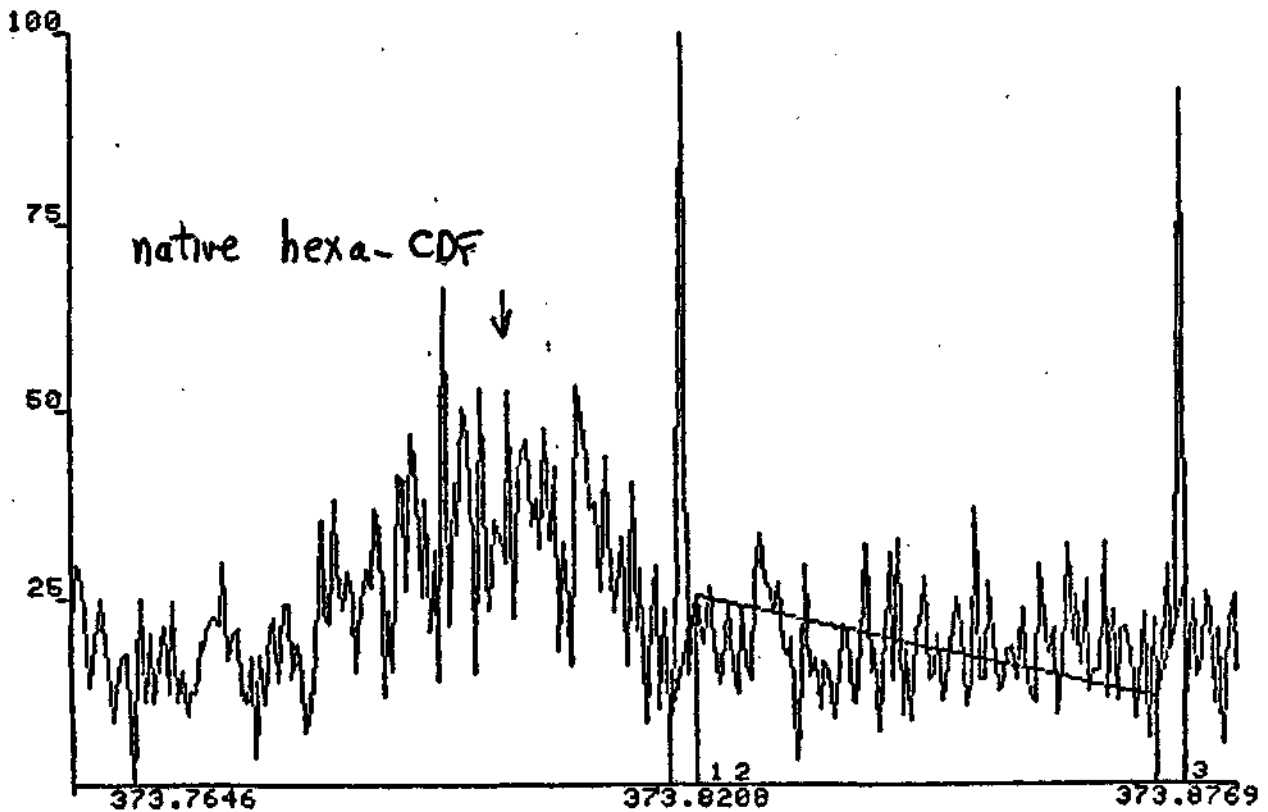


Figure 4: mixed labeled standard + native hexa CDF added

DS55 HIGH RESOLUTION MPM  
RUNNAME MDHM2 DATE 4/ 4/83 TIME 8:28



RUNNAME MDHM2 DATE 4/ 4/83 TIME 8:28  
MASS 373.8208 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 282-297 100% INTENSITY 17744

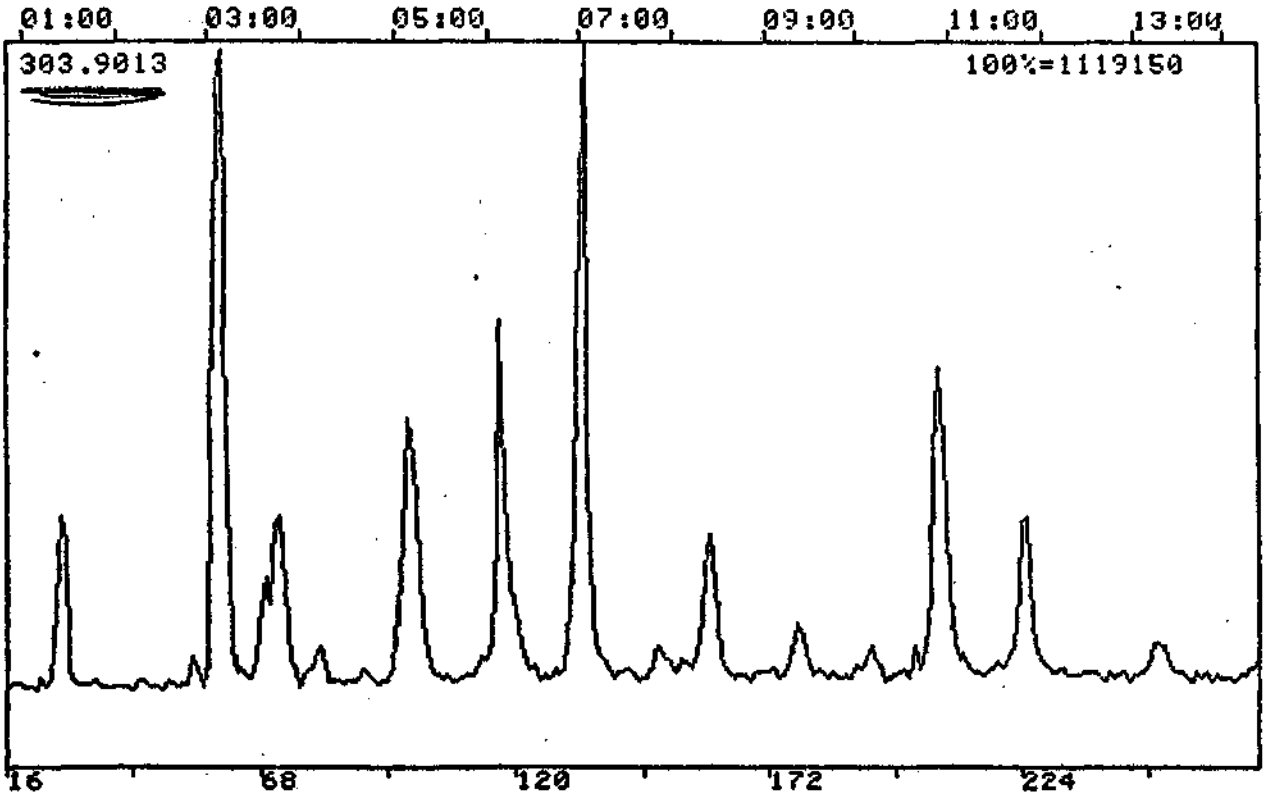


D555 HIGH RESOLUTION MPM  
RUNNAME TUDHM6

DATE 3/29/83

TIMI Figure 5: Tetra CDFs in Floor 14  
Sample 14C1

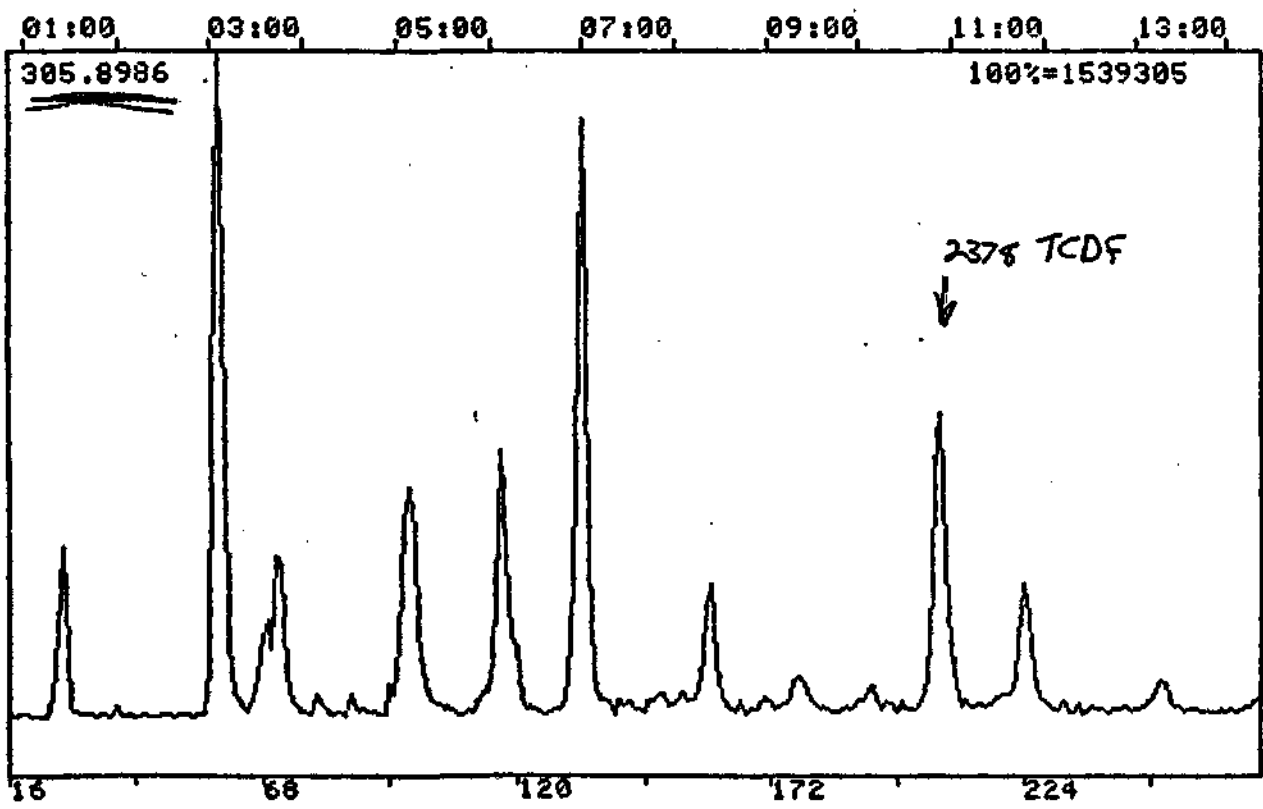
RETN  
TIME



\*  
D555 HIGH RESOLUTION MPM  
RUNNAME TUDHM6 DATE 3/29/83 TIME 12:47

14C1

RETN  
TIME

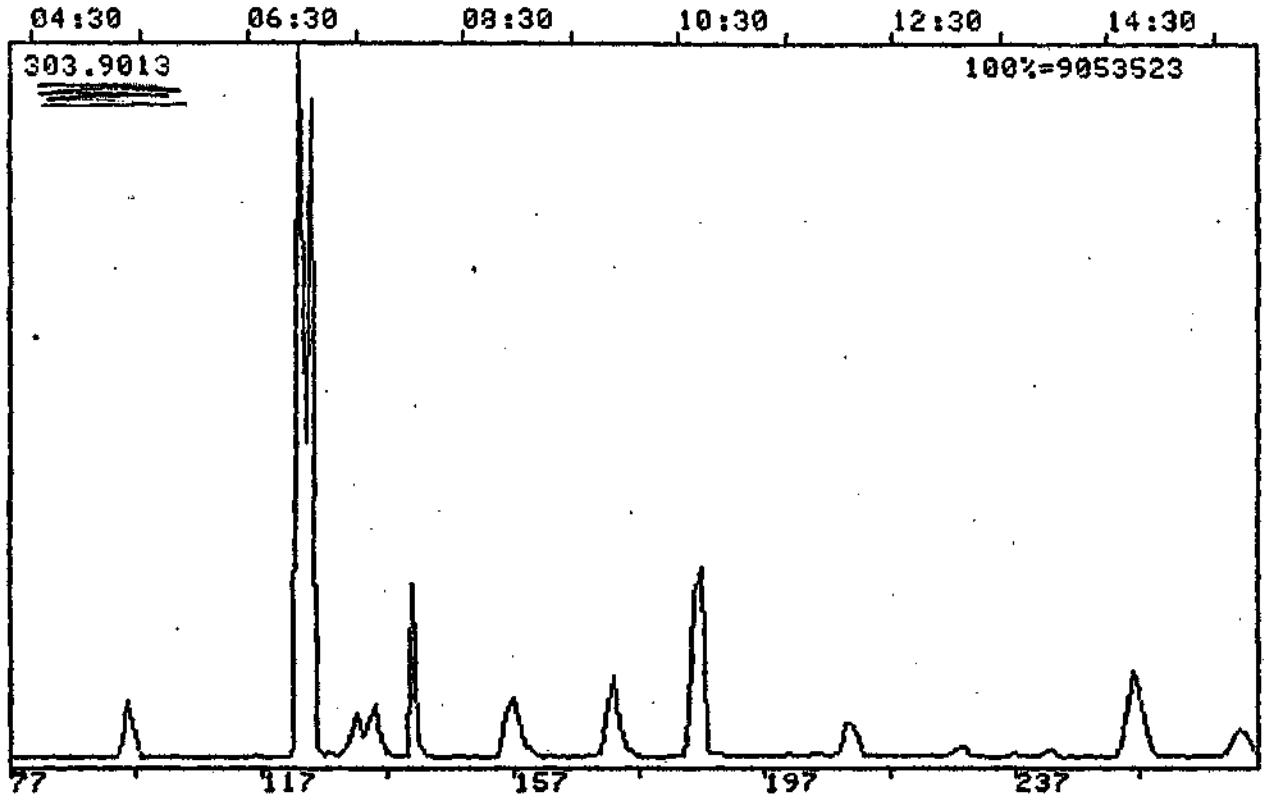


DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHM9

DATE 3/29/83

TIME Figure 6: tetra CDFs in Floor 14  
Sample 14A3

RETN  
TIME



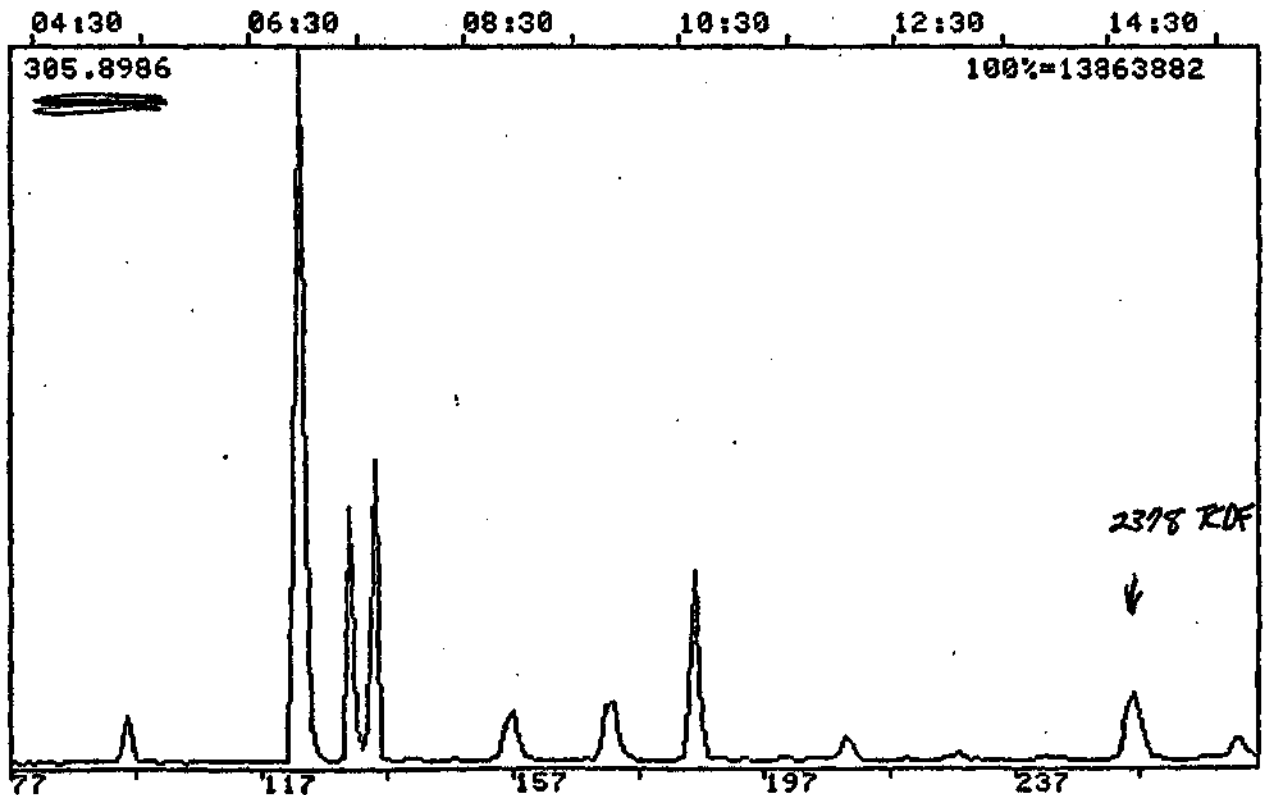
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DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHM9

DATE 3/29/83

TIME 15:44

RETN  
TIME

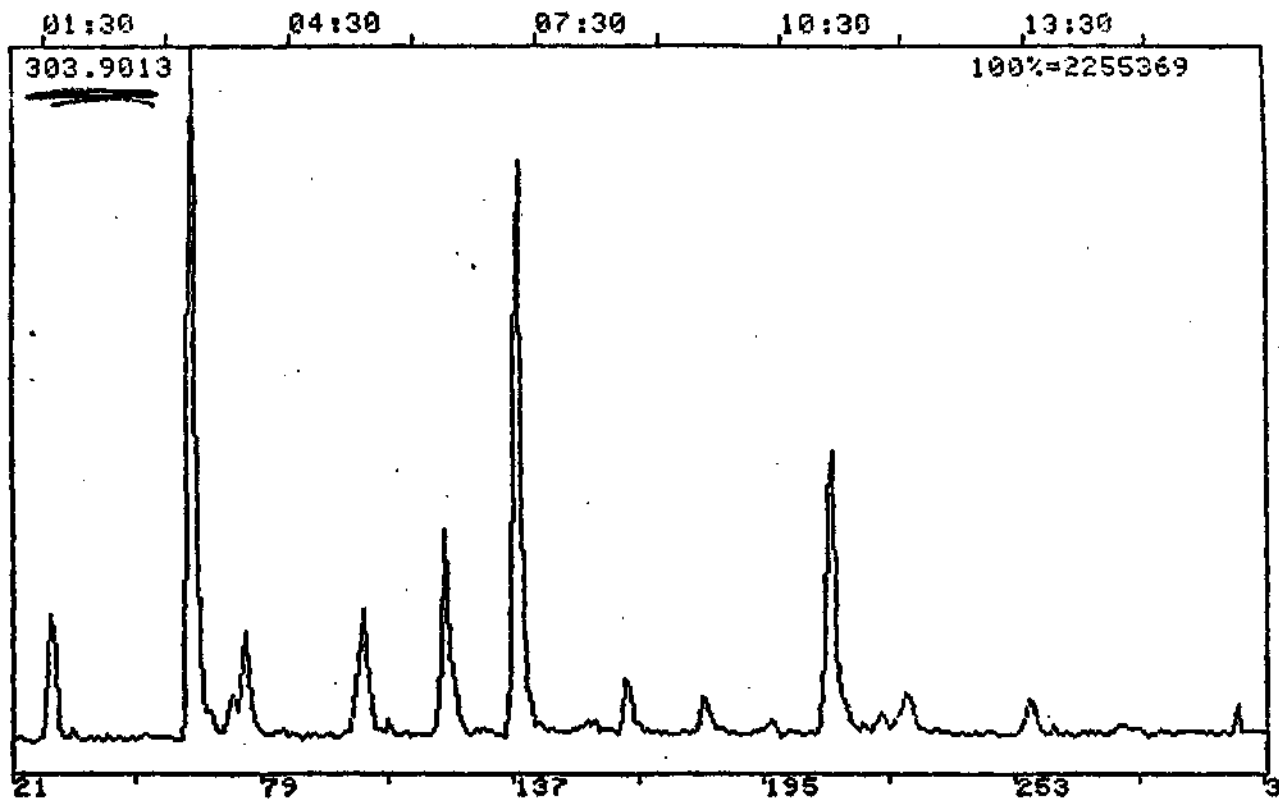


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DS55 HIGH RESOLUTION MPM  
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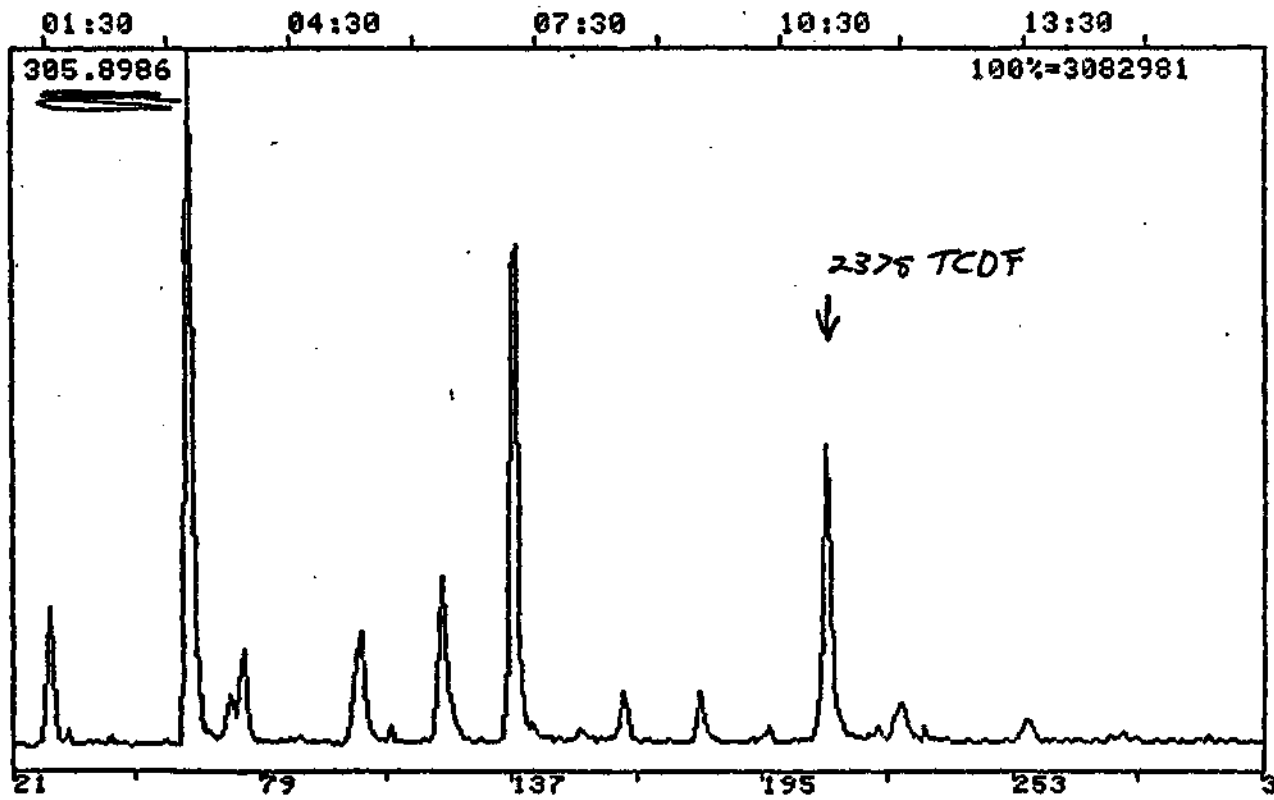
TIME Figure 7: Tetra CDFs in Floor 16  
Sample 16 C1

RETN  
TIME



DS55 HIGH RESOLUTION MPM  
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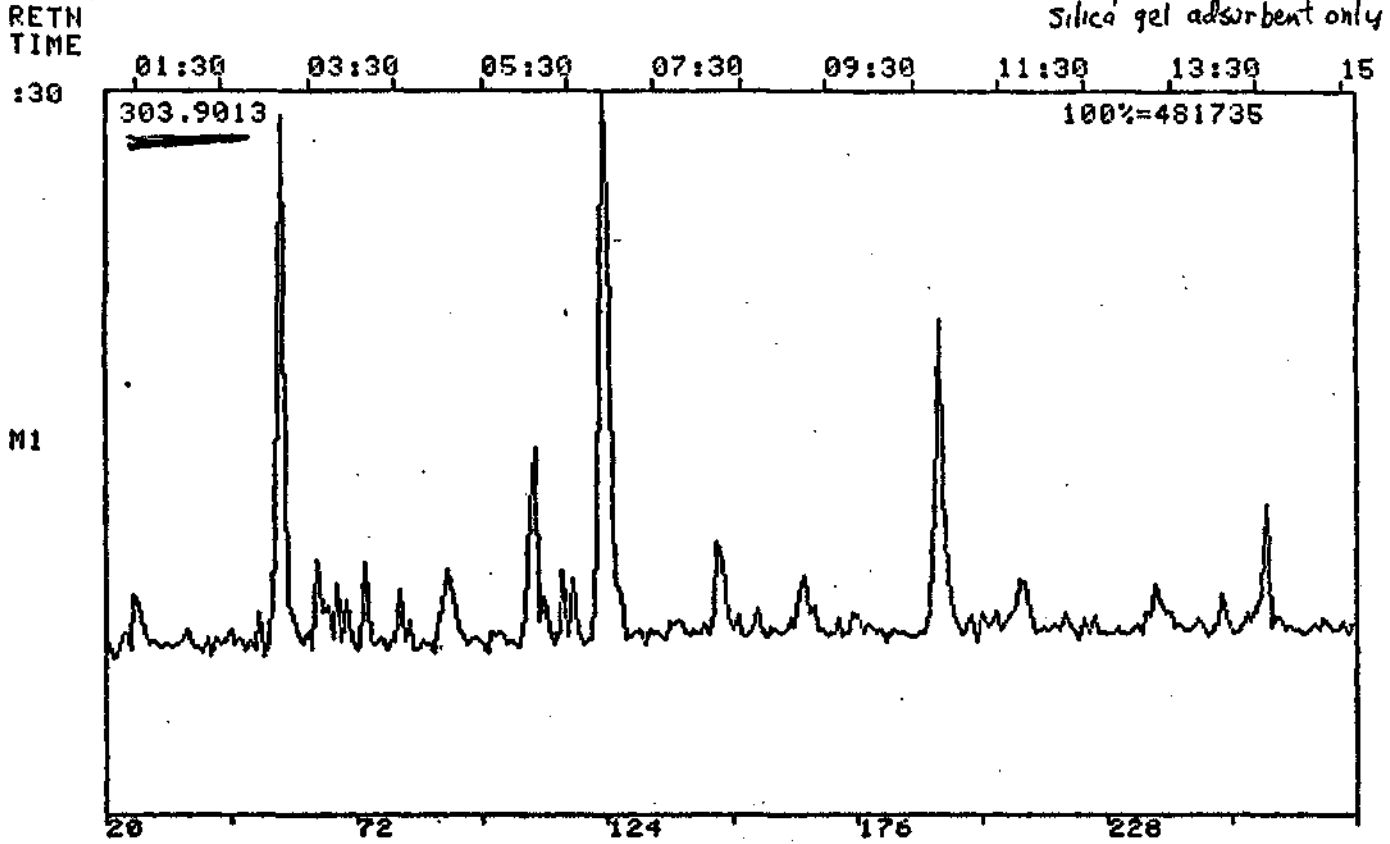
RETN  
TIME



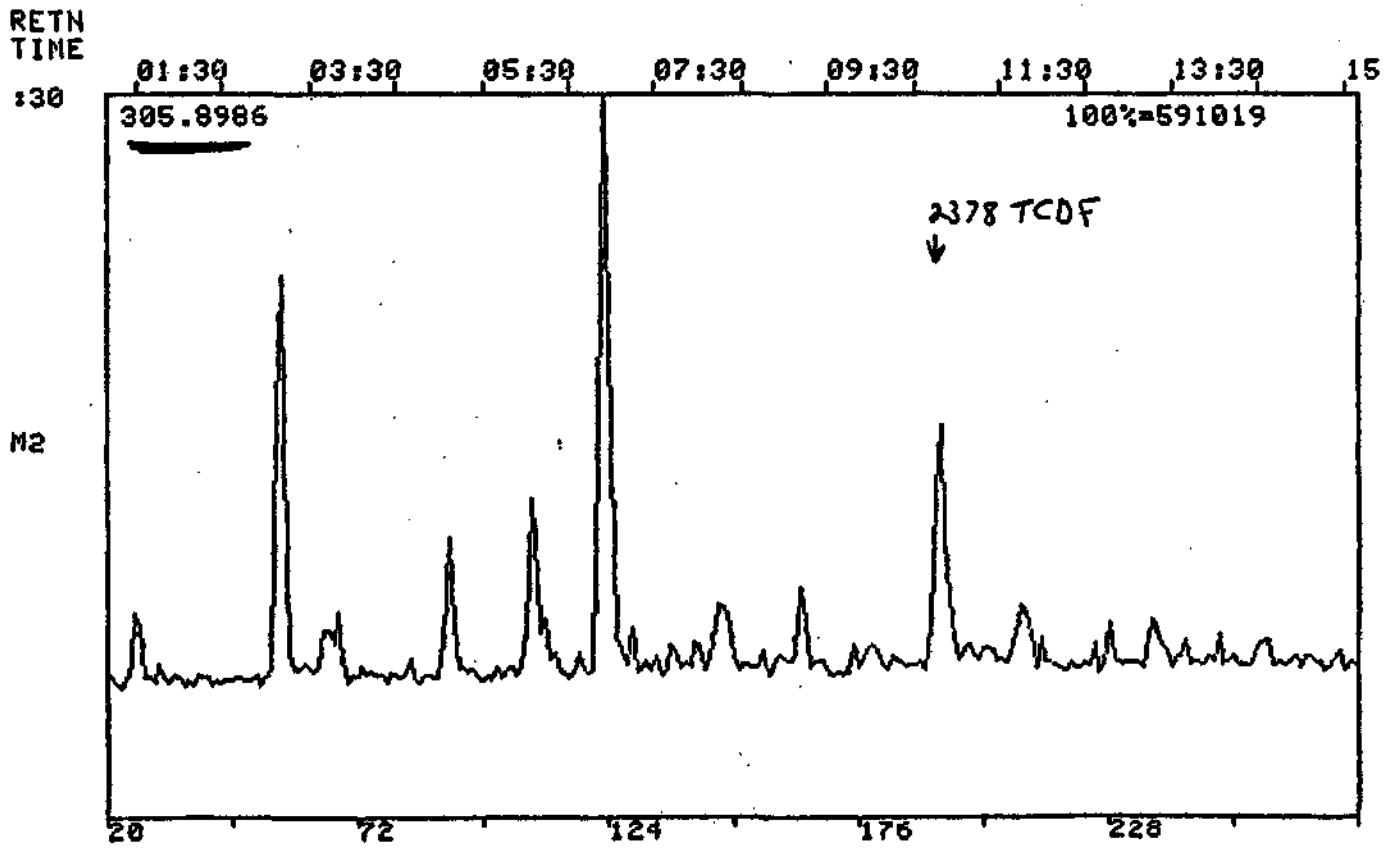
DS55 HIGH RESOLUTION MPM  
RUNNAME MDHMS DATE 3/28/83 TIME

Figure 8: tetra COFs in Floor 17

Sample 17C1 G  
Silica gel adsorbent only

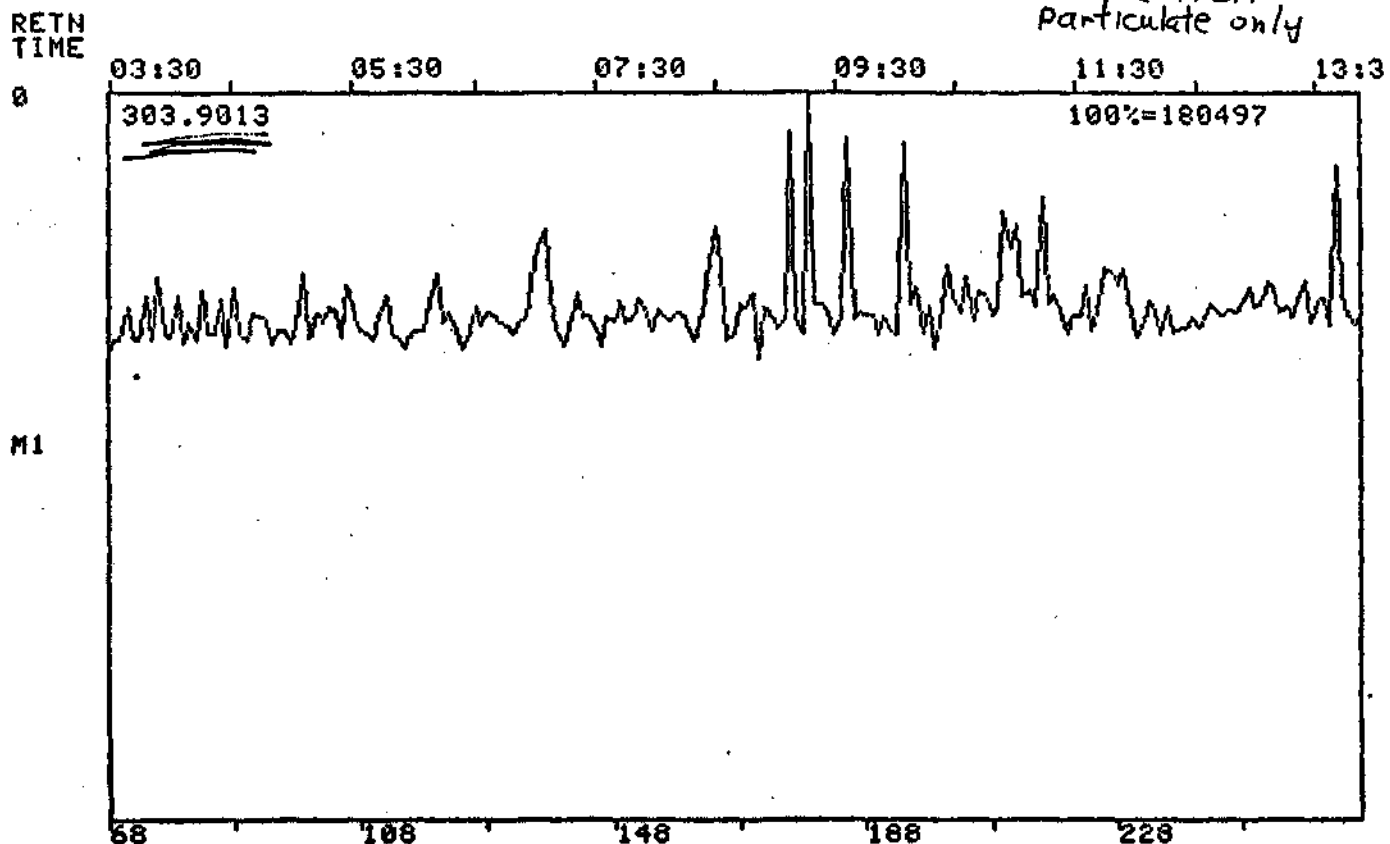


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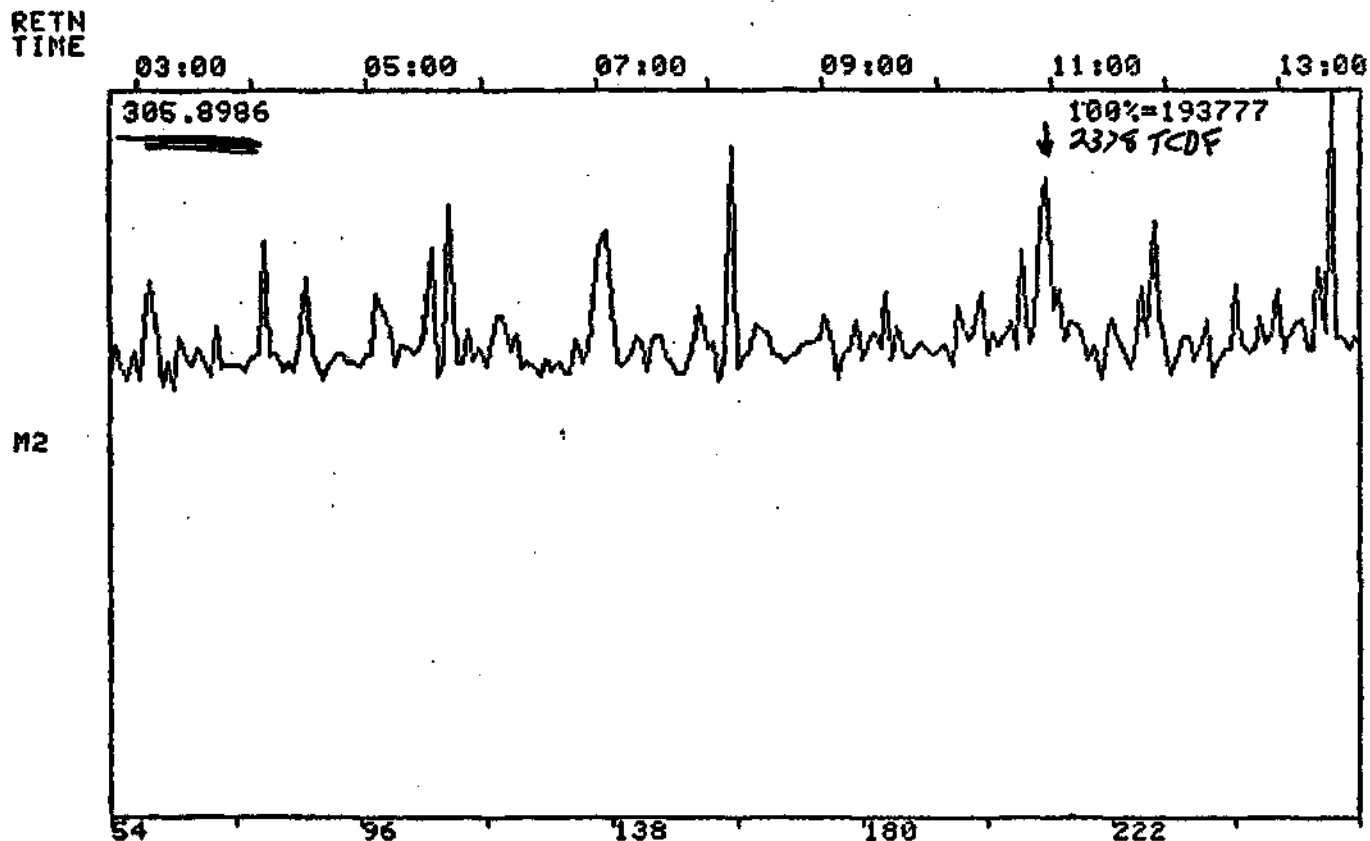


DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHM4 DATE 3/29/83

TIME Figure 9: tetra CDFs in Floor 17  
Sample 17CIP  
particulate only



DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHM4 DATE 3/29/83 TIME 10:29



DS55 HIGH RESOLUTION MPM  
RUNNAME TDHM10

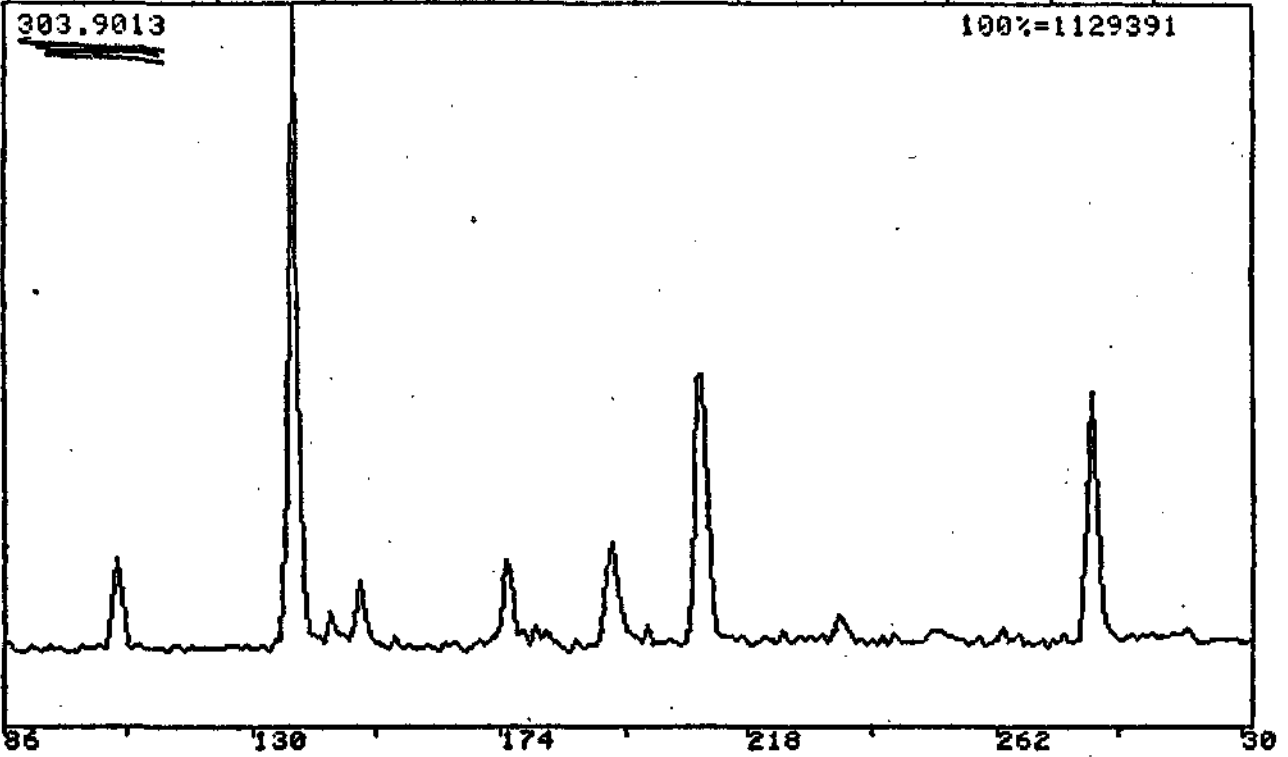
DATE 3/29/83

TIME Figure 10: tetra CDFs in Flour 17

Sample 17C26  
Silica gel adsorbent only

RETN  
TIME

04:30 06:30 08:30 10:30 12:30 14:30



M1

6

DS55 HIGH RESOLUTION MPM  
RUNNAME TDHM10

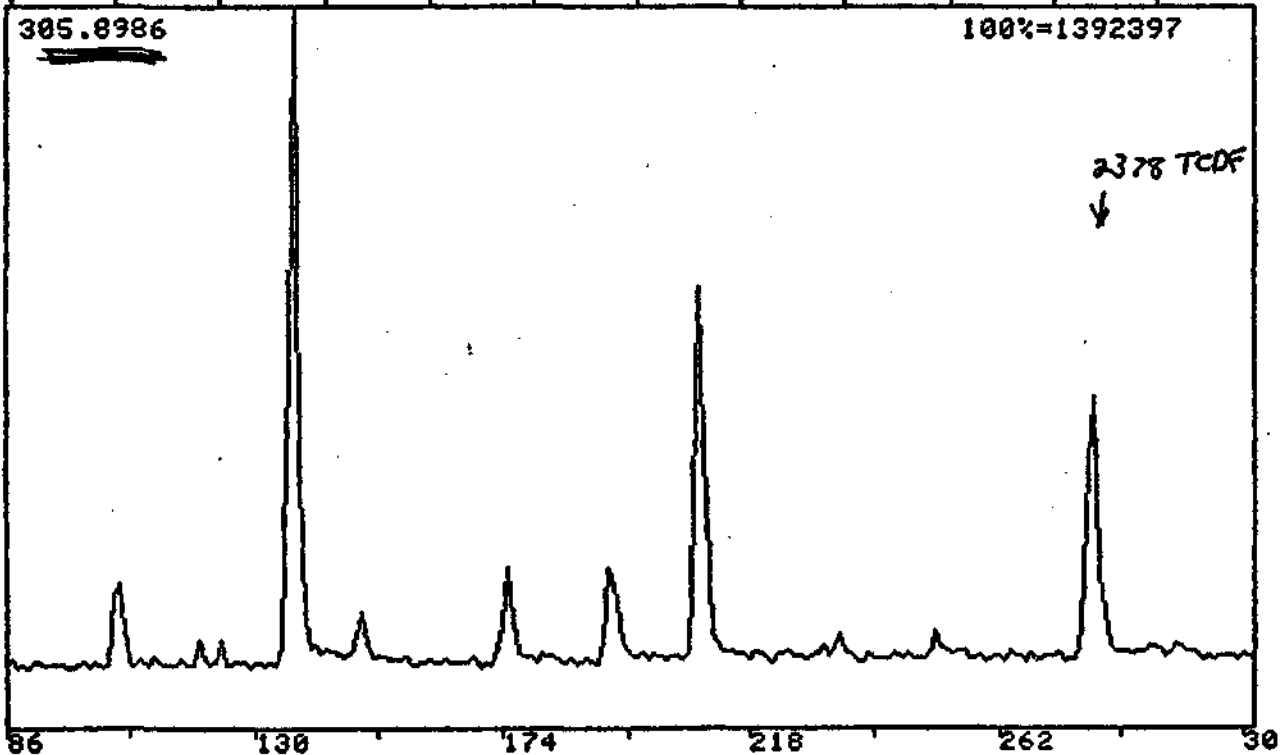
DATE 3/29/83

TIME 16:57

17C26

RETN  
TIME

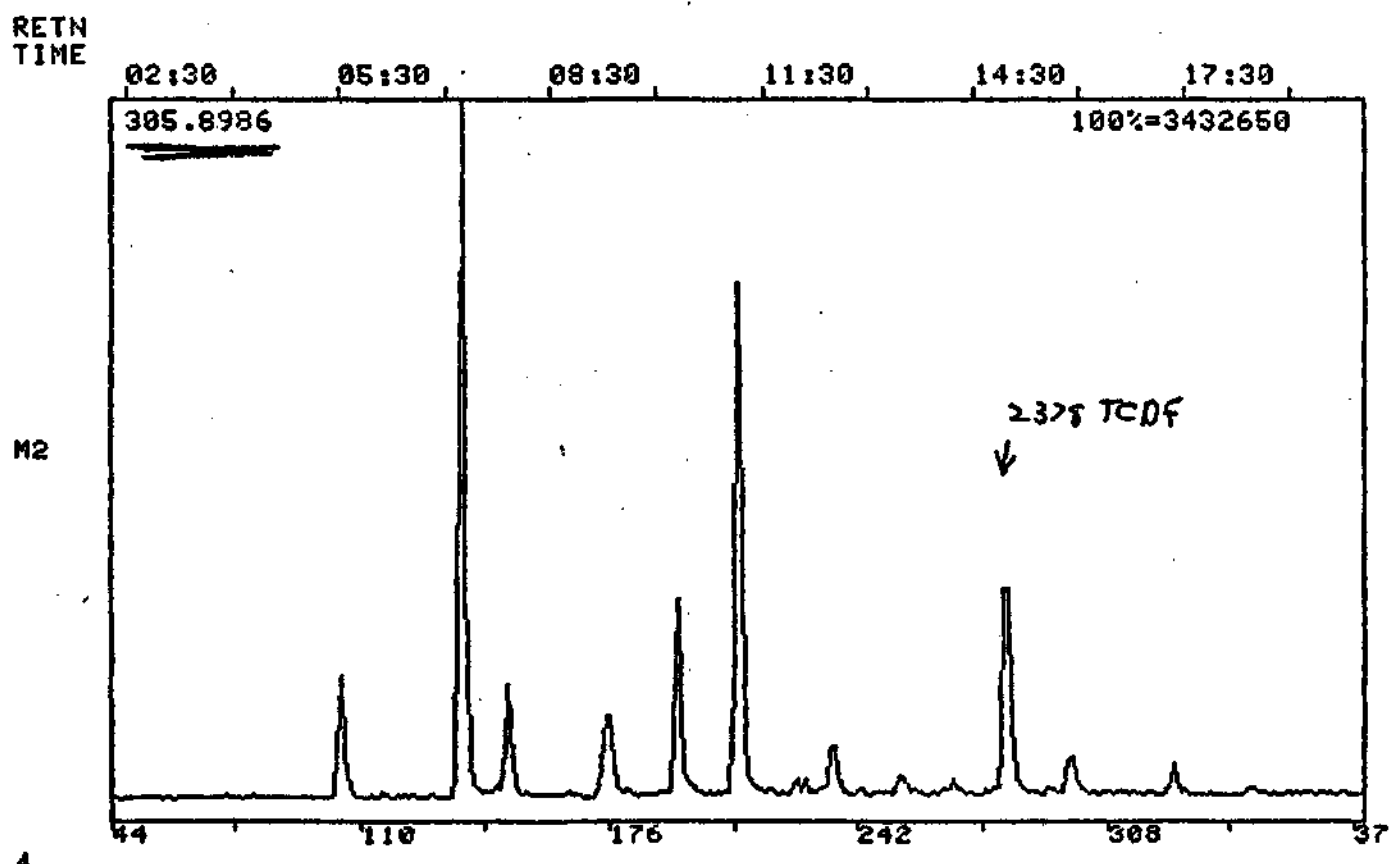
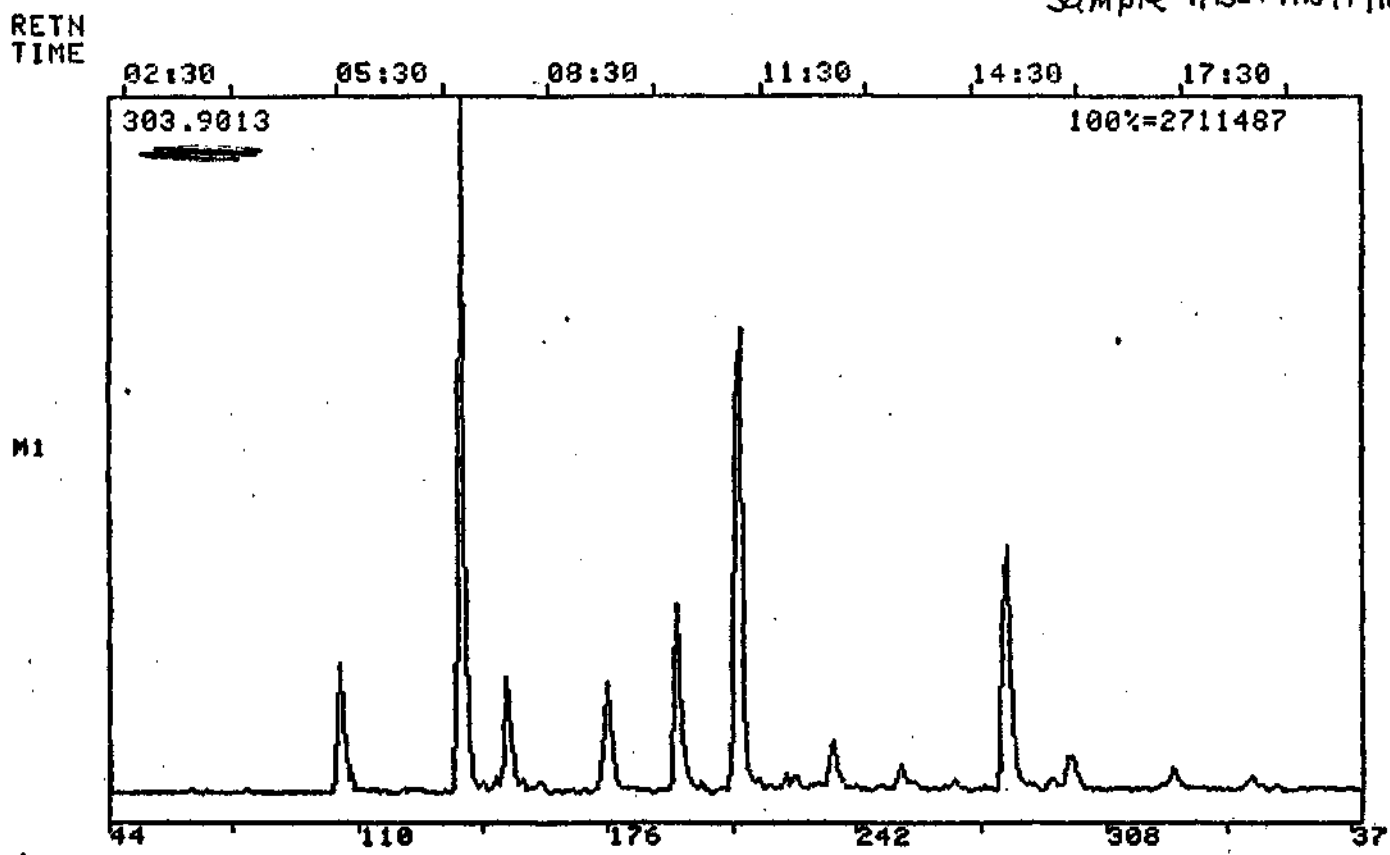
04:30 06:30 08:30 10:30 12:30 14:30



M2

6





DS55 HIGH RESOLUTION MPM  
RUNNAME TDHMS DATE 3/31/83

TIME : Figure 12: tetra CDFs in Floor 11  
Sample 11C1+11C2+11C4+11C5

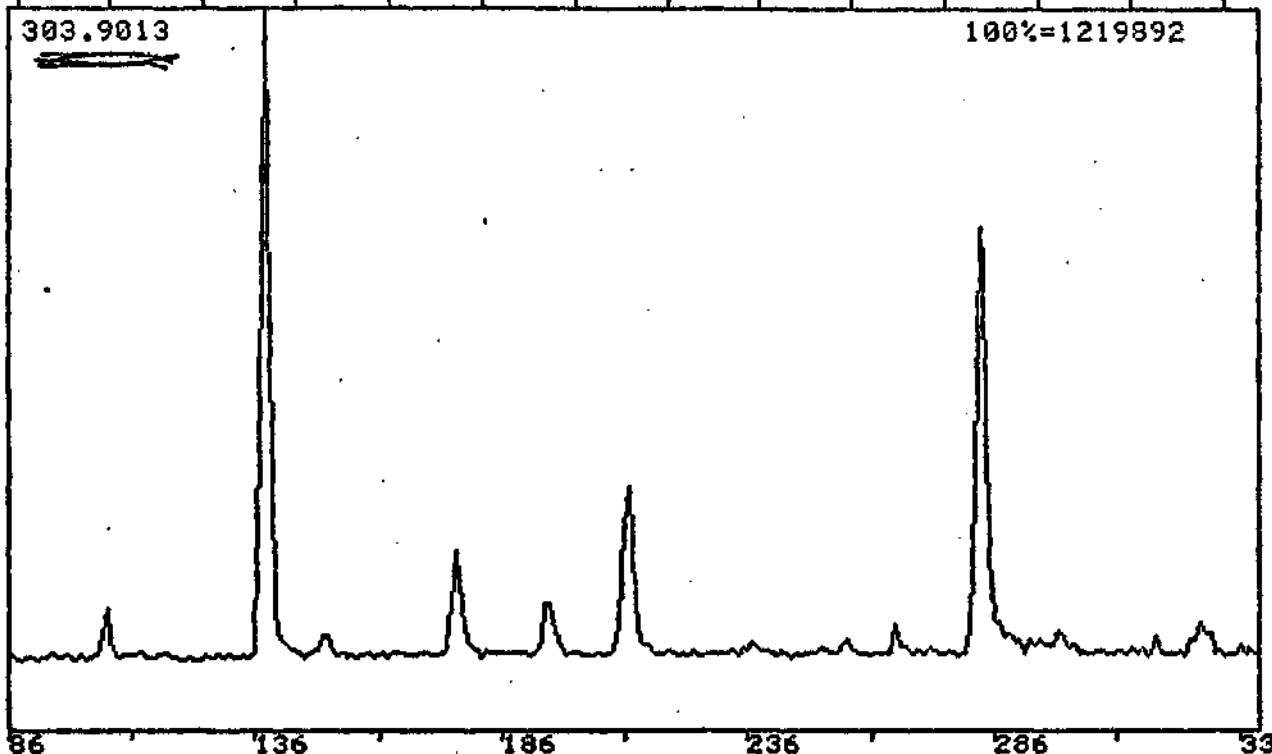
RETN  
TIME

04:30 06:30 08:30 10:30 12:30 14:30 16:30

303.9813

100%=1219892

M1



DS55 HIGH RESOLUTION MPM  
RUNNAME TDHMS DATE 3/31/83 TIME 11:31

RETN  
TIME

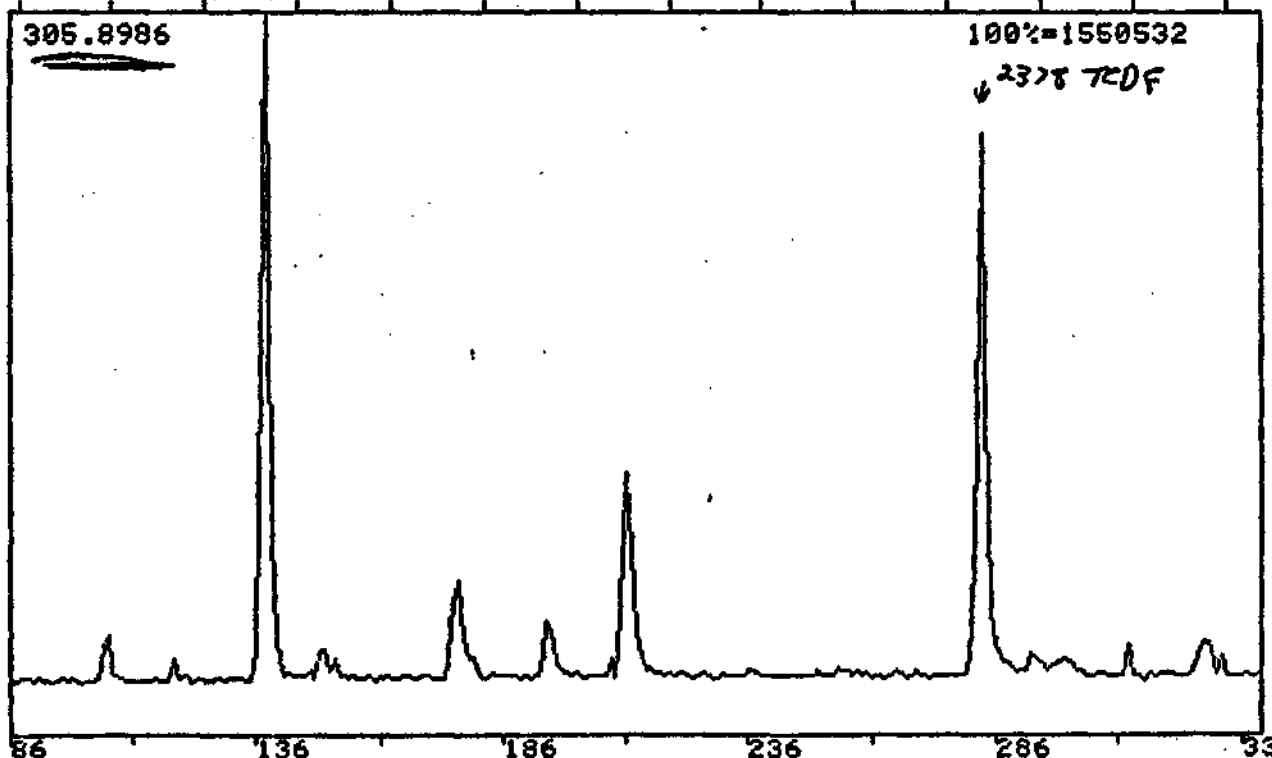
04:30 06:30 08:30 10:30 12:30 14:30 16:30

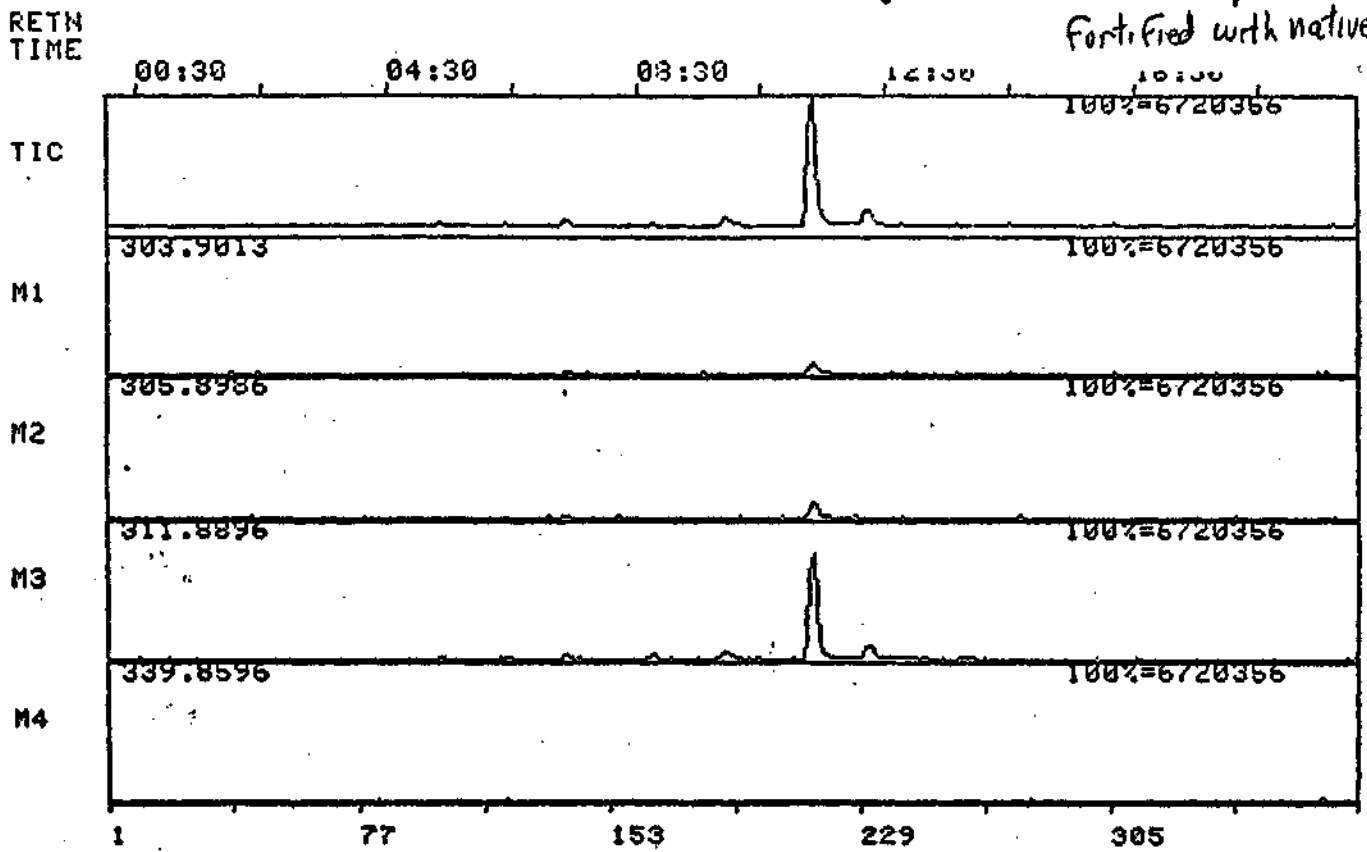
305.8986

100%=1550532

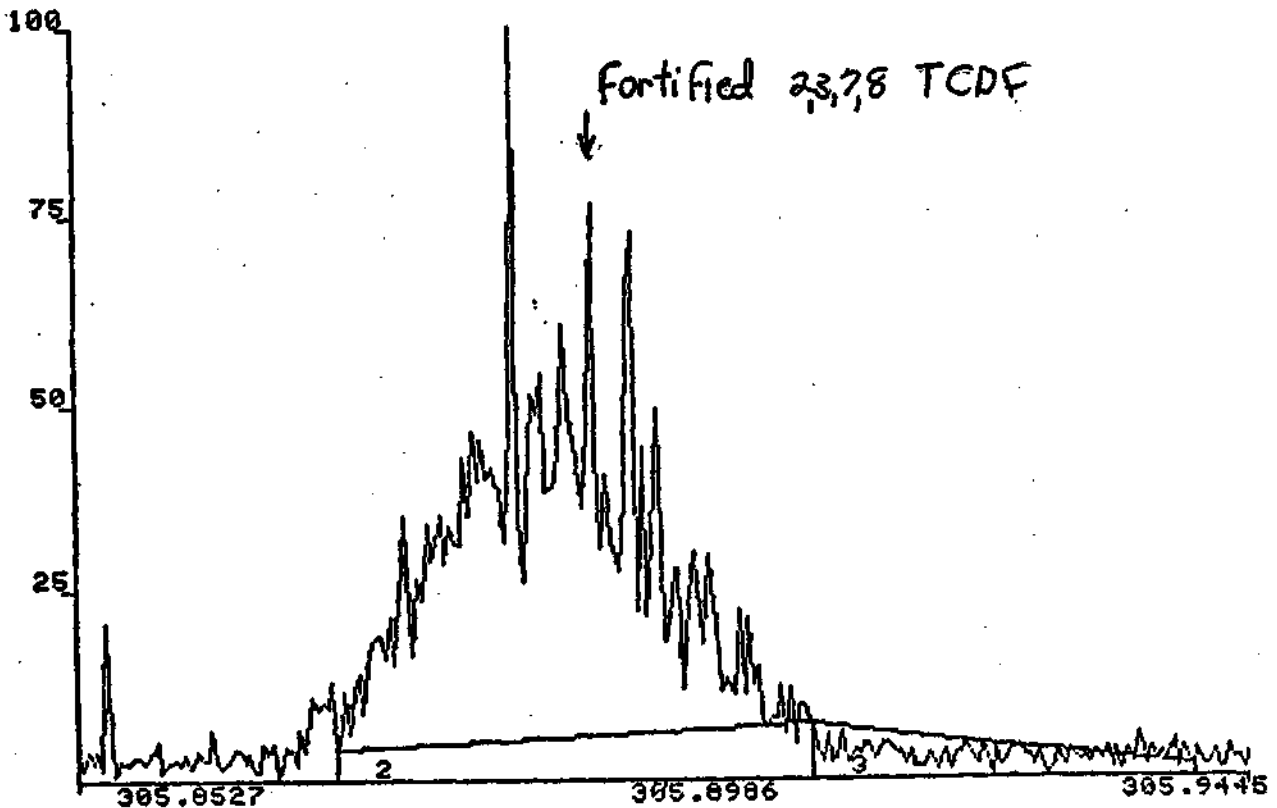
2378 TDF

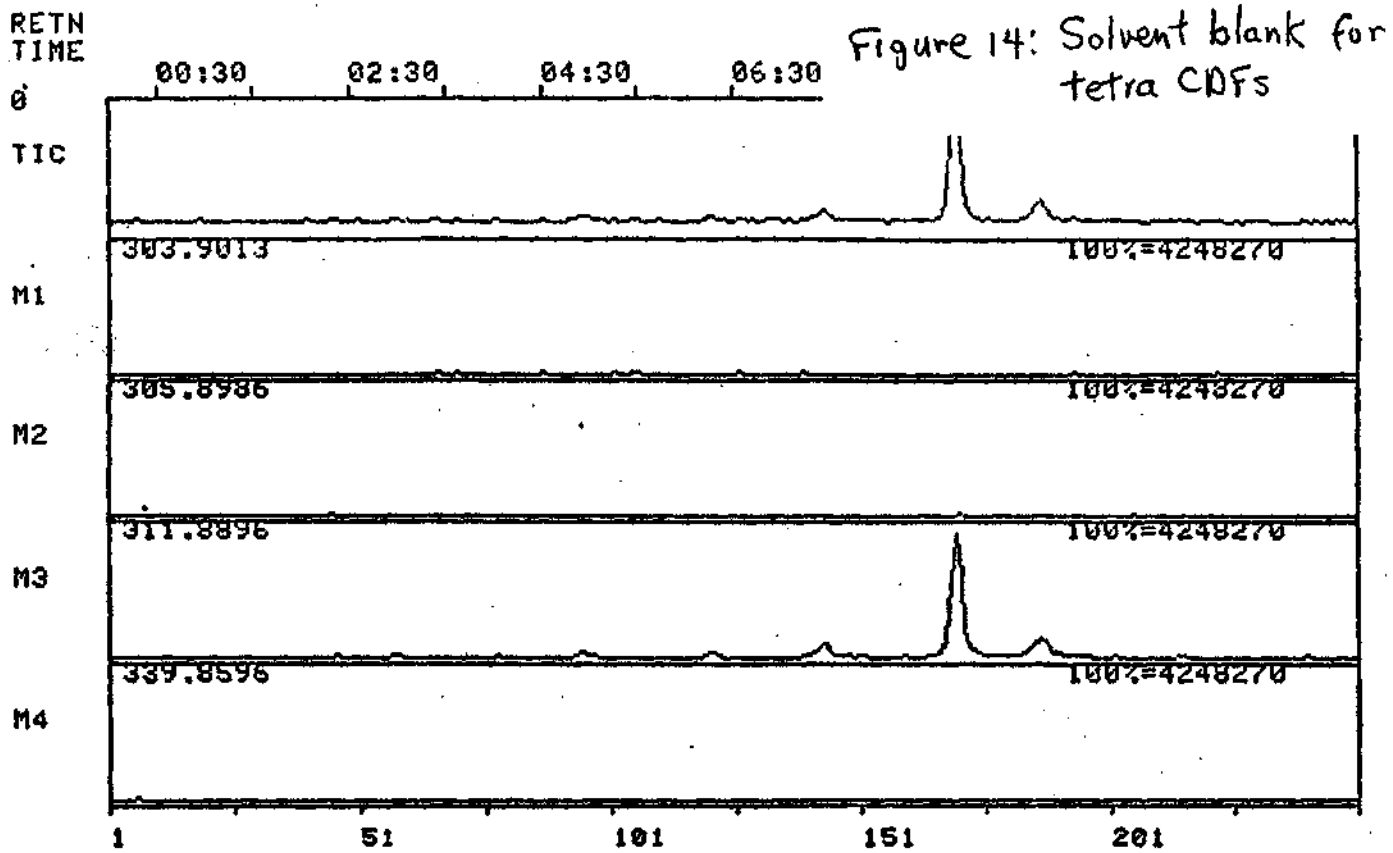
M2



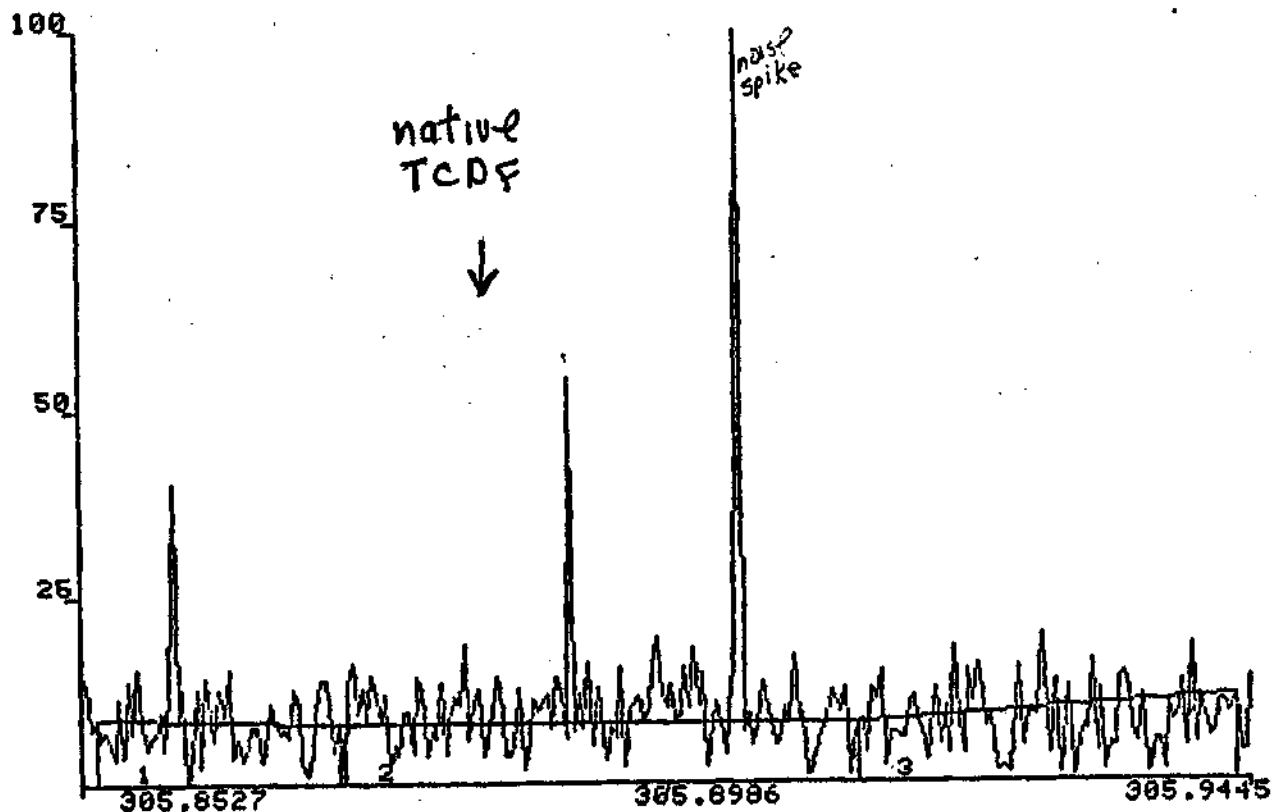


RUNNAME MDHM2 DATE 3/28/83 TIME 15:7  
 MASS 305.8986 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
 SCANS 203-224 100% INTENSITY 83355



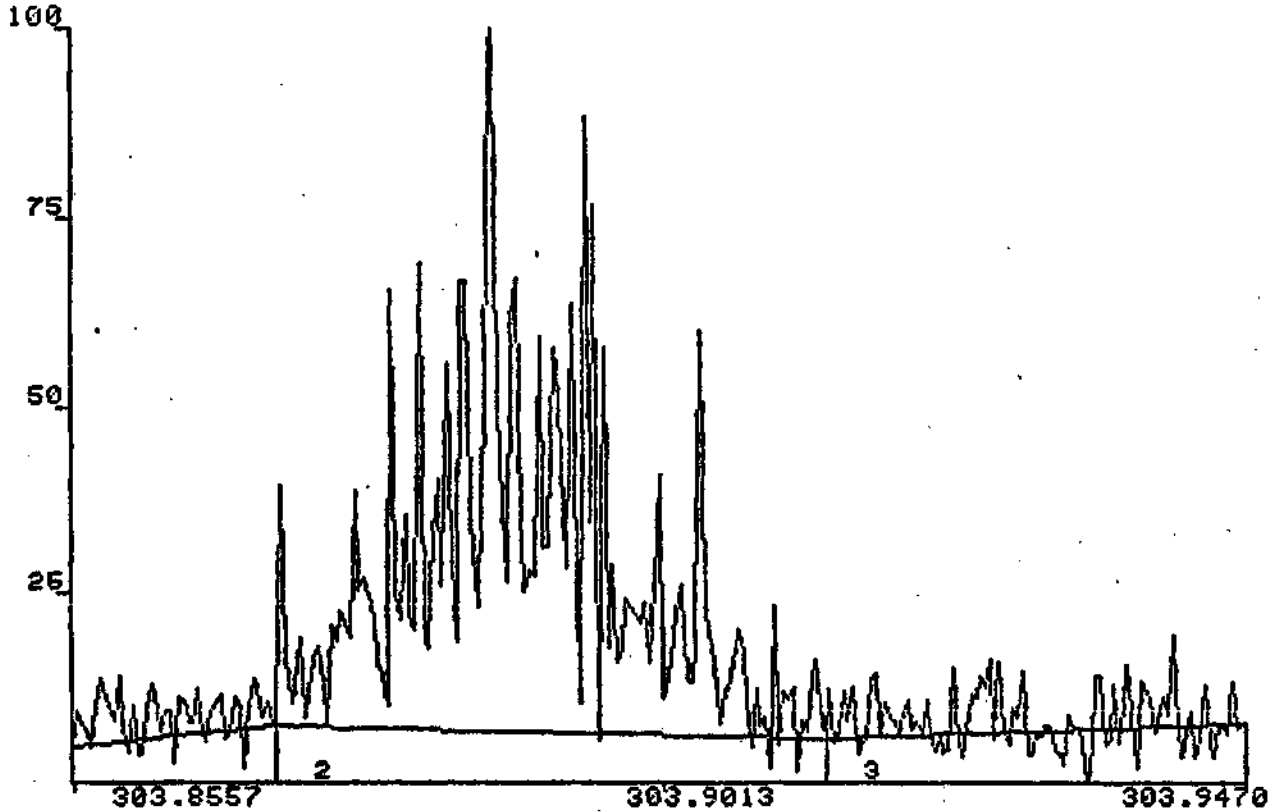


RUNNAME TUDHM5 DATE 3/29/83 TIME 11:40  
 MASS 305.8986 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
 SCANS 164-178 100% INTENSITY 26752

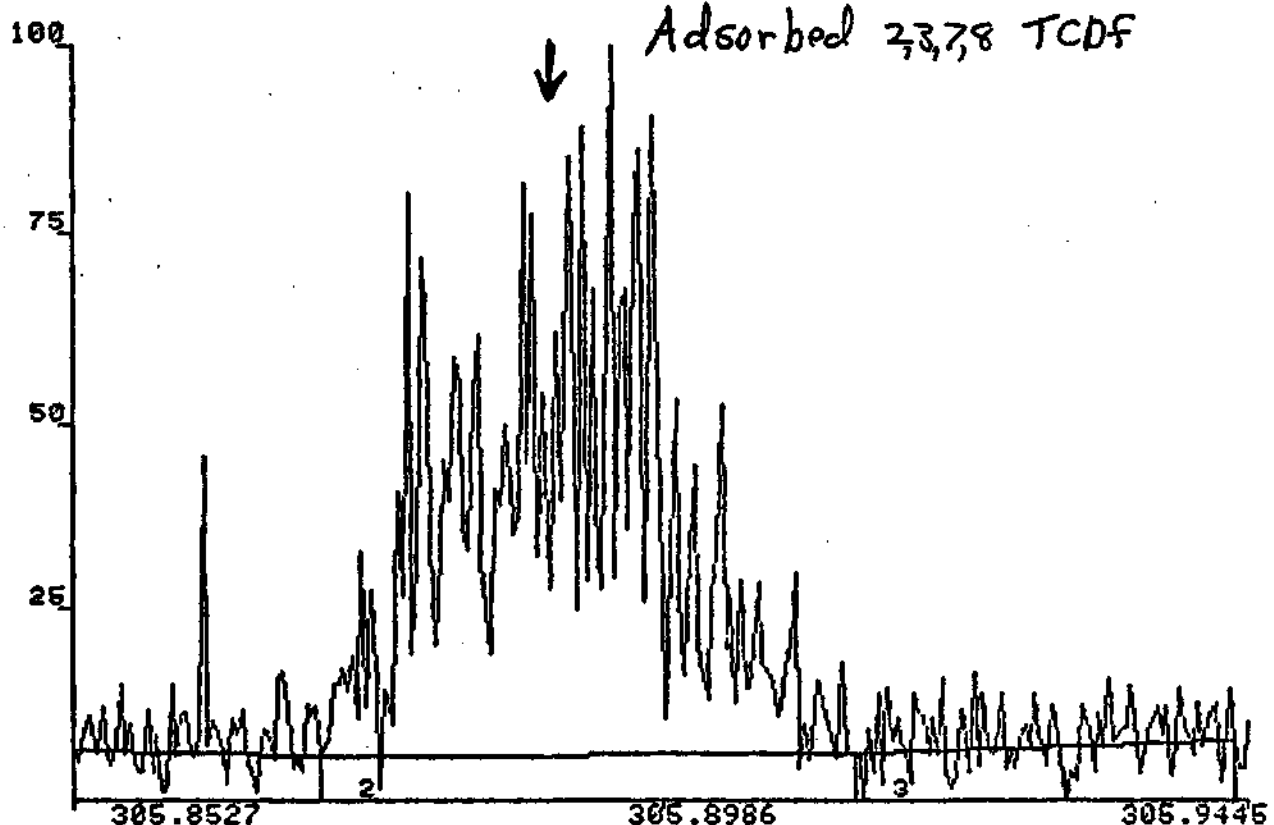


RUNNAME MDHMS      DATE 3/28/83      TIME  
MASS 303.9013      SWEEP 300 (PPM)  
SCANS 189-199      100% INTENSITY 24601

Figure 15: tetra CDFs in  
Sample 17CIG  
Silica Gel cartridge only



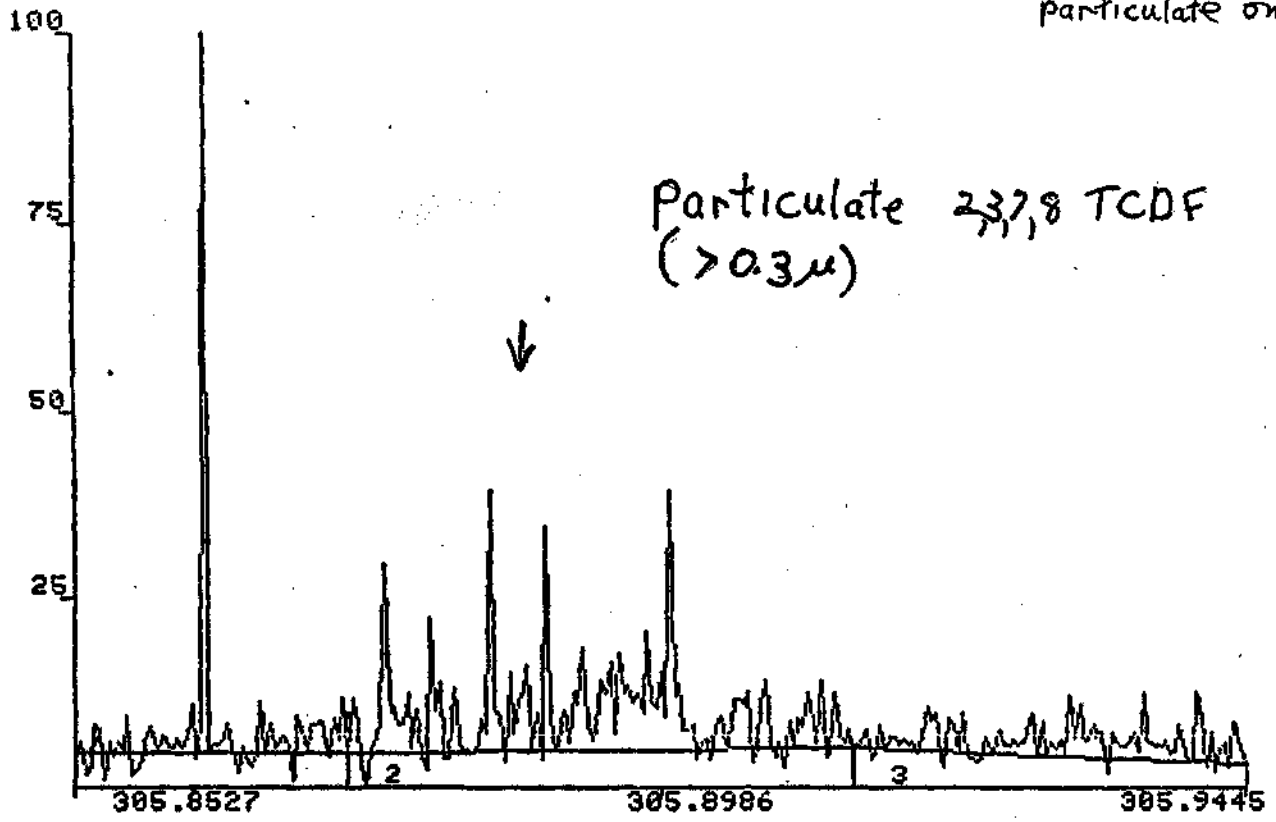
\*SK AREA ID:1      \*  
RUNNAME MDHMS      DATE 3/28/83      TIME 18:0  
MASS 305.8986      SWEEP 300 (PPM)      SCANTIME 0.3 (SECS)  
SCANS 189-199      100% INTENSITY 23146



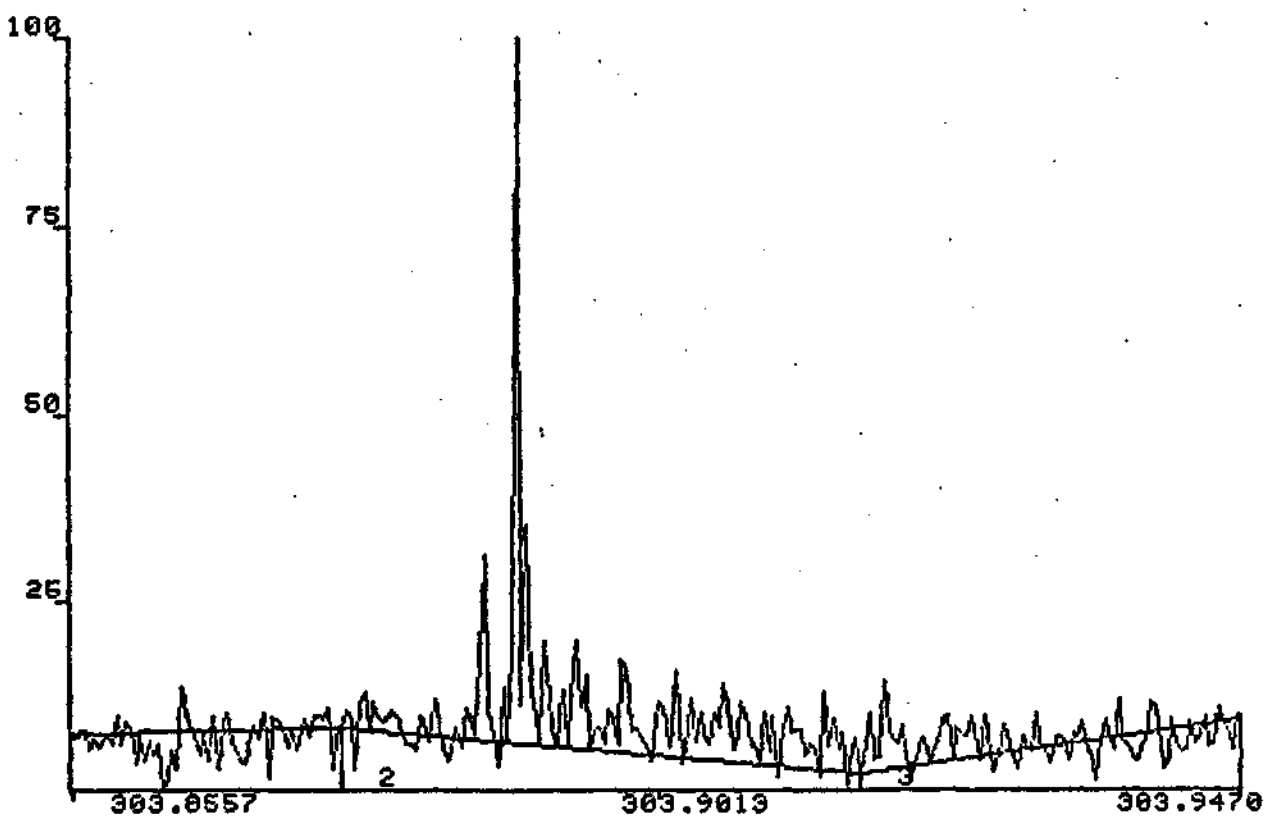
\*SK AREA ID:1      \*

RUNNAME TUDHM4      DATE 3/29/83      TIM  
MASS 305.8986      SWEEP 300 (PPM)  
SCANS 207-217 100% INTENSITY 32973

Figure 16: Tetra CDFs in  
Sample 1? CIP  
particulate only



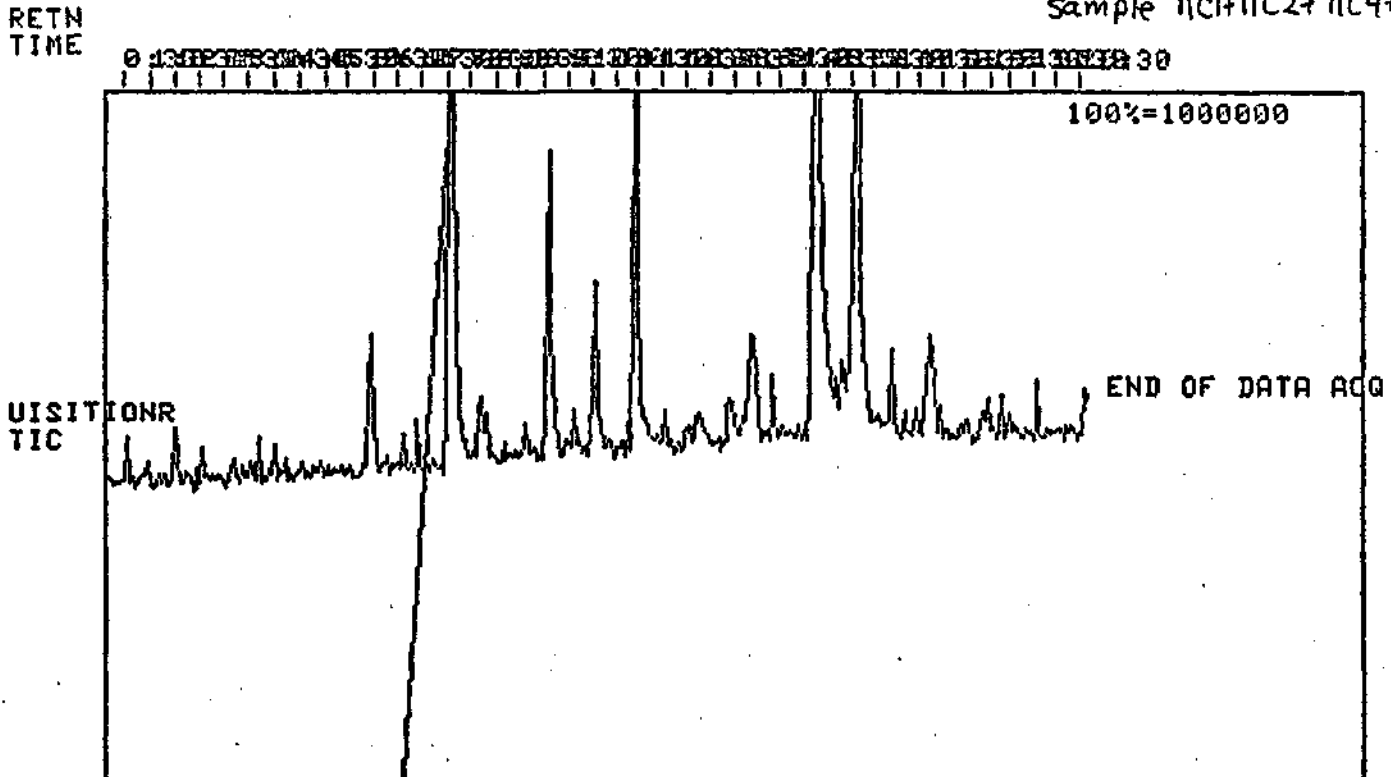
\*SK AREA ID:1      \*  
RUNNAME TUDHM4      DATE 3/29/83      TIME 10:29  
MASS 303.9013      SWEEP 300 (PPM)      SCANTIME 0.3 (SECS)  
SCANS 207-217 100% INTENSITY 31235



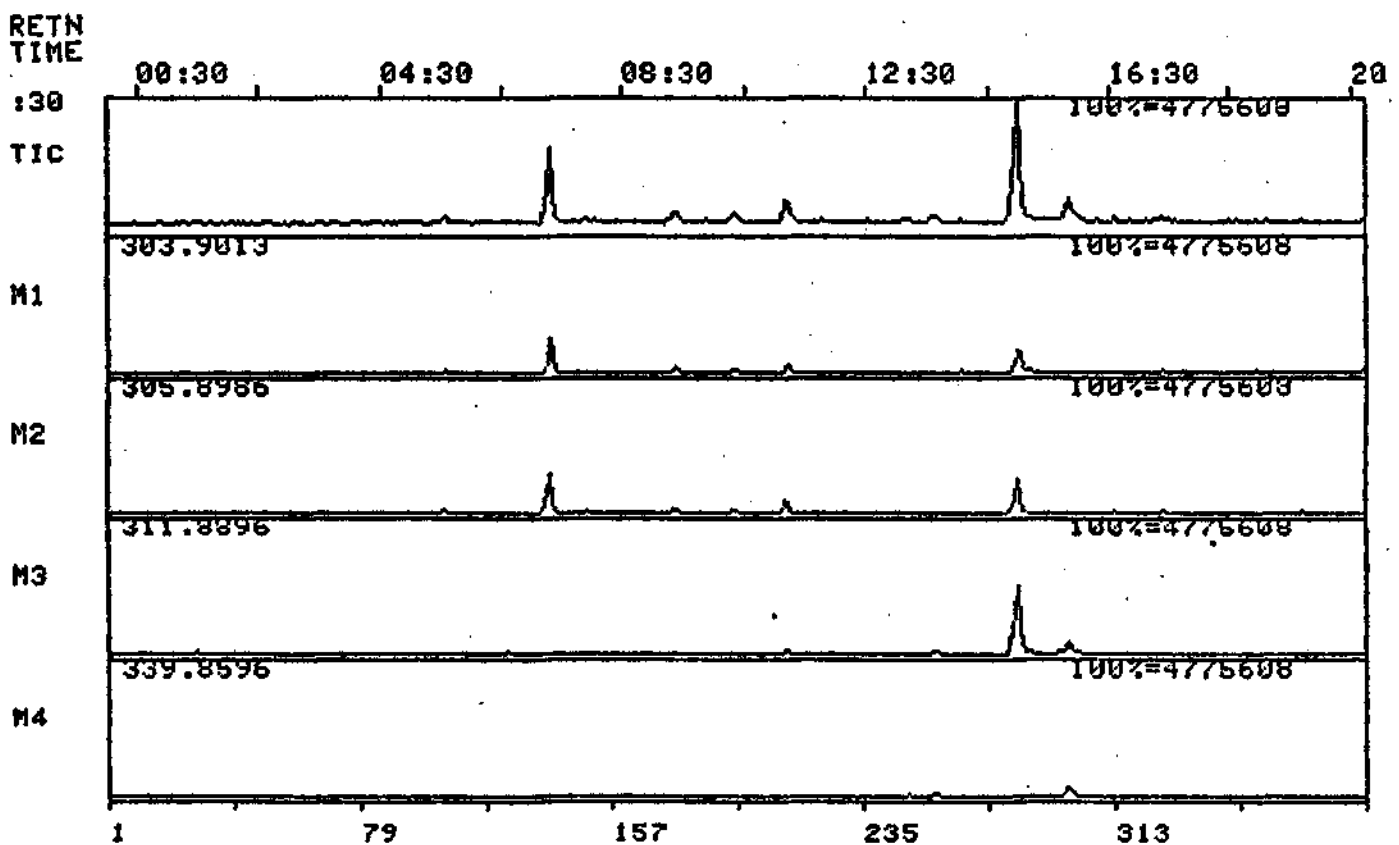
\*SK AREA ID:1      \*

DS55 HIGH RESOLUTION MPM  
RUNNAME TDHNS DATE 3/31/83

Figure 17: Ion chromatograms  
tetra (and penta) CDFs  
sample 11C1+11C2+11C4+11C5



DS55 HIGH RESOLUTION MPM  
RUNNAME TDHNS DATE 3/31/83 TIME 11:31



\*

DSSS HIGH RESOLUT  
PEAK SUMMATION R

Figure 18: mass profile + area  
Sample 11C1+11C2+11C4+11C5

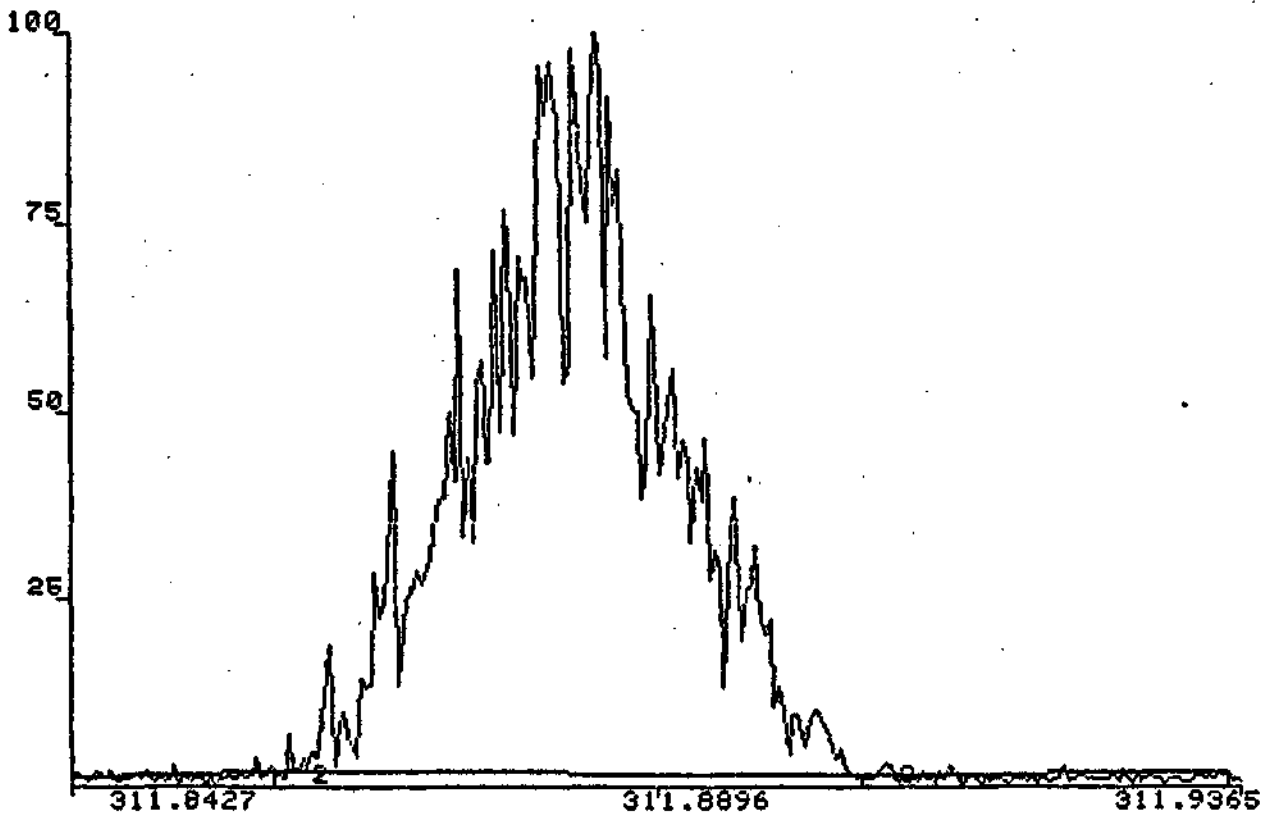
RUNNAME TDHMS DATE 3/31/83 TIME 11:31

MASS 311.8897  
SCAN WIDTH 300 PPM  
SCAN TIME 0.3 SECS  
SCAN NUMBERS 279-287  
STANDARD 0.0000  
FACTOR 0

1.95 OF 7.6UL SAMPLE #C11(1+2+4+5)

MASS CENTROID	ITEM TOTAL	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
311.8836	TOTAL	5945755.	YES	NO	88.80	0.00
311.8508	1	8642.	YES	YES	0.13	0.00
311.8823	2	5458992.	YES	YES	80.79	0.00
311.9207	3	7957.	YES	YES	0.12	0.00

\*  
RUNNAME TDHMS DATE 3/31/83 TIME 11:31  
MASS 311.8896 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 279-287 100% INTENSITY 127239



\*SK AREA ID:1



DS55 HIGH RESOLUT  
PEAK SUMMATION RI

Figure 19: areas  
Sample 11C1+11C2+11C4+11C5

RUNNAME TDHMS DATE 3/31/83 TIME 11:31

MASS 303.9014  
SCAN WIDTH 300 PPM  
SCAN TIME 0.3 SECS  
SCAN NUMBERS 279- 287  
STANDARD 0.0000  
FACTOR 0

1.95 OF 7.6UL SAMPLE #C11(1+2+4+5)

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
303.8958	TOTAL	2350334.	YES	NO	75.84	0.00
303.8628	1	7640.	YES	YES	0.25	0.00
303.8928	2	2049323.	YES	YES	66.13	0.00
303.9319	3	14135.	YES	YES	0.46	0.00

\*  
DS55 HIGH RESOLUTION MPM  
PEAK SUMMATION REPORT

RUNNAME TDHMS DATE 3/31/83 TIME 11:31

MASS 305.8987  
SCAN WIDTH 300 PPM  
SCAN TIME 0.3 SECS  
SCAN NUMBERS 279- 287  
STANDARD 0.0000  
FACTOR 0

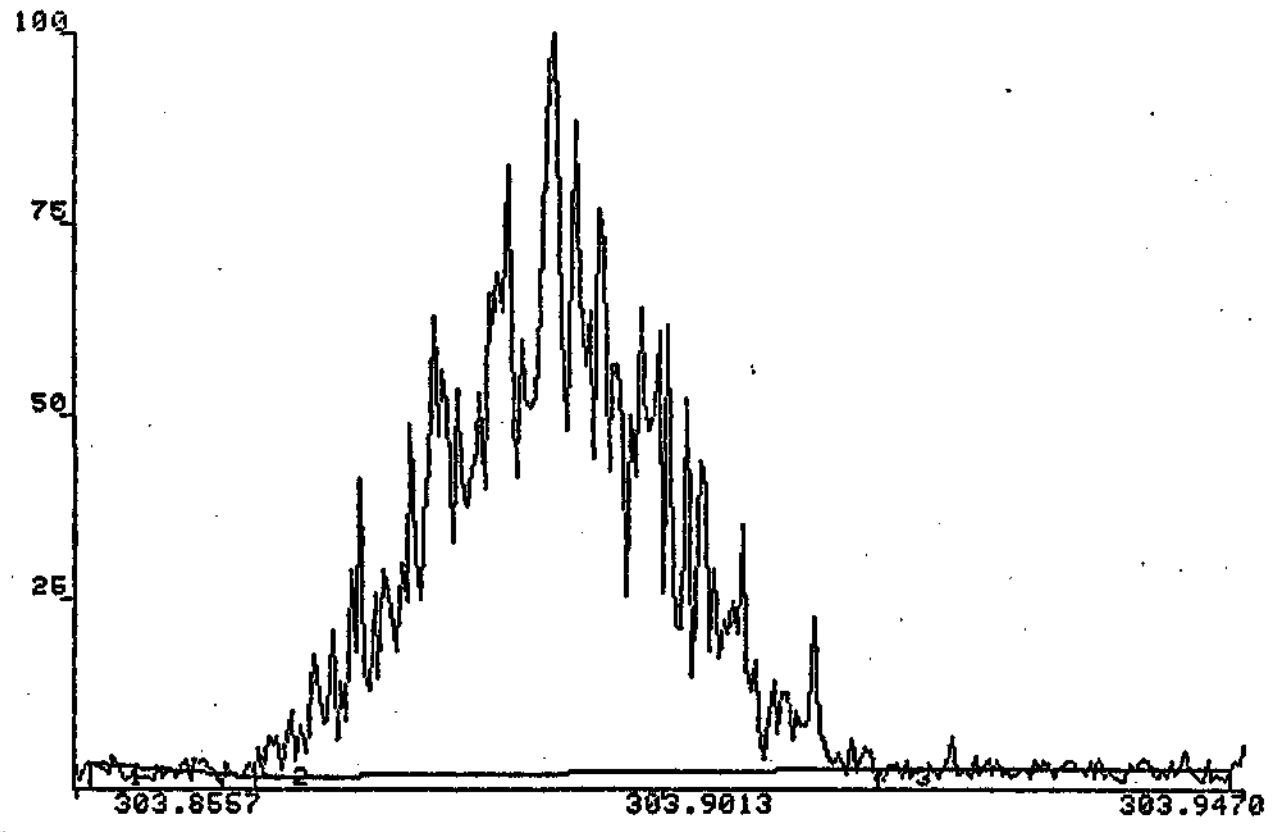
1.95 OF 7.6UL SAMPLE #C11(1+2+4+5)

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
305.8953	TOTAL	2896173.	YES	NO	77.40	0.00
305.8621	1	18242.	YES	YES	0.49	0.00
305.8936	2	2549809.	YES	YES	68.15	0.00
305.9304	3	8232.	YES	YES	0.22	0.00

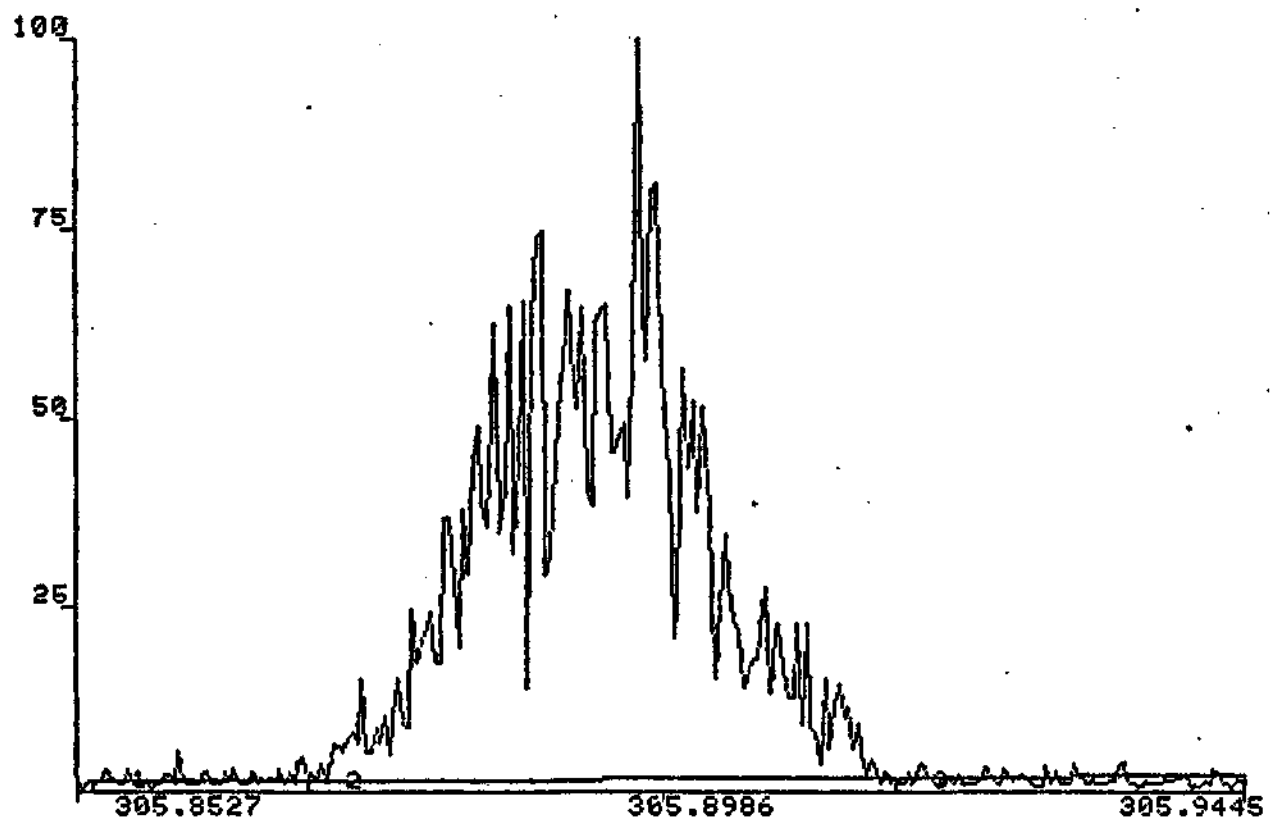
\*

RUNNAME TDHMS      DATE 3/31/83      TIM  
MASS 303.9013      SWEEP 300 (PPM)  
SCANS 279-287 100% INTENSITY 54145

Figure 20: mass profiles  
Sample 11C1+11C2+11C4+11C5



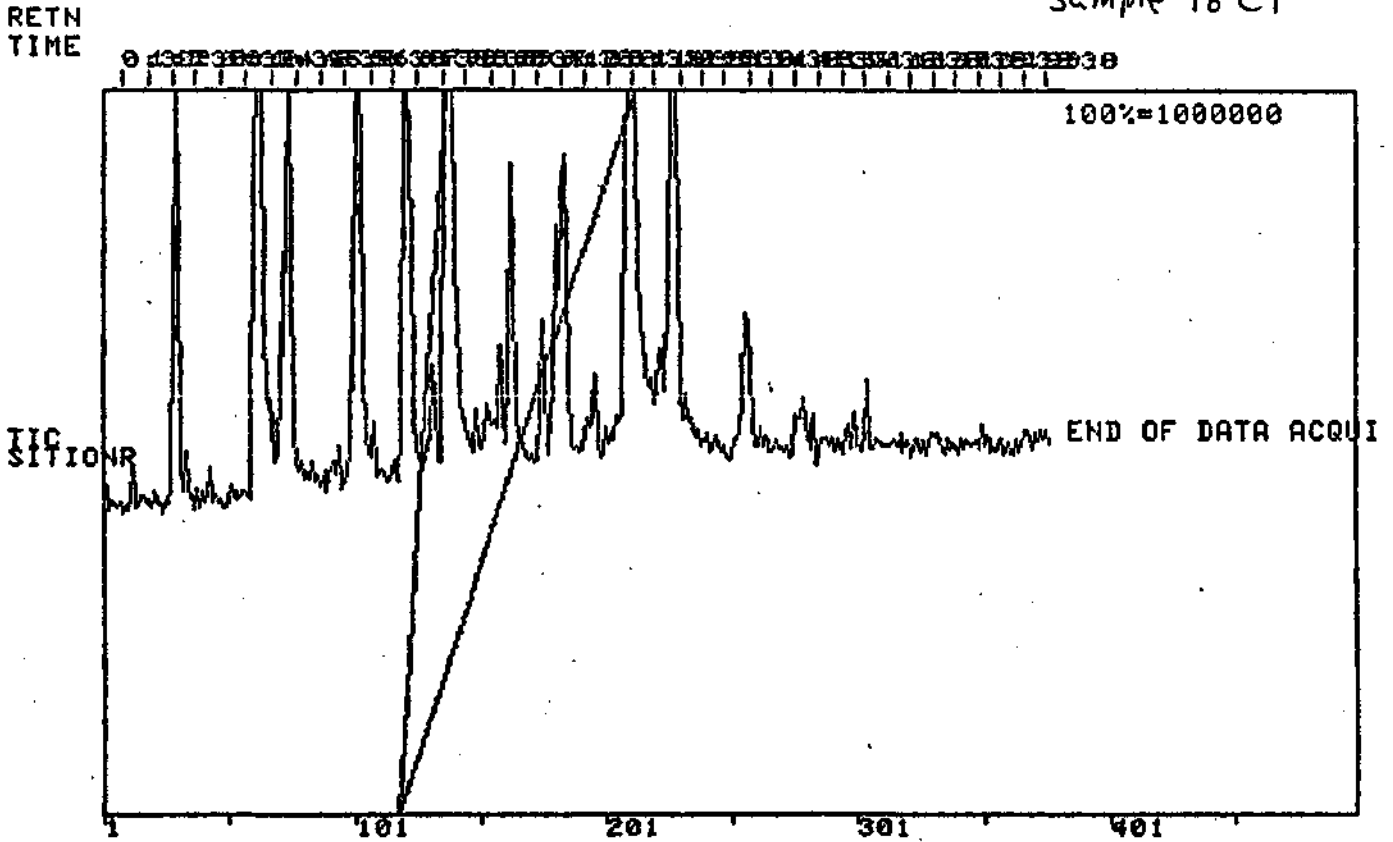
\*  
RUNNAME TDHMS      DATE 3/31/83      TIME 11:31  
MASS 305.8986      SWEEP 300 (PPM)      SCANTIME 0.3 (SECS)  
SCANS 279-287 100% INTENSITY 76709



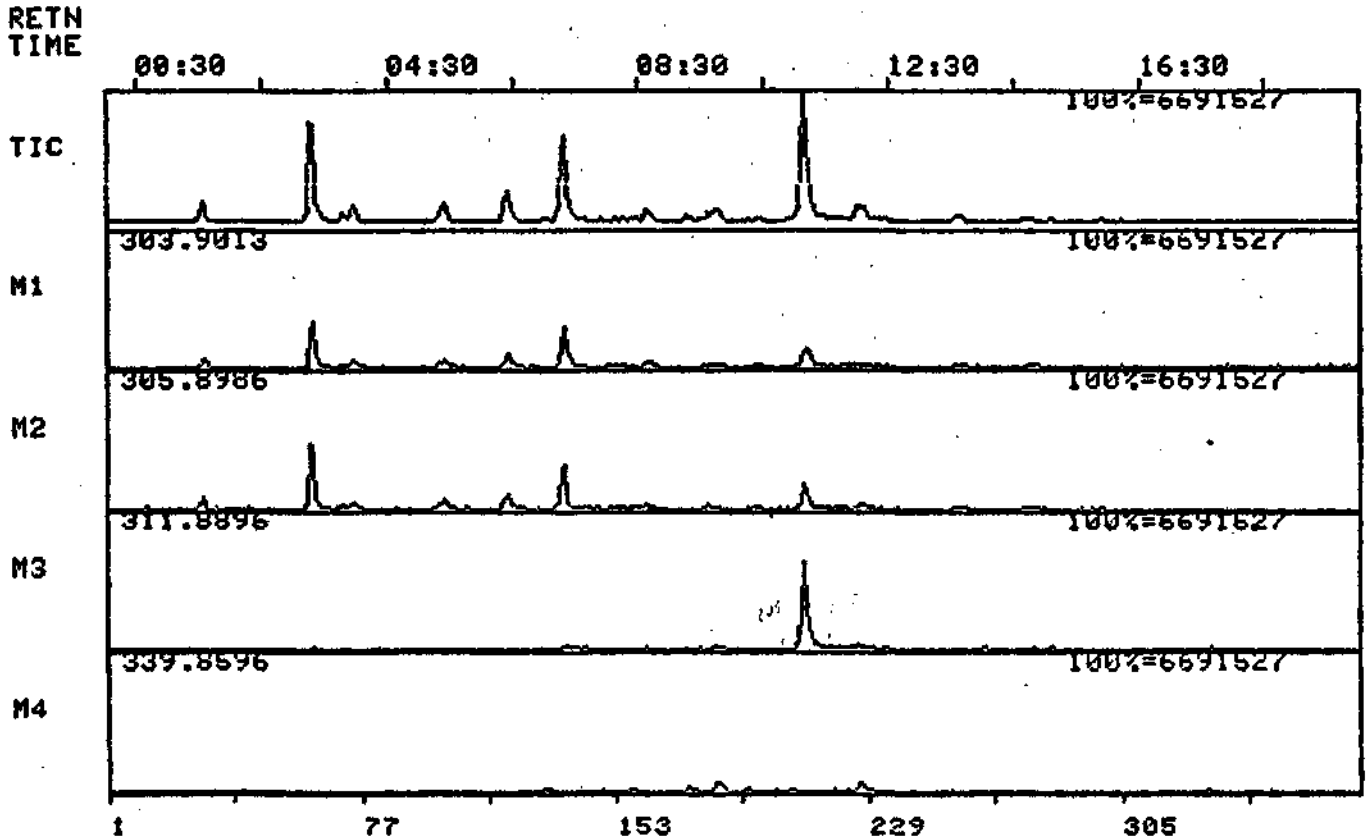
\*

DS55 HIGH RESOLUTION MPM  
RUNNAME MDHM3 DATE 3/28/83 TIME

Figure 21: Ion chromatograms  
tetra (and penta) CDFs  
sample 16 C1



DS55 HIGH RESOLUTION MPM  
RUNNAME MDHM3 DATE 3/28/83 TIME 16:14



PEAK SUMMATION R *Figure 22: mass profile + areas*  
*Sample 16 C1*

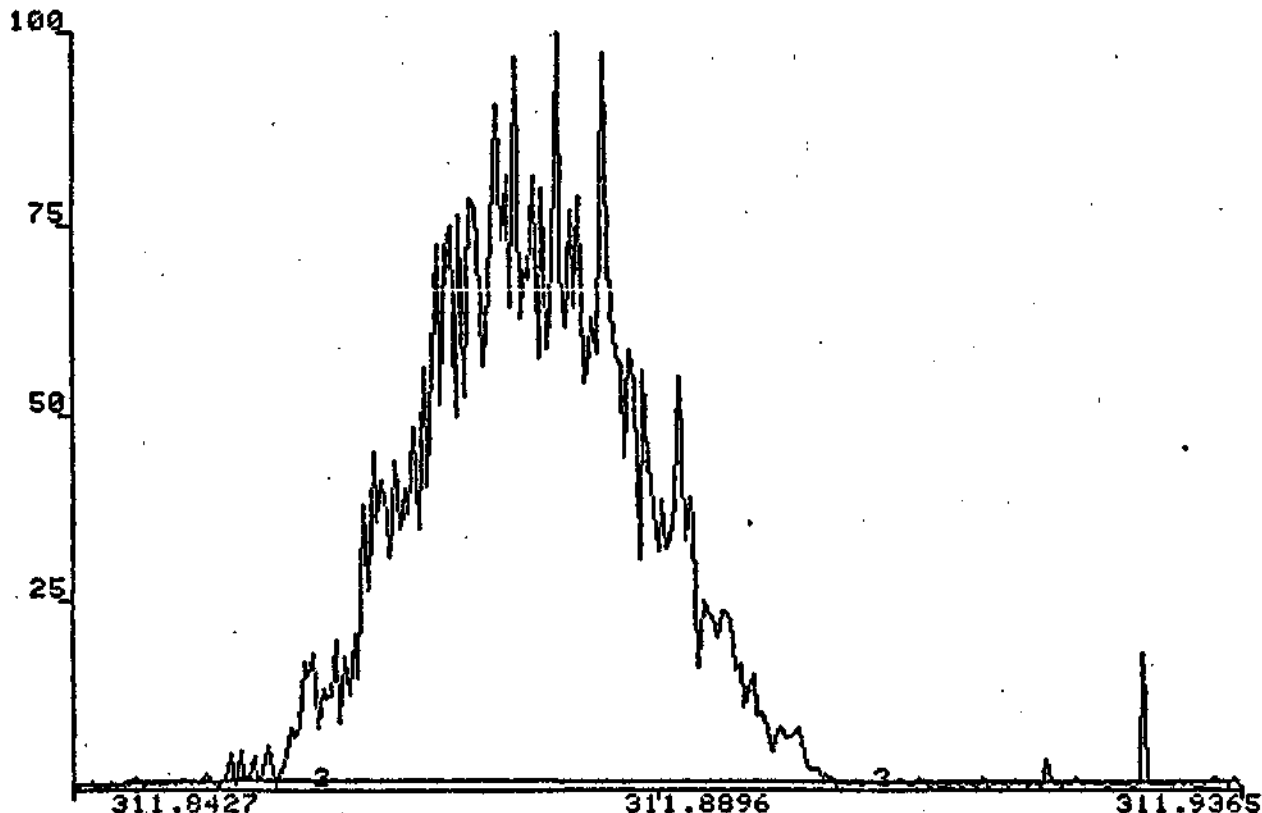
RUNNAME MDHM3 DATE 3/28/83 TIME 16:14

MASS 311.8897  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 207- 215  
 STANDARD 0.0000  
 FACTOR 0

16C1 2UL OF 8UL TOTAL

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
311.8806	TOTAL	9961496.	YES	NO	93.25	0.00
311.8521	1	42788.	YES	YES	0.40	0.00
311.8799	2	9403350.	YES	YES	88.02	0.00
311.9202	3	54098.	YES	YES	0.51	0.00

\*  
 RUNNAME MDHM3 DATE 3/28/83 TIME 16:14  
 MASS 311.8896 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
 SCANS 207-215 100% INTENSITY 207755



\*SK AREA ID:1

Figure 23: areas  
sample 16C1

DS55 HIGH RESOLU

PEAK SUMMATION R. ....

RUNNAME MDHM3 DATE 3/28/83 TIME 16:14

MASS 303.9014  
SCAN WIDTH 300 PPM  
SCAN TIME 0.3 SECS  
SCAN NUMBERS 207- 215  
STANDARD 0.0000  
FACTOR 0

16C1 2UL OF 8UL TOTAL

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
303.8936	TOTAL	2709686.	YES	NO	78.03	0.00
303.8611	1	6255.	YES	YES	0.18	0.00
303.8904	2	2261770.	YES	YES	65.13	0.00
303.9304	3	35166.	YES	YES	1.01	0.00

DS55 HIGH RESOLUTION MPM

PEAK SUMMATION REPORT

RUNNAME MDHM3 DATE 3/28/83 TIME 16:14

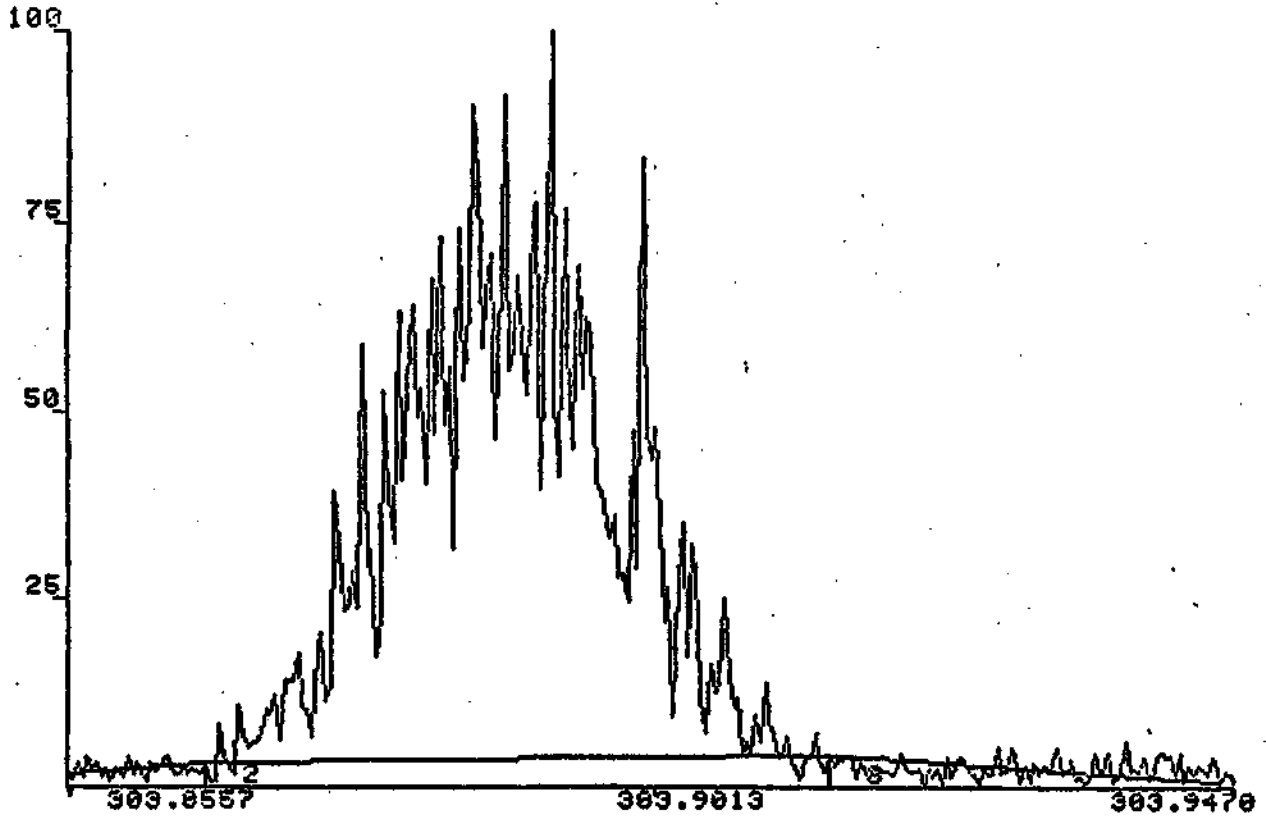
MASS 305.8987  
SCAN WIDTH 300 PPM  
SCAN TIME 0.3 SECS  
SCAN NUMBERS 207- 215  
STANDARD 0.0000  
FACTOR 0

16C1 2UL OF 8UL TOTAL

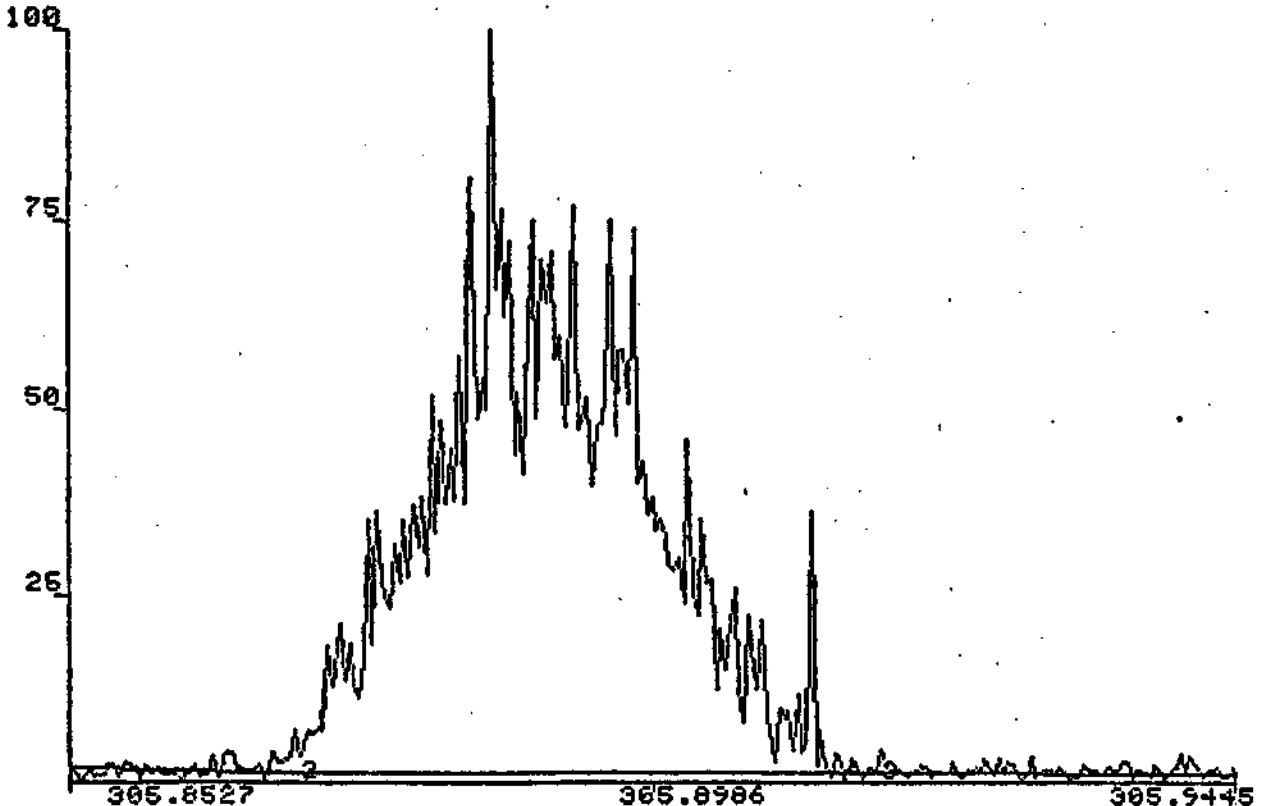
MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
305.8923	TOTAL	3324966.	YES	NO	81.09	0.00
305.8604	1	13730.	YES	YES	0.33	0.00
305.8901	2	3044845.	YES	YES	74.26	0.00
305.9292	3	44193.	YES	YES	1.08	0.00

RUNNAME MDHM3      DATE 3/28/83      TIM  
MASS 303.9013      SWEEP 300 (PPM)  
SCANS 207-215      100% INTENSITY 61571

Figure 24: mass profiles  
sample 16 C1



\*SK AREA ID:1      \*  
RUNNAME MDHM3      DATE 3/28/83      TIME 16:14  
MASS 305.8986      SWEEP 300 (PPM)      SCANTIME 0.3 (SECS)  
SCANS 207-215      100% INTENSITY 81473



\*SK AREA ID:1      \*  
DSS5 HIGH RESOLUTION NPM

Figure 25: Calculations  
Sample 16 Cl

MDHM3 - 2.0 ml of 8.0 ml 16 Cl  
Acqui. @ 20 min.

Amt of <sup>31</sup>Cl-TCDF = 20 x 60 pg/ml = 1200

Conc. of 2378-TCDF = (3044845 / 9403350) x 1200 x 2.5  
= 971 pg (÷ 62.4 = 15.6)

Det. Limit = ((13730 + 44193) / 2 / 9403350) x 1200 x 2.5 x 2.5  
= 23 pg (÷ 62.4 = .37)

Ratio = (2261770 / 3044845) x 100 = 74%

Recovery = (9403356 / 35793 counts/pg / (1200 x 1/4)) x 100 = 889%

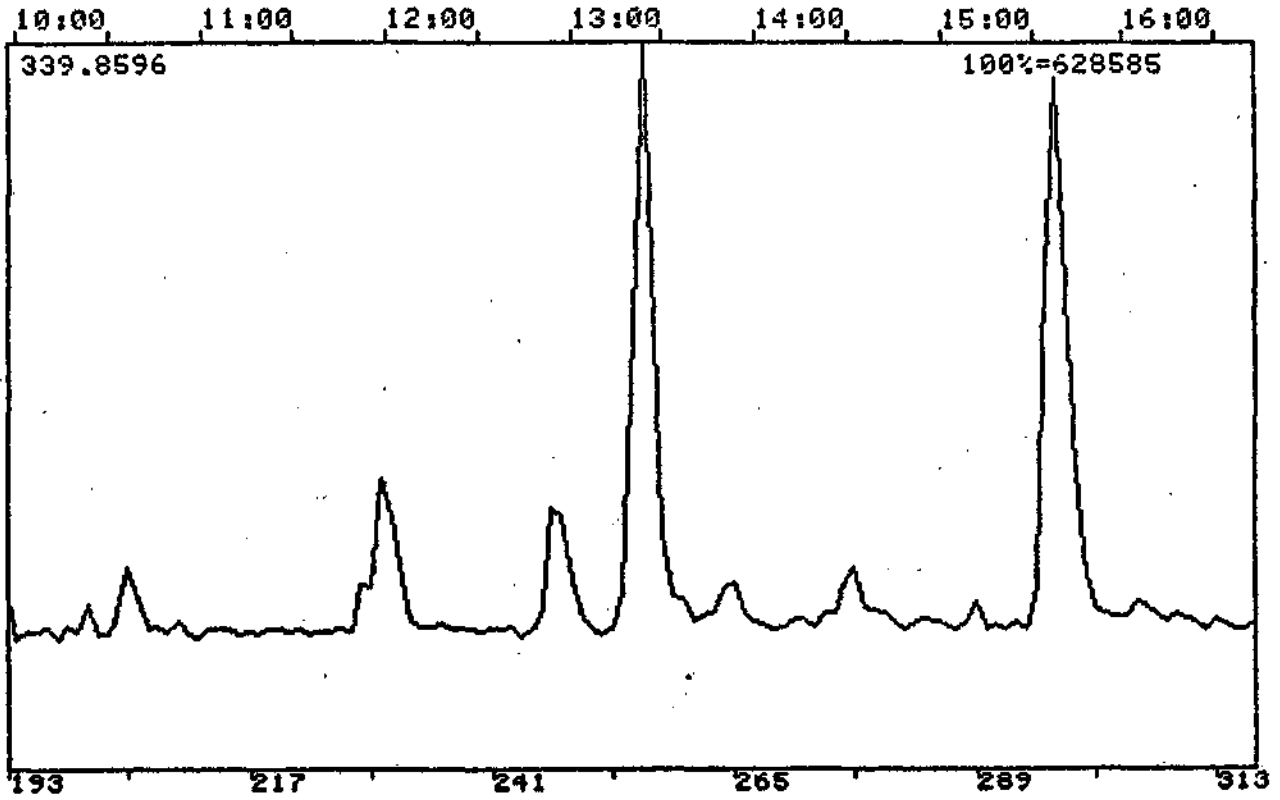
Retention Time = 20 + 10.5 + (.53 / 1.65) x 2 = 31.1 min.

RRT = 1.0

DS55 HIGH RESOLUTION MPM  
RUNNAME WDHM7 DATE 3/30/83 T:

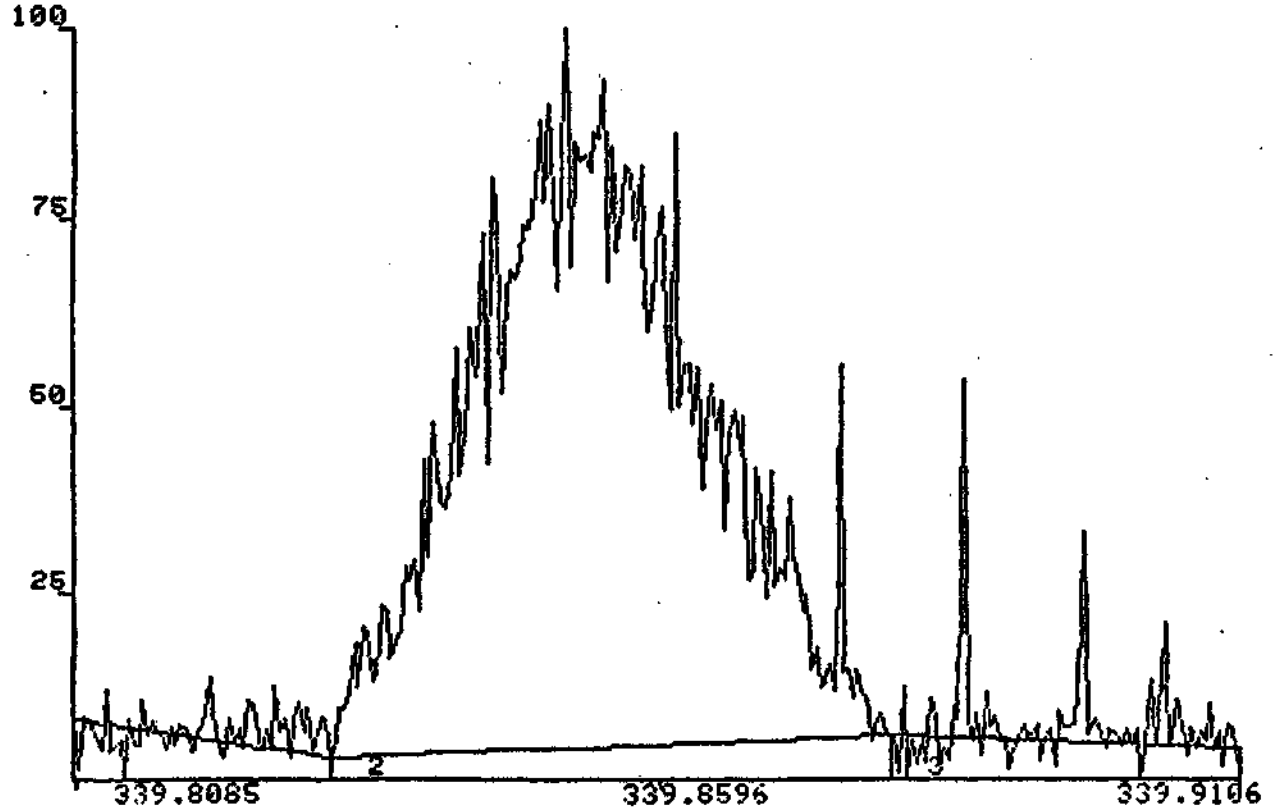
Figure 26: penta CDFs  
Sample 11B3+11B4+11C1

RETN  
TIME



M4

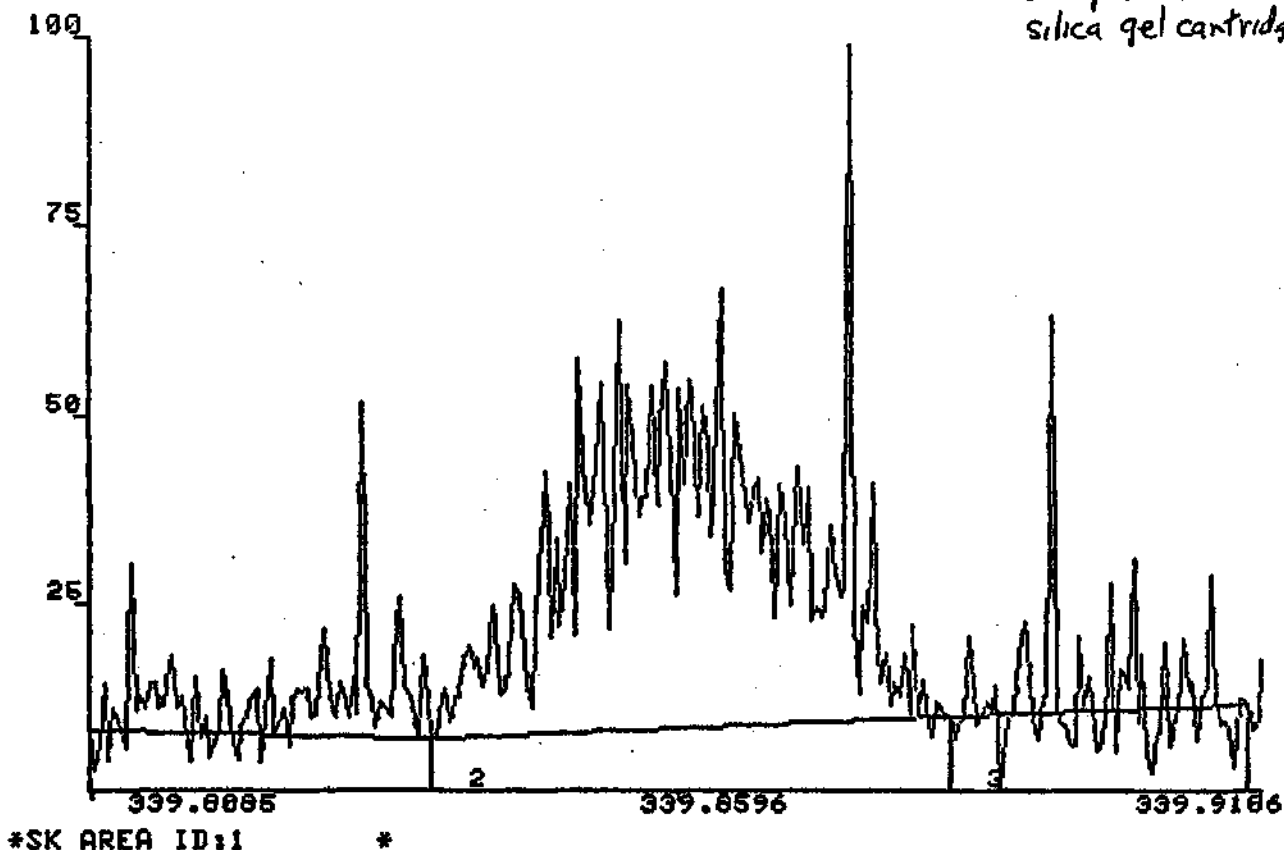
RUNNAME WDHM7 DATE 3/30/83 TIME 12:57  
MASS 339.8596 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 193-317 100% INTENSITY 132598



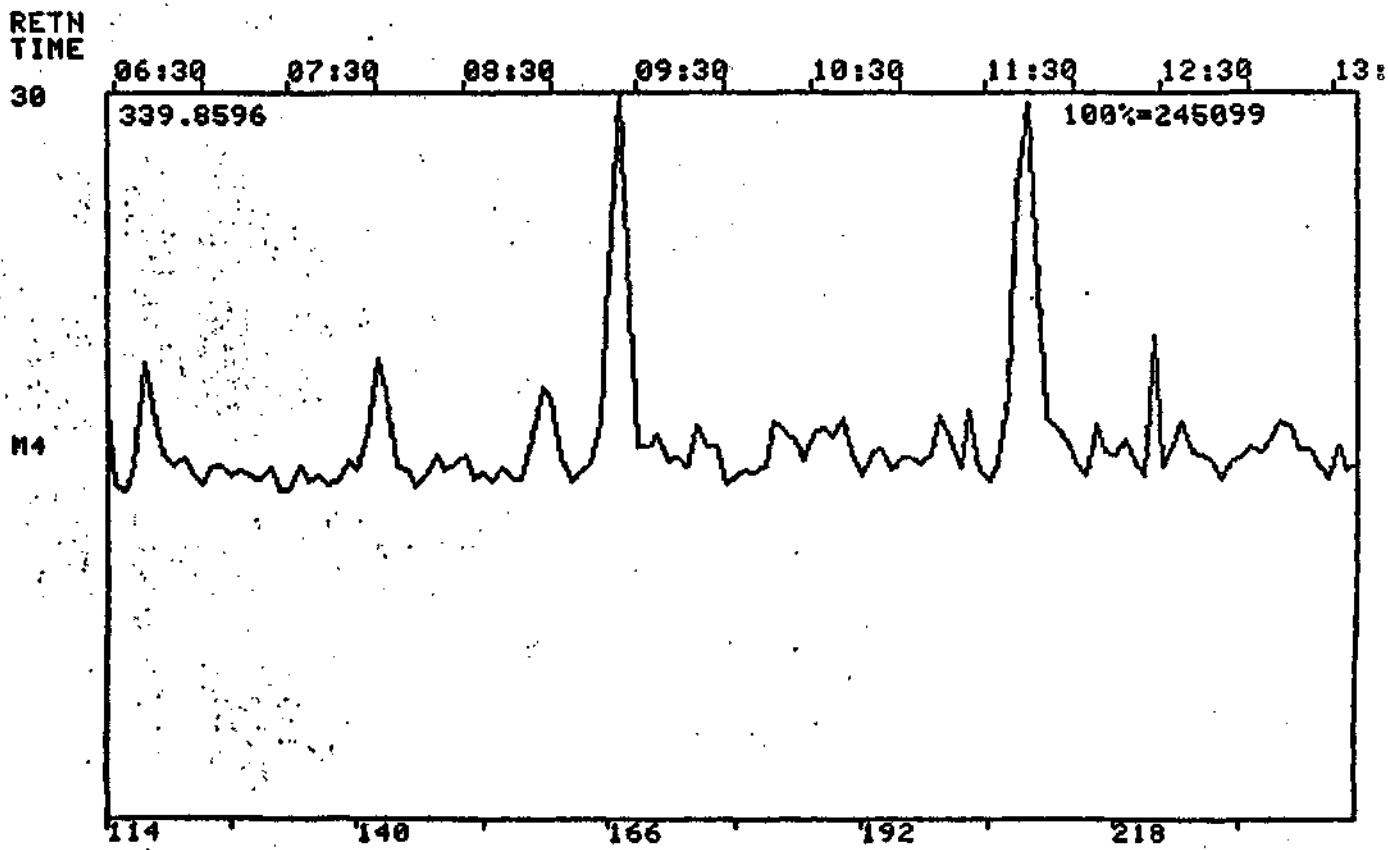


RUNNAME MDHMS DATE 3/28/83 T1  
MASS 339.8596 SWEEP 300 (PPM)  
SCANS 114-243 100% INTENSITY 106788

Figure 27: penta CDFs  
Sample 17C16  
silica gel cartridge only



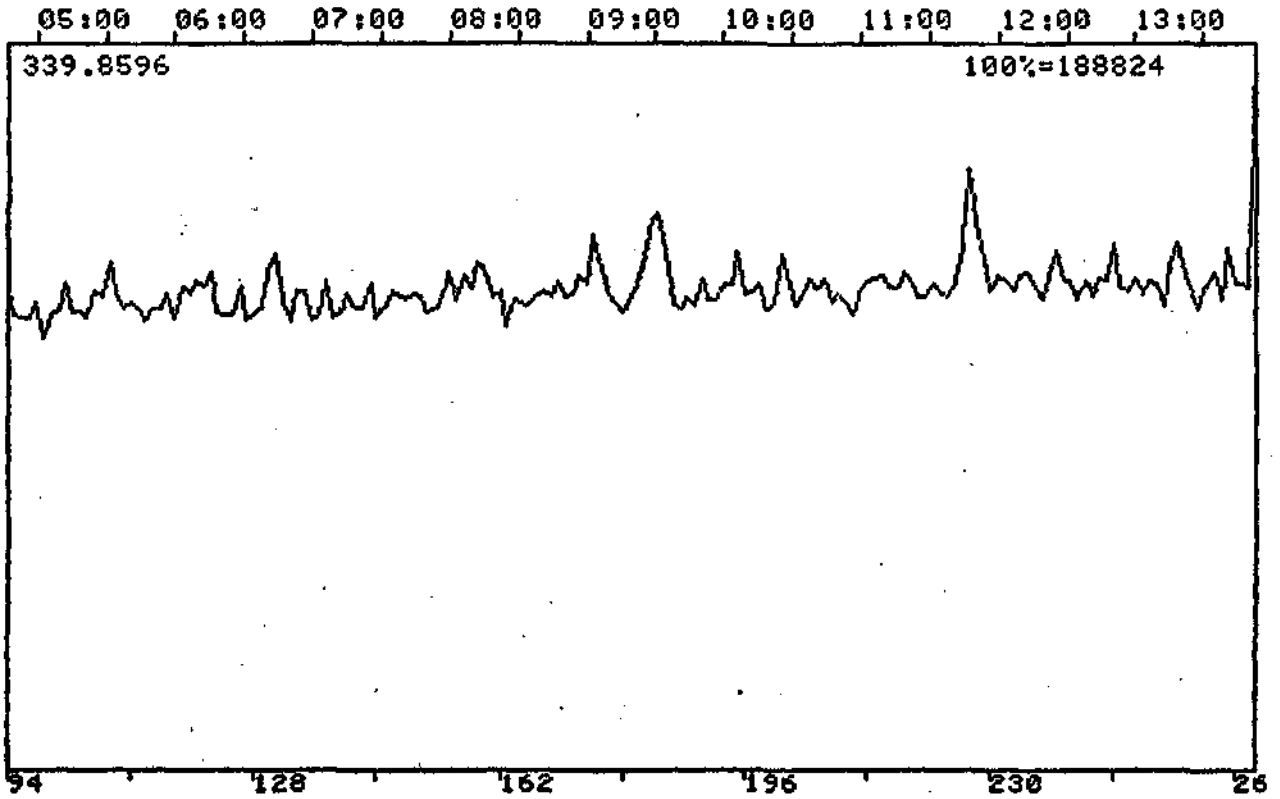
DS55 HIGH RESOLUTION MPM  
RUNNAME MDHMS DATE 3/28/83 TIME 18:0



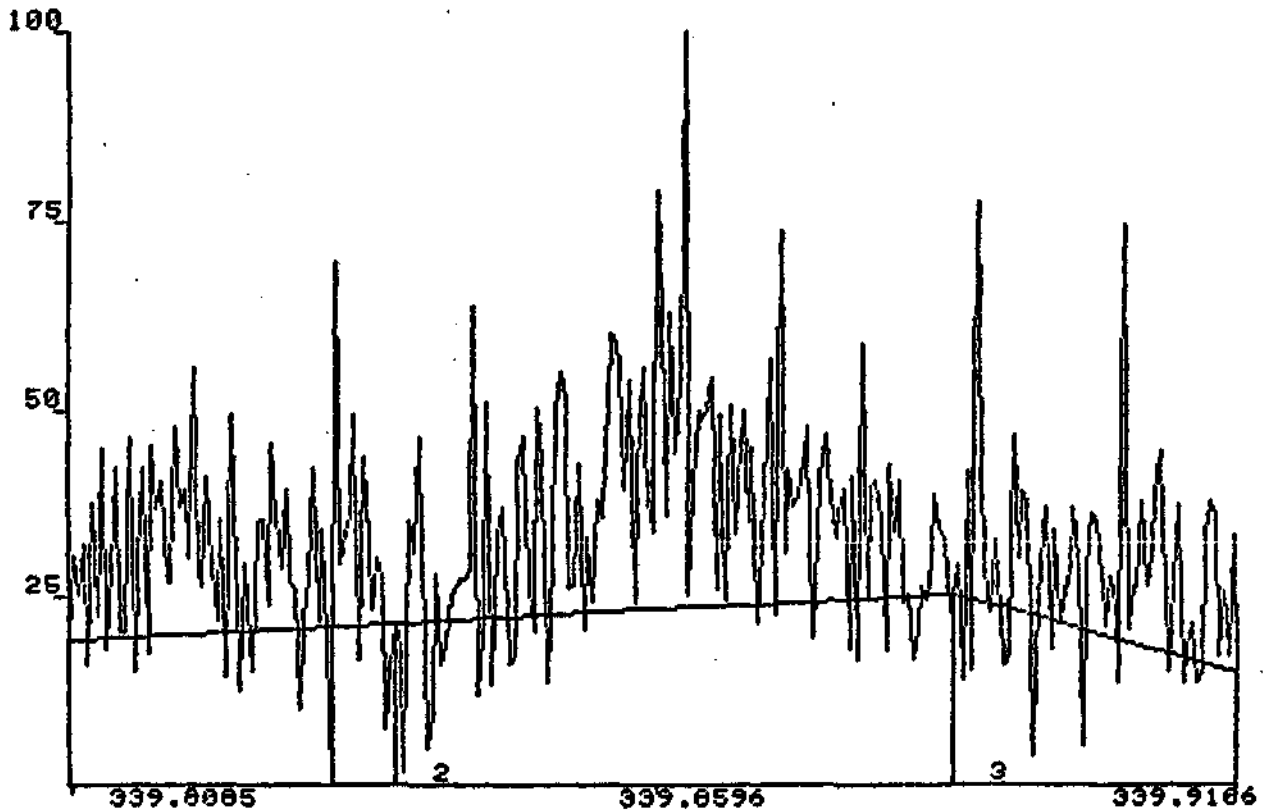
DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHM4 DATE 3/29/83

TIM Figure 28: penta CDFs  
Sample 17C1P  
particulate only

RETN  
TIME



RUNNAME TUDHM4 DATE 3/29/83 TIME 10:29  
MASS 339.8596 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 111-259 100% INTENSITY 88429



\*SK AREA ID:1 \*

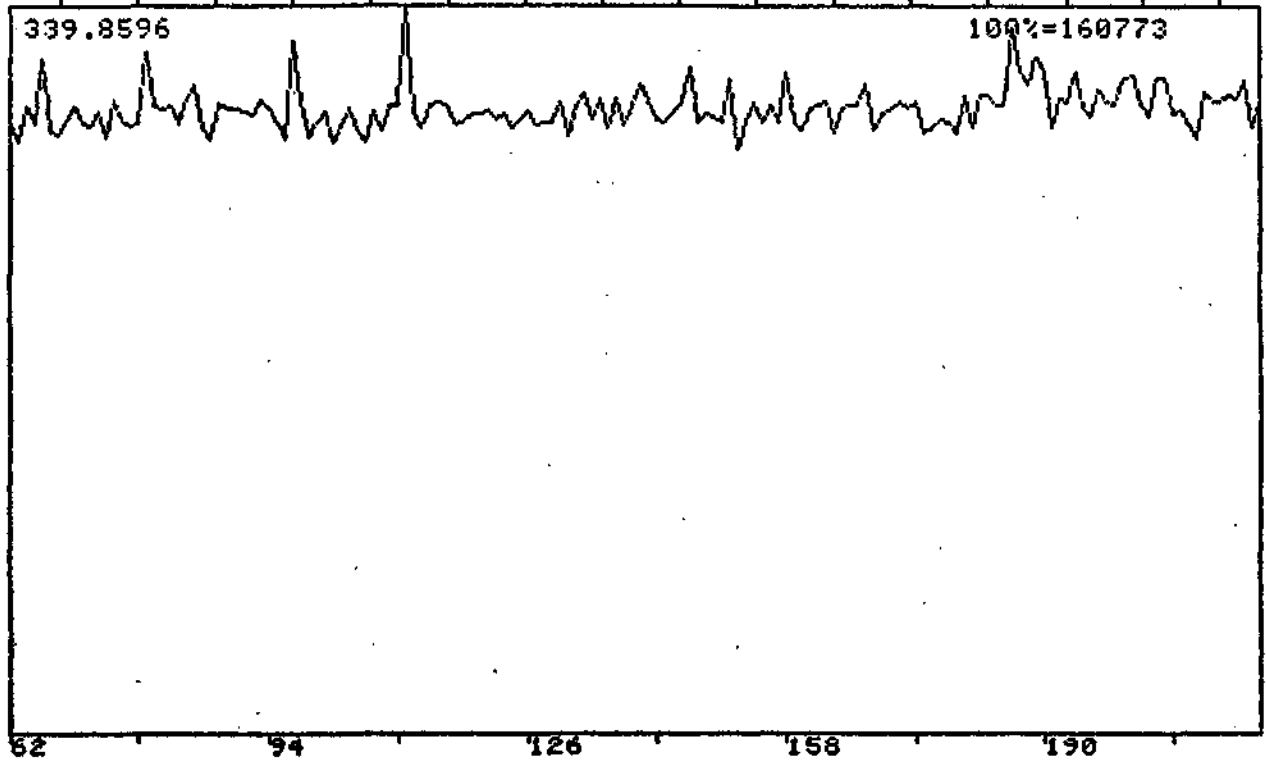
DS55 HIGH RESOLUTION MPM  
RUNNAME TUDHMS

DATE 3/29/83

TIM Figure 29: penta COFs  
Solvent blank

RETN  
TIME

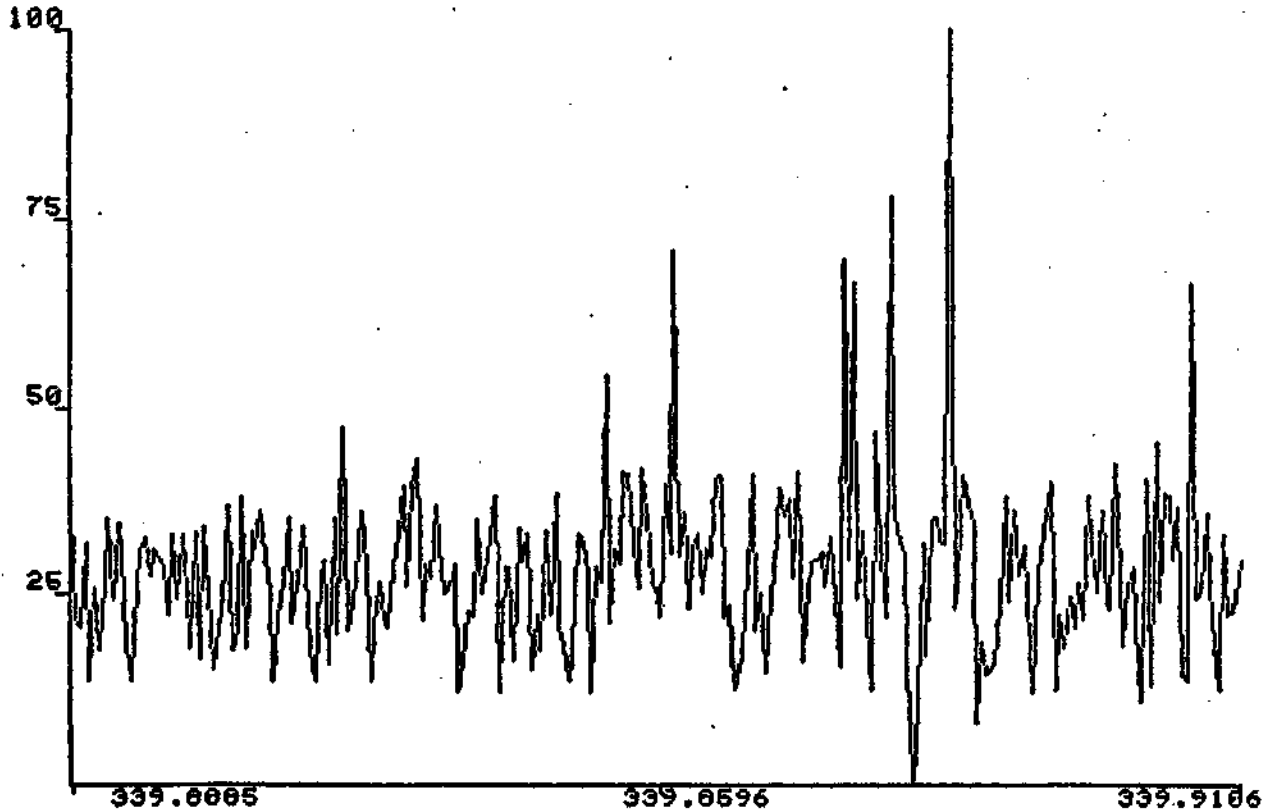
03:30 04:30 05:30 06:30 07:30 08:30 09:30 10:30



N4

\*

RUNNAME TUDHMS DATE 3/29/83 TIME 11:40  
MASS 339.8596 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 62-217 100% INTENSITY 108549

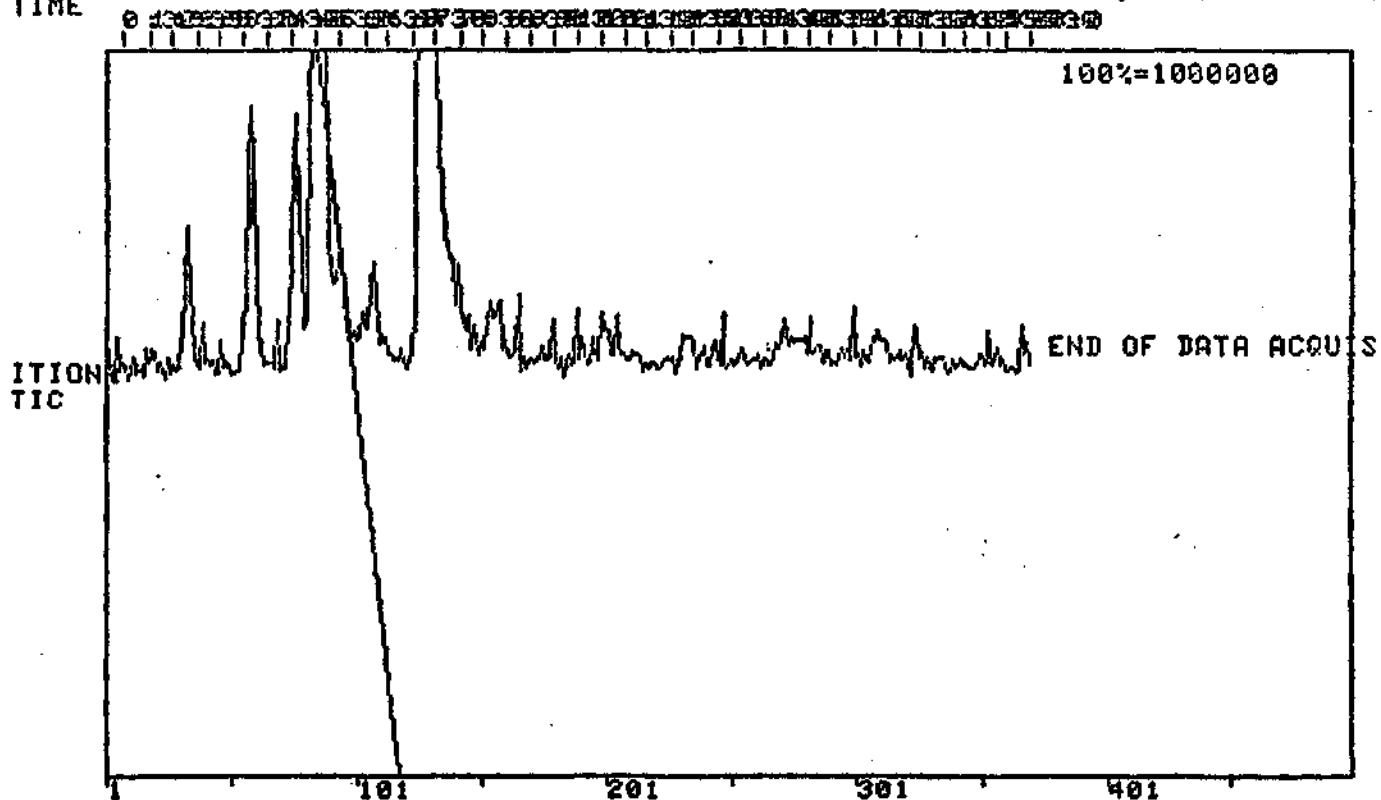


\*

DS55 HIGH RESOLUTION MPM  
RUNNAME MDHM7 DATE 4/ 4/83 TIME

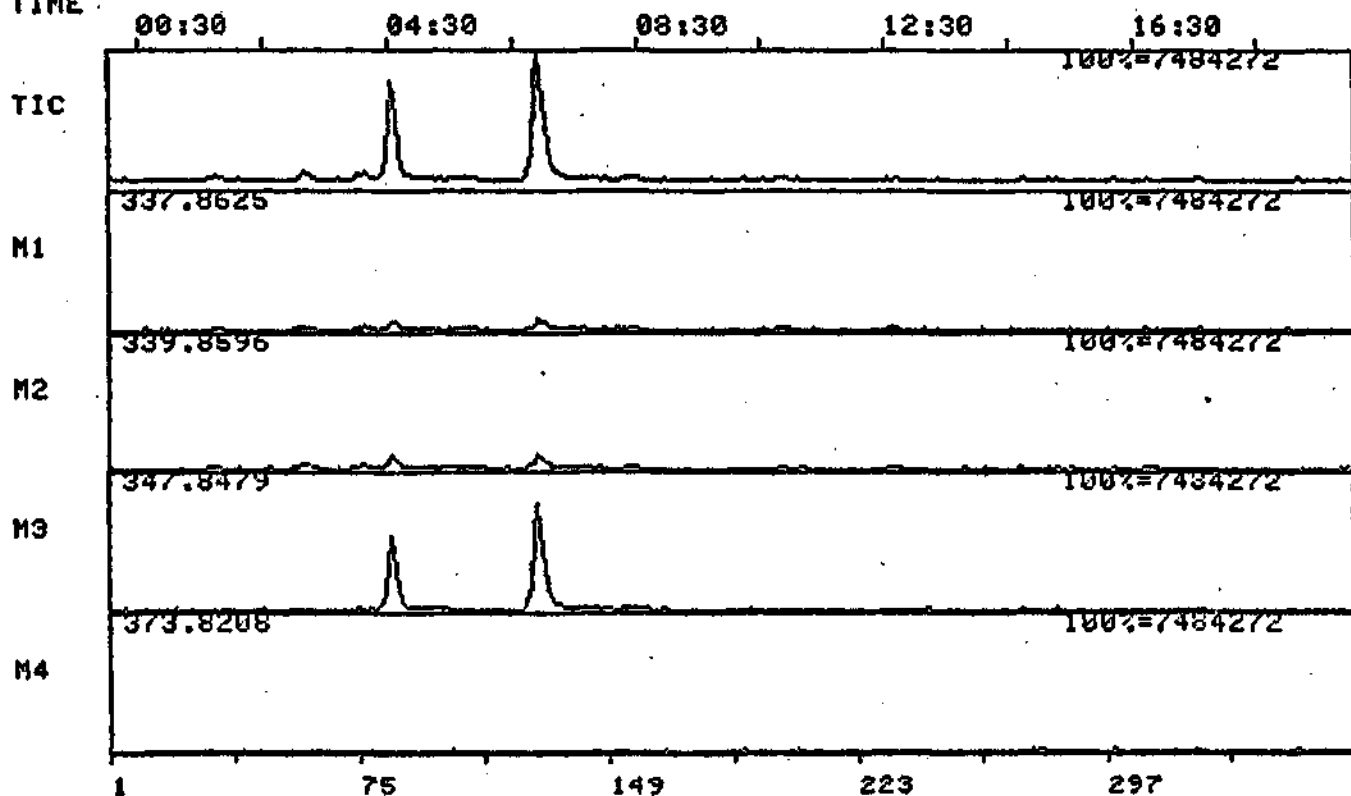
Figure 30: penta+hexa CDFs  
2nd injection  
10n chromatograms  
Sample 1183+1184+11C1

RETN  
TIME



DS55 HIGH RESOLUTION MPM  
RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11

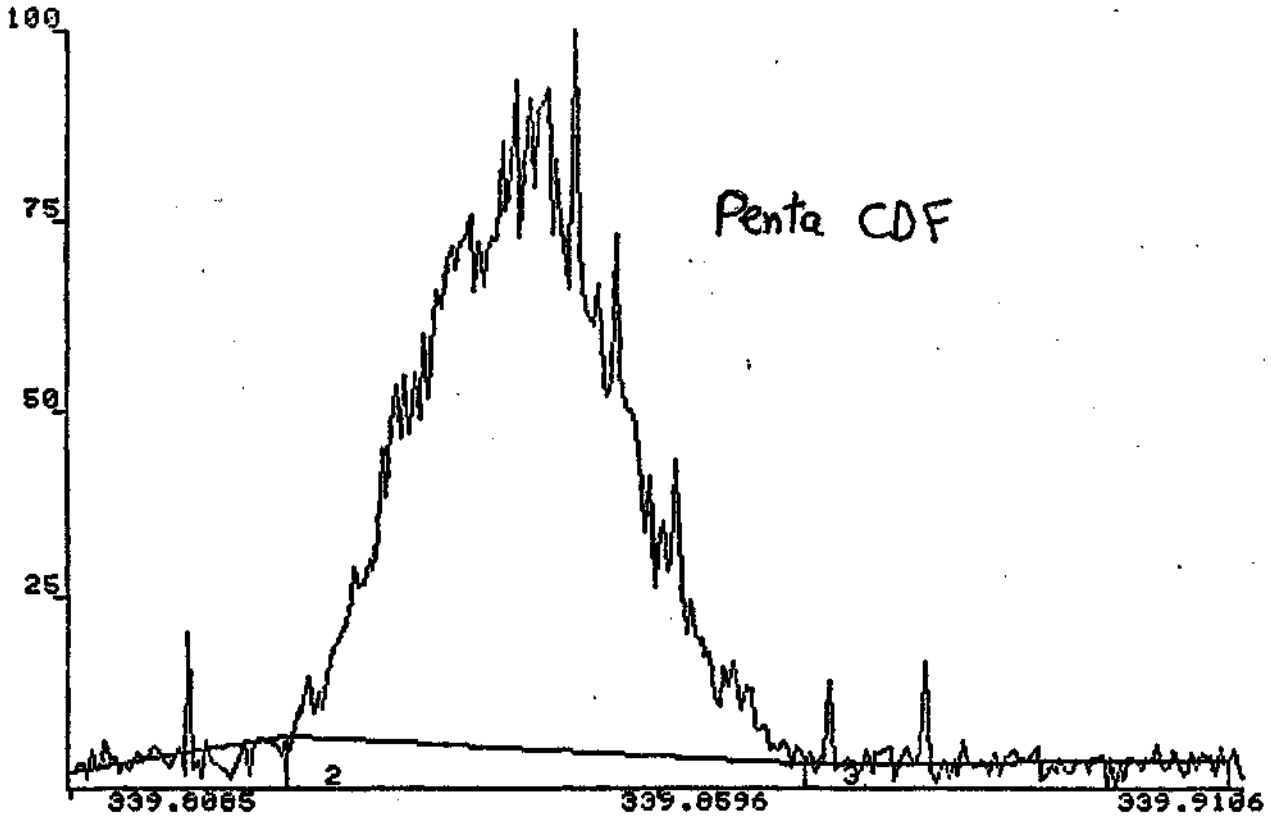
RETN  
TIME



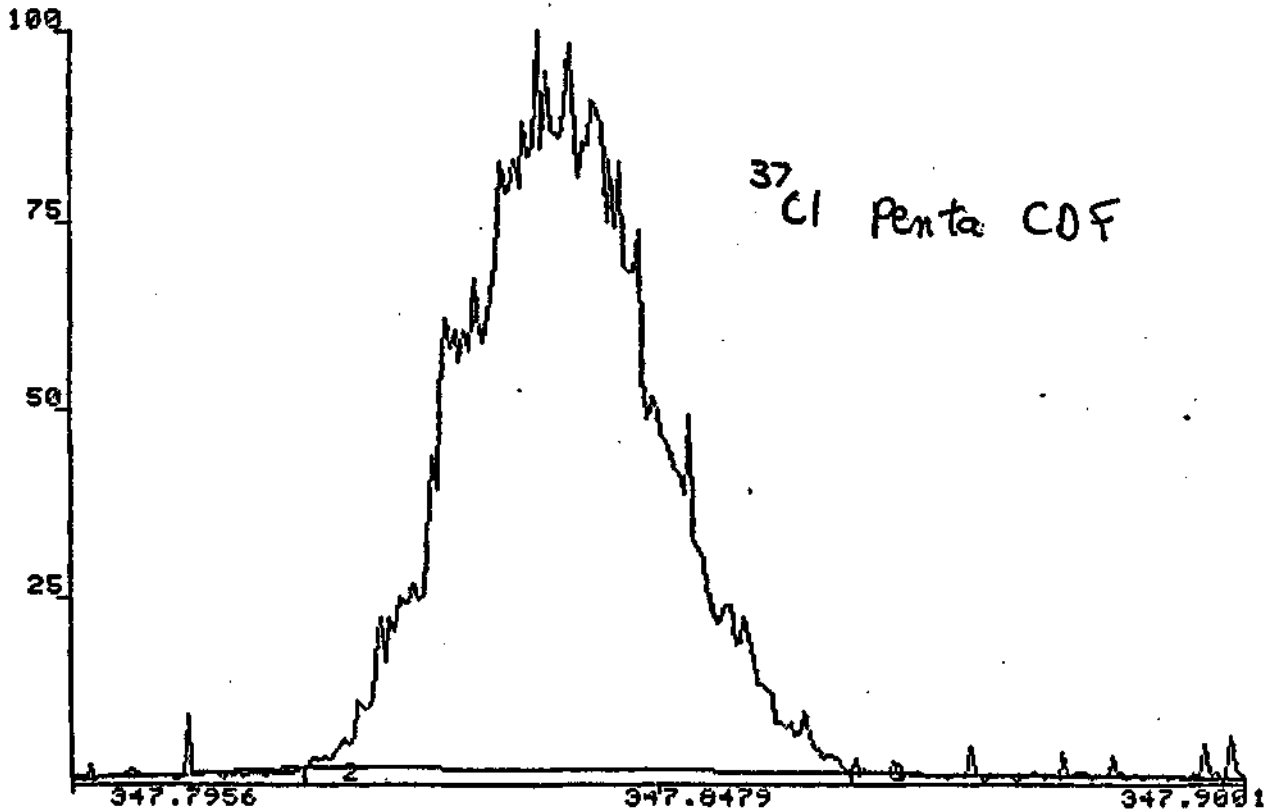
\*

RUNNAME MDHM7      DATE 4/ 4/83      TIME  
MASS 339.8596      SWEEP 300 (PPM)  
SCANS 21-166 100% INTENSITY 282181

Figure 31: Mass profiles - penta CDF  
2nd injection  
Sample 11B3+11B4+11C1



\*SK AREA ID:1      \*  
RUNNAME MDHM7      DATE 4/ 4/83      TIME 12:11  
MASS 347.8479      SWEEP 300 (PPM)      SCANTIME 0.3 (SECS)  
SCANS 21-166 100% INTENSITY 783769



\*SK AREA ID:1      \*

RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11

MASS 337.8626  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 21- 166  
 STANDARD 0.0000  
 FACTOR 0

2.9 OF 5.8UL SAMPLE #'S 11 B3 + B4 + C1

MASS . CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
337.8582	TOTAL	7331942.	YES	NO	29.02	0.00
337.8201	1	35521.	YES	YES	0.14	0.00
337.8496	2	5423822.	YES	YES	21.47	0.00
337.8914	3	59235.	YES	YES	0.23	0.00

RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11  
 MASS 337.8625 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
 SCANS 21-166 100% INTENSITY 215215

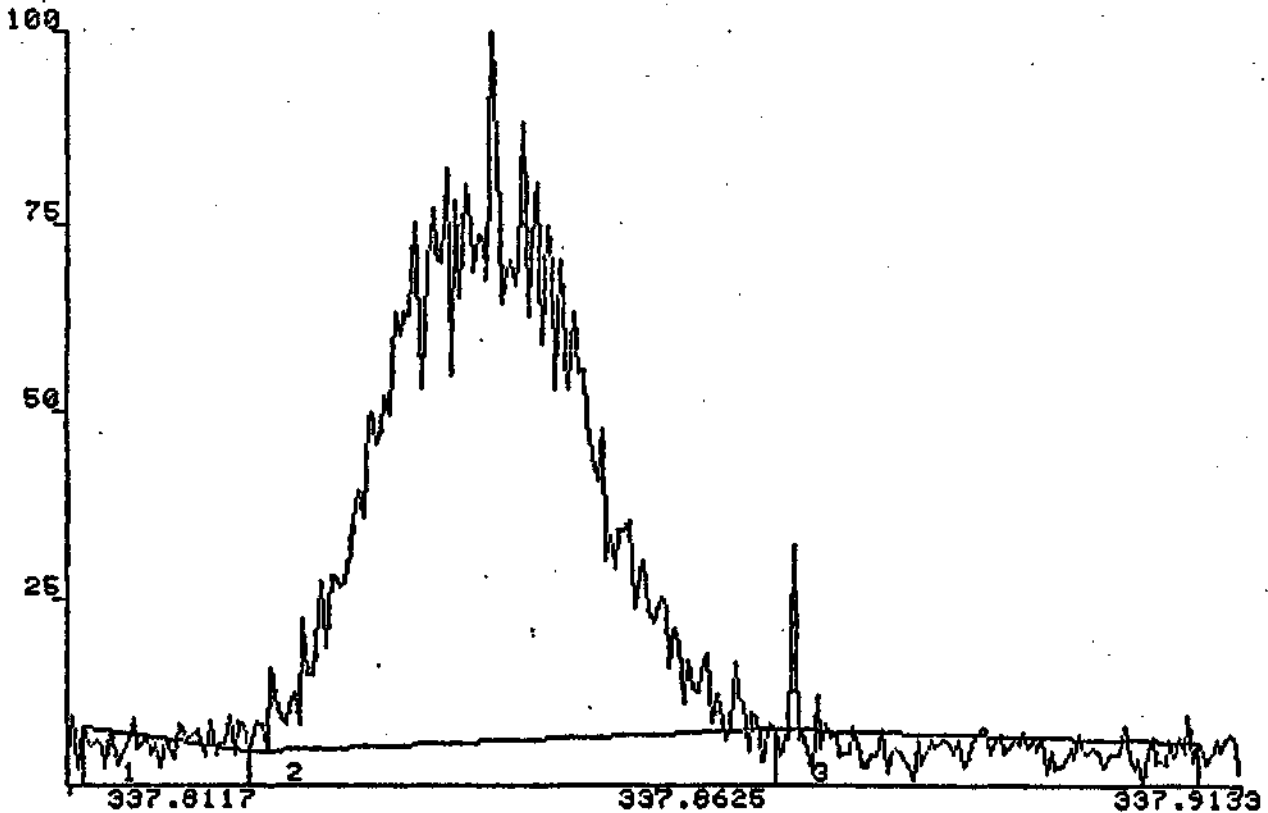


Figure 33: areas - penta CDF  
 Sample 11B3+11B4+11C1

DS55 HIGH RESOLUT

PEAK SUMMATION REPORT

RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11

MASS 339.8596  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 21- 166  
 STANDARD 0.0000  
 FACTOR 0

2.9 OF 5.8UL SAMPLE #'S 11 B3 + B4 + C1

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
339.8555	TOTAL	11009550.	YES	NO	37.11	0.00
339.8179	1	61729.	YES	YES	0.21	0.00
339.8489	2	8781231.	YES	YES	29.60	0.00
339.8909	3	151049.	YES	YES	0.51	0.00

DS55 HIGH RESOLUTION MPM

PEAK SUMMATION REPORT

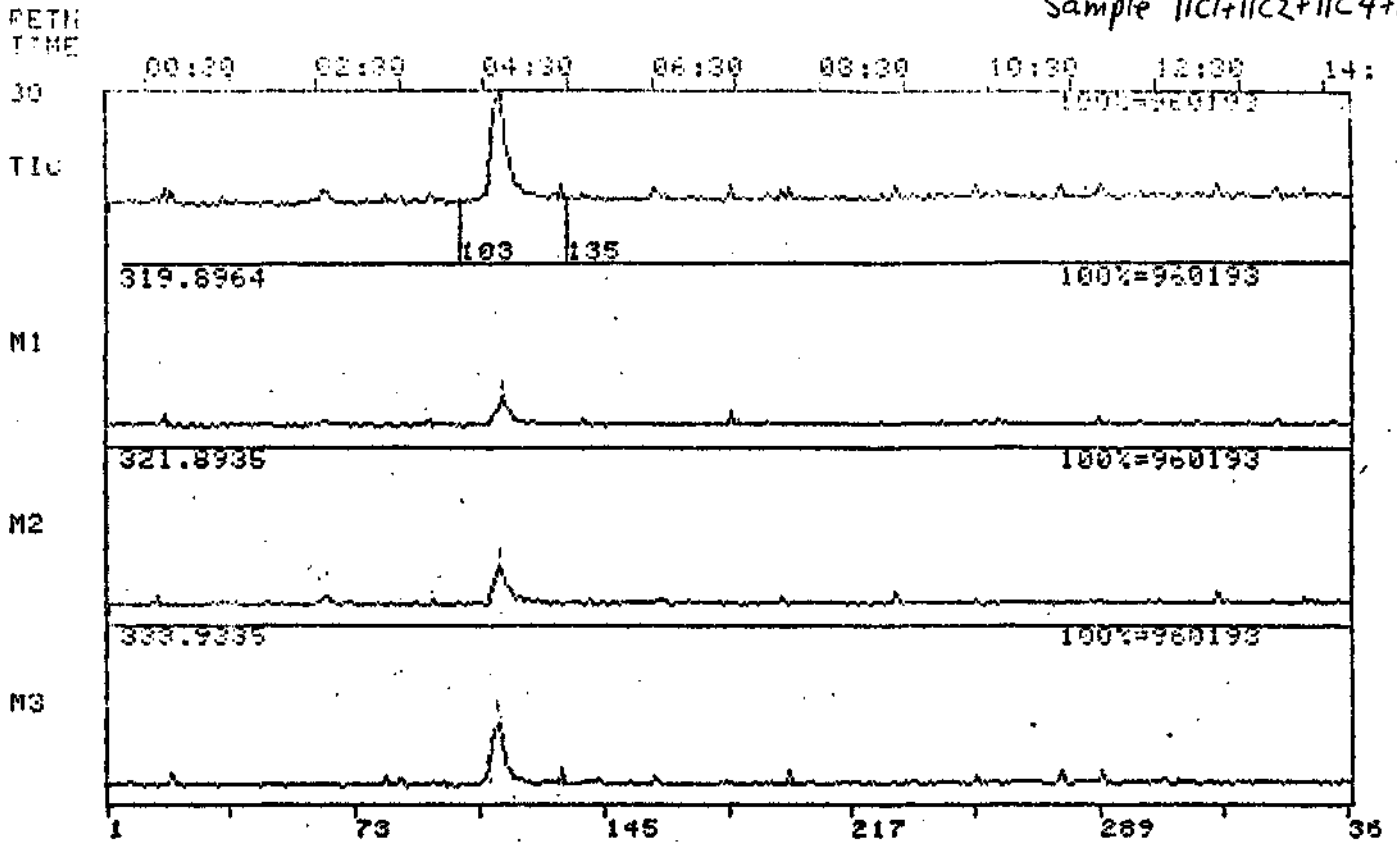
RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11

MASS 347.8479  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 21- 166  
 STANDARD 0.0000  
 FACTOR 0

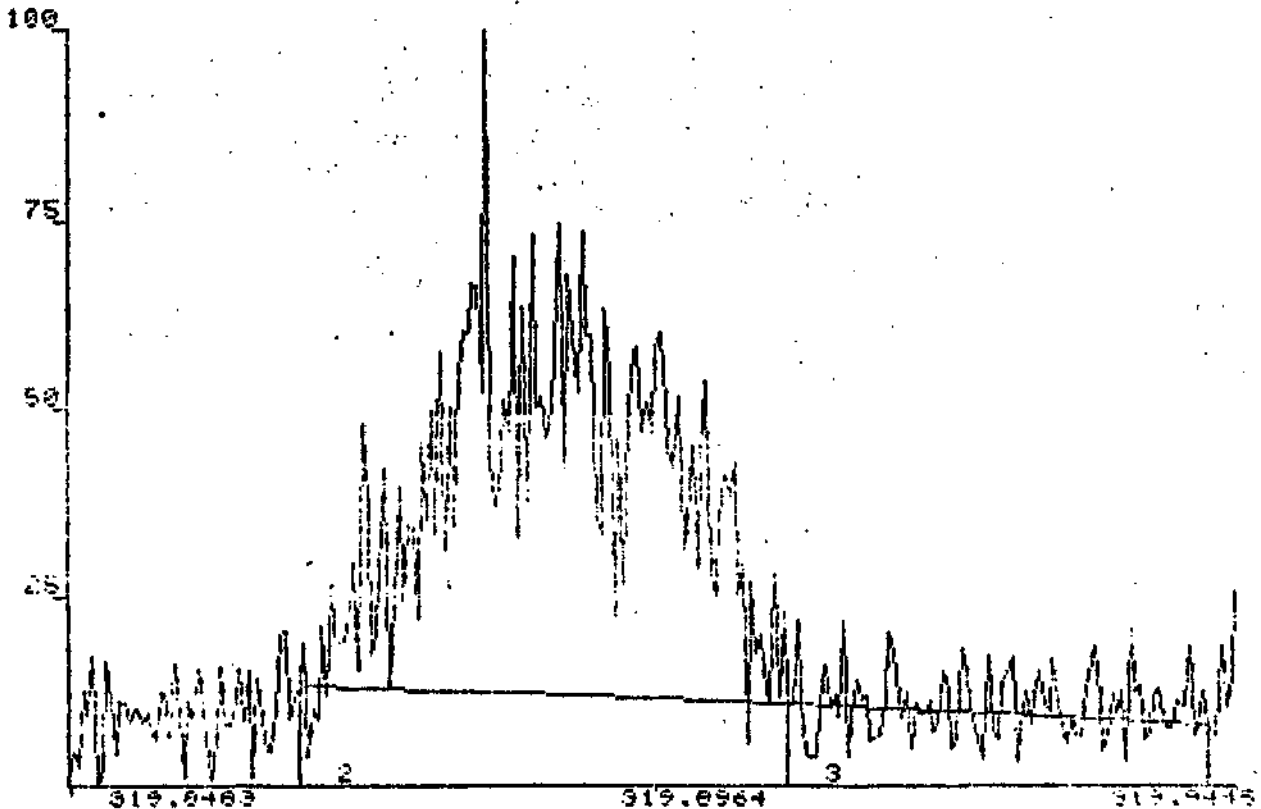
2.9 OF 5.8UL SAMPLE #'S 11 B3 + B4 + C1

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
347.8428	TOTAL	35692510.	YES	NO	65.95	0.00
347.8059	1	93650.	YES	YES	0.17	0.00
347.8394	2	32597840.	YES	YES	60.23	0.00
347.8823	3	304061.	YES	YES	0.56	0.00

Figure 34: TCDDs  
ion chromatograms for  
Sample 11C1+11C2+11C4+11C5

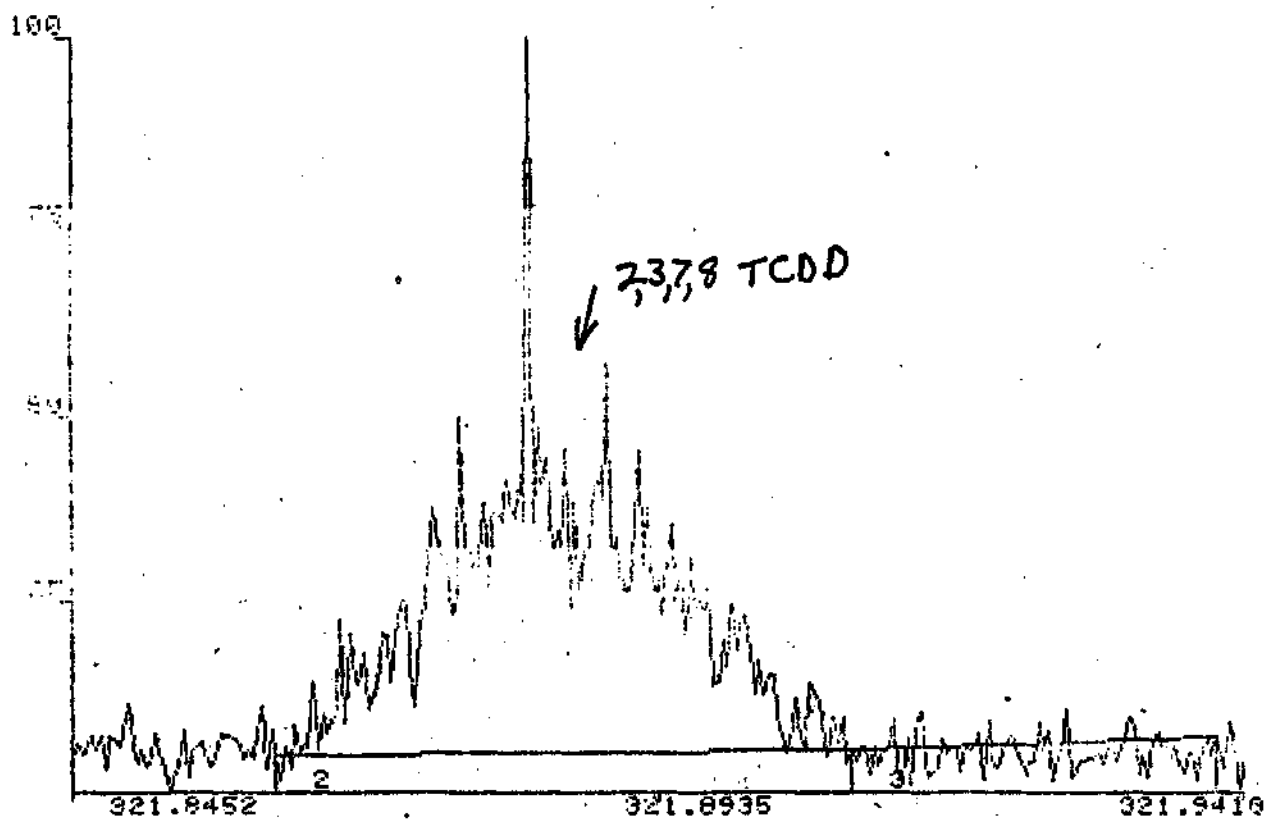


\*DL \*  
RUNNAME FDHM9 DATE 3/25/83 TIME 16:49  
MASS 319.8964 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)  
SCANS 103-135 100% INTENSITY 36335





RUNNAME FDHM9 DATE 3/25/83 TIME Figure 35: mass profile  
 MASS 321.8935 SWEEP 300 (PPM) Sample 11C1+11C2+11C4+11C5  
 SCANS 103-135 100% INTENSITY 58332



DS55 HIGH RESOLUTION NFM  
 PEAK SUMMATION REPORT

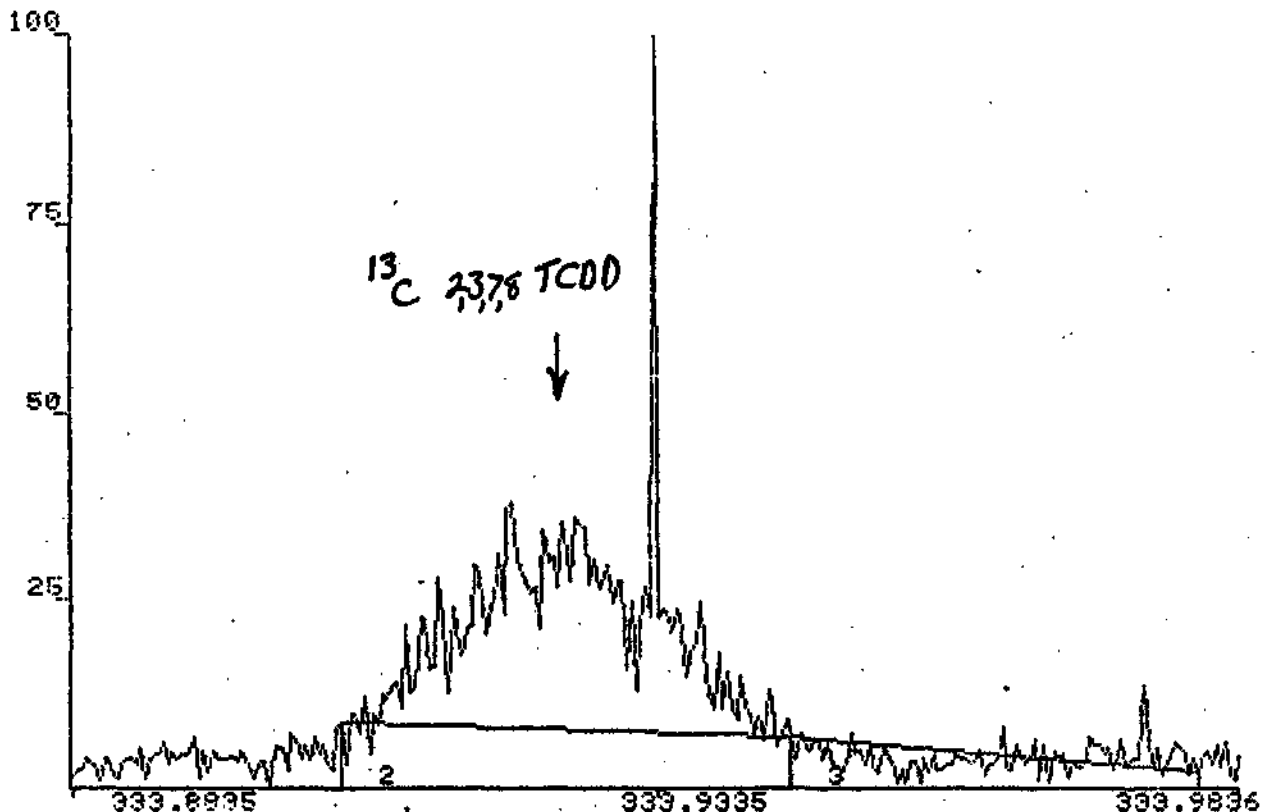
RUNNAME FDHM9 DATE 3/25/83 TIME 16:49  
 MASS 321.8935  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 103- 135  
 STANDARD 0.0000  
 FACTOR 0

11C 1+2+4+5 3.0UL OF 4.4UL

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIPPED	%TOTAL AREA	RELATIVE TO STANDARD
321.8906	TOTAL	1636672.	YES	NO	33.33	0.00
321.8535	1	26722.	YES	YES	0.54	0.00
321.8853	2	1040165.	YES	YES	21.12	0.00
321.9248	3	15910.	YES	YES	0.31	0.00

RUNNAME F0NN9 DATE 3/25/83 TIME  
 MASS 333.9335 SWEEP 300 (PPM)  
 SCANS 103-135 100% INTENSITY 98053

Figure 36: mass profile  
 Sample 11C1+11C2+11C4+11C5



DS55 HIGH RESOLUTION MPM  
 PEAK SUMMATION REPORT

RUNNAME F0NN9 DATE 3/25/83 TIME 16:49

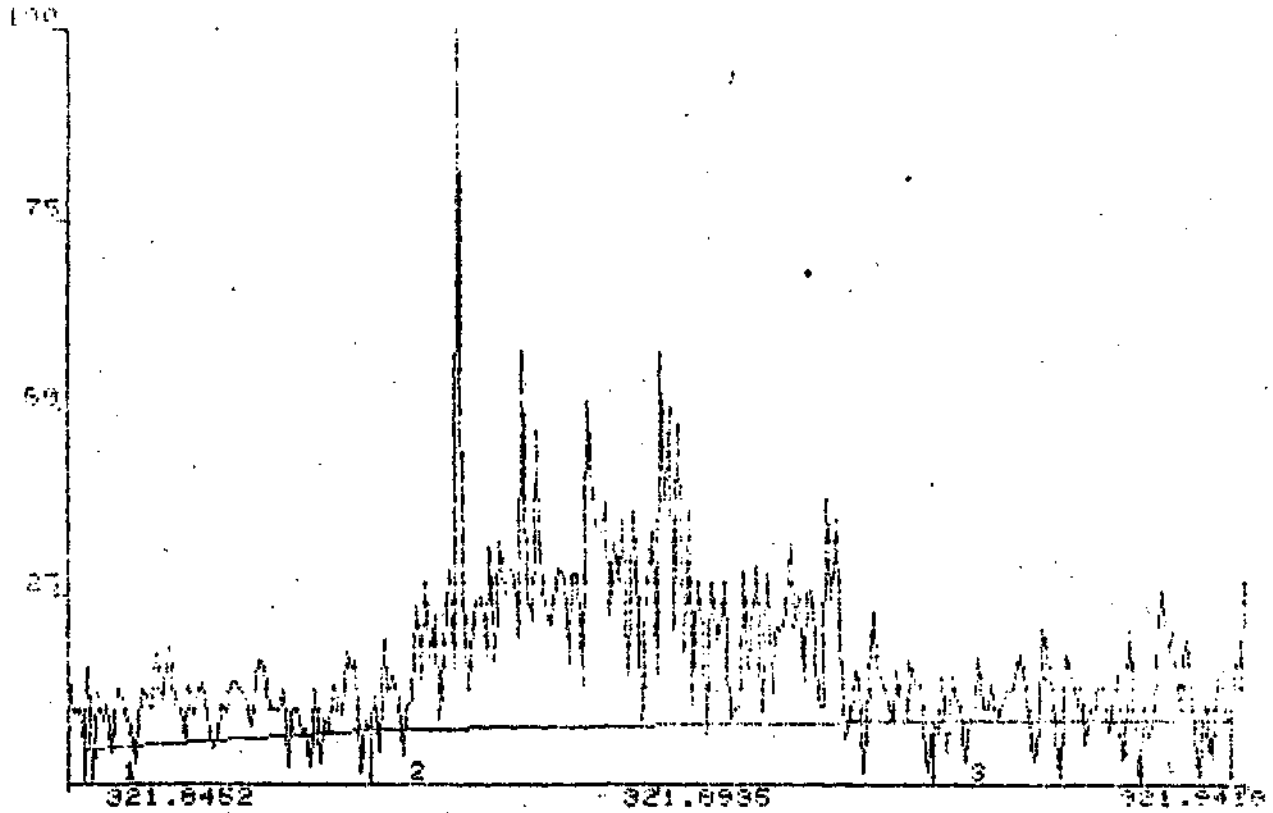
MASS 333.9336  
 SCAN WIDTH 300 PPM  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 103- 135  
 STANDARD 0.0000  
 FACTOR 0

11C 1+2+4+5 3.0UL OF 4.4UL

MASS CENTROID	ITEM	AREA	BASELINE SUBTRACTED	BASELINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
333.9302	TOTAL	2215955.	YES	NO	41.29	0.00
333.8953	1	17444.	YES	YES	0.33	0.00
333.9260	2	1106605.	YES	YES	20.62	0.00
333.9629	3	53728.	YES	YES	1.00	0.00

MODEL 17-1000  
 MASS 321.8935  
 SCANN 100-120 1907 2182 11

Figure 37: Mass profile for solvent blank TCDDs



DS55 HIGH RESOLUTION MPM  
 PEAK SUMMATION REPORT

RUNNAME FDHMS DATE 3/25/83 TIME 14:28

MASS 321.8935  
 SCAN WIDTH 300 MPH  
 SCAN TIME 0.3 SECS  
 SCAN NUMBERS 100-120  
 STANDARD 0.0000  
 FACTOR 0

2.1 OF 2.0UL SOLVENT BLANK (2+3)

MASS CENTROID	ITEM	AREA	BASLINE SUBTRACTED	BASLINE SKIMMED	%TOTAL AREA	RELATIVE TO STANDARD
321.8935	TOTAL	659269.	YES	NO	31.84	0.00
321.8582	1	45387.	YES	YES	2.19	0.00
321.9281	2	196261.	YES	YES	11.09	0.00
321.9293	3	69542.	YES	YES	11.94	0.00