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# 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

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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/m³ 2,3,7,8 TCDF and 52-102 pg/m³ 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/m³. 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/m³, 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 $^{13}C$ 2,3,7,8 TCDD, $^{37}Cl$ OCDD, $^{37}Cl$ 2,3,7,8 TCDF and $^{37}Cl$ penta-CDF (3 isomers), 50 m³ 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 (<.3μ) 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 pg/m³ 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 m³ 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 pg/m³ 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 pg $^{13}C$ 2,3,7,8 TCDD, 5000 pg $^{37}Cl$ OCDD and 1200 pg $^{37}Cl$ 2,3,7,8 TCDF), 24000 pg of $^{37}Cl$ penta-CDF (3 unspiked

isomers) in benzene was deposited directly onto the silica gel (activated at 140°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 µL for GC/MS injection.

Capillary GC/HRMS

In summary: 2 µL 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° for 3 min, 60°/min to 190°, 2.5°C/min to 240° hold. Kratos MS-50 mass spectrometer was used in the EI mode (70 ev), approx. 10,000 resolution (10% valley), source 250°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 $^{37}Cl$ 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 pg/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 intepreting 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 pg/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 μ) 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 pg/m³ (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 pg/m³.

To obtain adequate sensitivity for TCDDs, four 50 m³ 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 pg/m³ 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 asignificant concentration). The HR single ion chromatograms and mass profiles for a combined BSOB air sample 11C1 + 11C2 + 11C4 + 11C5 (figs. 35-37) showed 226 pg 2,3,7,8 TCDD equivalent to 0.94 pg/m³ air (also possible traces of other TCDDs). A quality control (120 m³ Albany air) sample fortified with 98 pg native 2,3,7,8 TCDD to 0.82 pg/m³ air was analyzed and found to have a concentration of 1.3 pg/m³. A solvent blank was similarly analyzed and found to contain an rather high 47 pg 2,3,7,8 TCDD equivalent to 0.39 pg/m³ 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 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 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.

INDEX TO FIGURES

Table 1. TETRA-CDFs

| Floor/sample[1] | Volume $m^3$ | Concentration of 2378 TCDF ($pg/m^3$) | Detection limit ($pg/m^3$) | Ion[2] ratio(%) 304/306 | Relative retention time | $^{37}Cl$ TCDF recovery (%) | Total TCDF ($pg/m^3$) | Detection limit ($pg/m^3$) | Ion[2] 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 $pg/m^3$ 2378 TCDF Fortified background air | 41.8 | 9.9 | 0.4 | 79 | 1.0 | 120 | 10.2 | 2.1 | — | 97 |
| DOH1 8.1 $pg/m^3$ 2378 TCDF Fortified background 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 µL) volumes prior to injection.

Table 2 PENTA CDFs

| Floor/Sample | Total Penta-CDF ($pg/m^3$) | Detection Limit ($pg/m^3$) | Ion ratio (%) 338/340 (2) |
|---|---|---|---|
| 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 (1) | 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 $^{37}Cl$ 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/m3

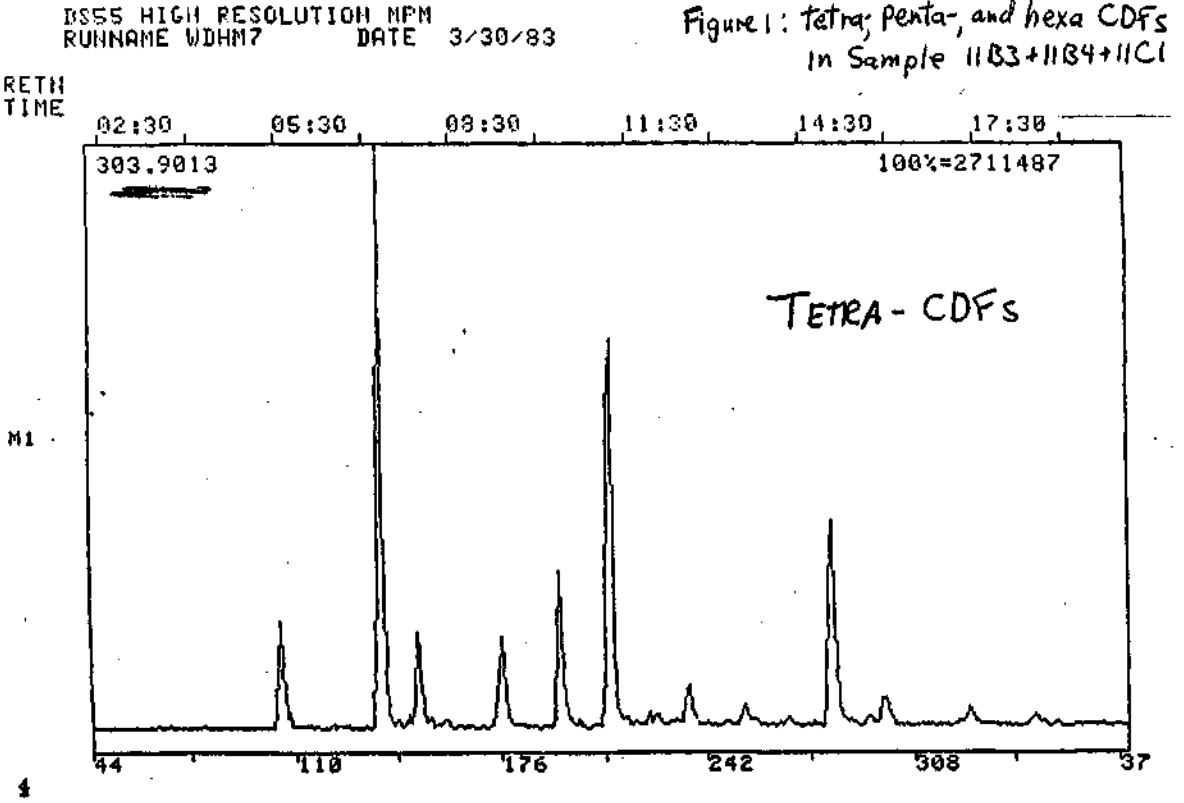
Table 3

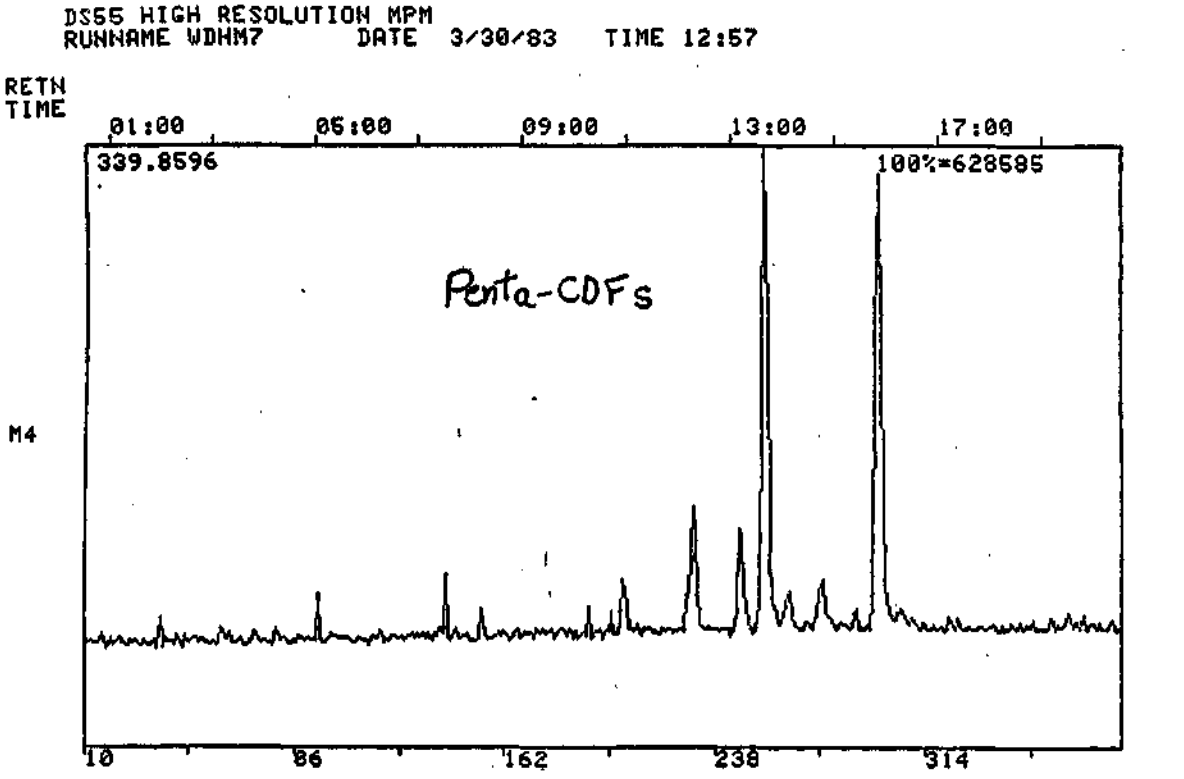
| Floor/sample | Air volume ($m^3$) | m/e 322 area (counts) | pg found | 2378 TCDD[1] pg/$m^3$ | Detection limit (pg) | Ratio[2] 320/322 | $^{13}C$ TCDD recovery (%) | GC relative retention time |
|---|---|---|---|---|---|---|---|---|
| 11C1 + 11C2 + 11C4 + 11C5 | 241 | 1040165 | 226 | 0.94 | 12 | 0.66 | 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/$m^3$) | 120 | 706423 | 150 | 1.3 | 13 | 0.89 | 78 | 1.00 |

(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

Figure 1: tetra, penta-, and hexa CDFs in Sample 1183+1184+11C1



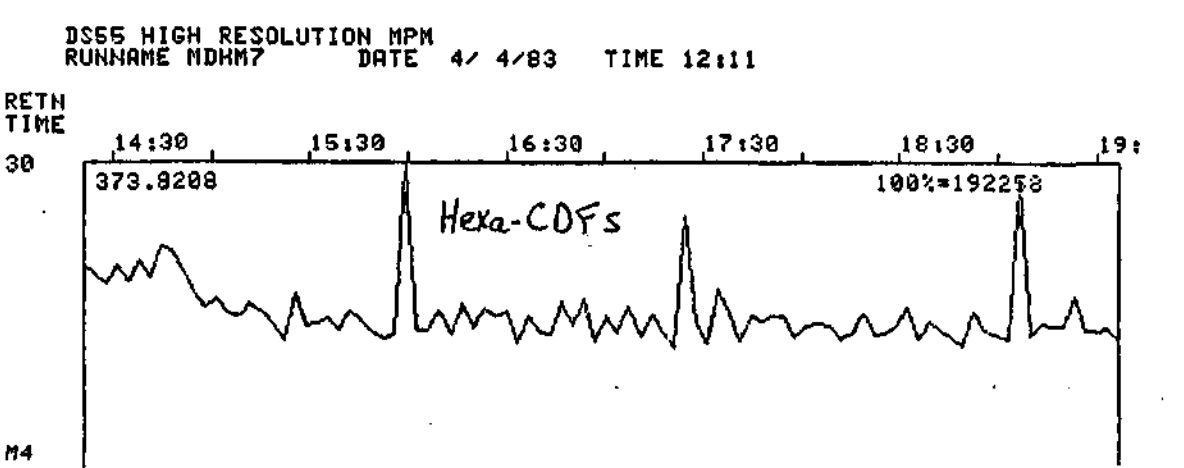
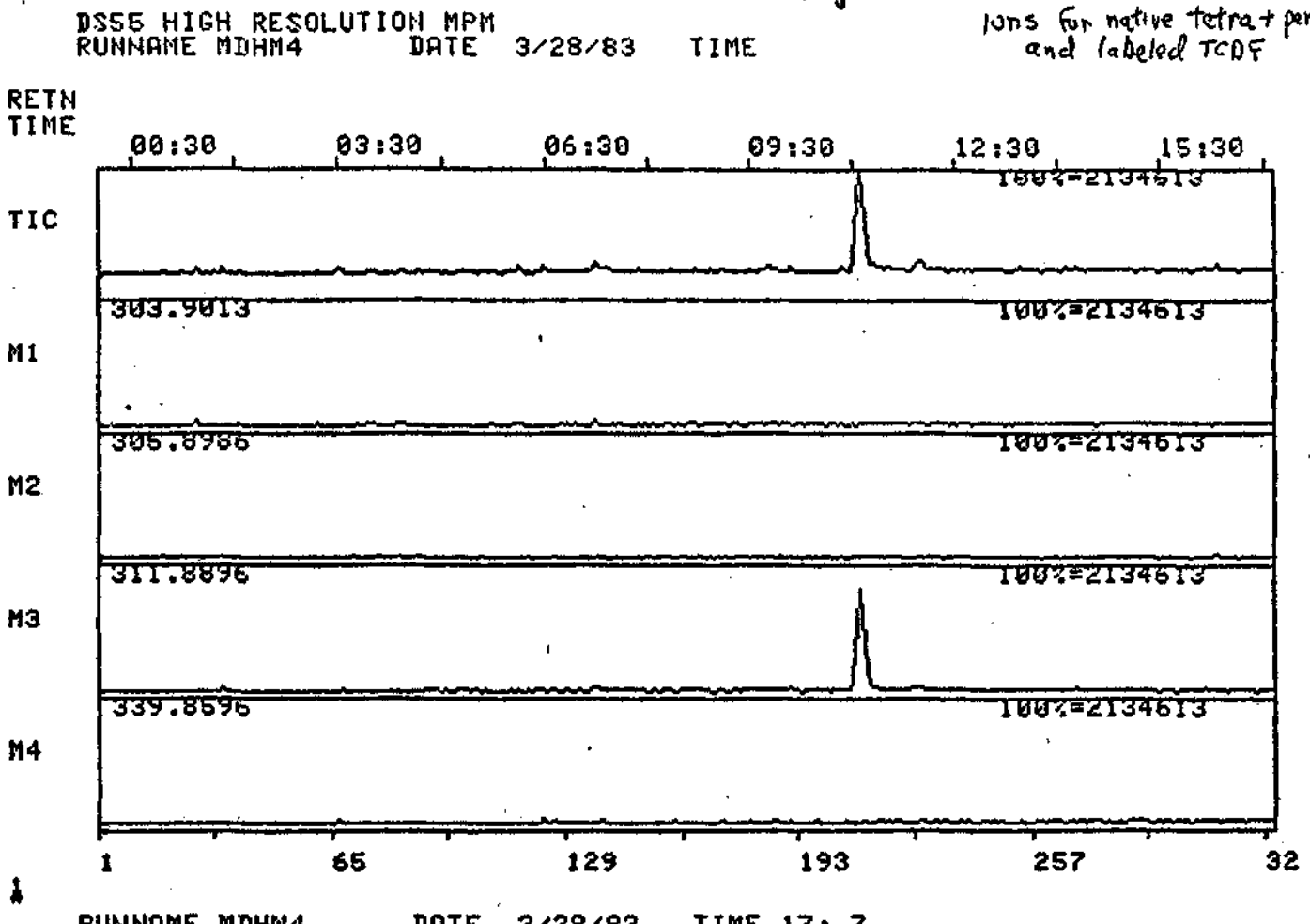
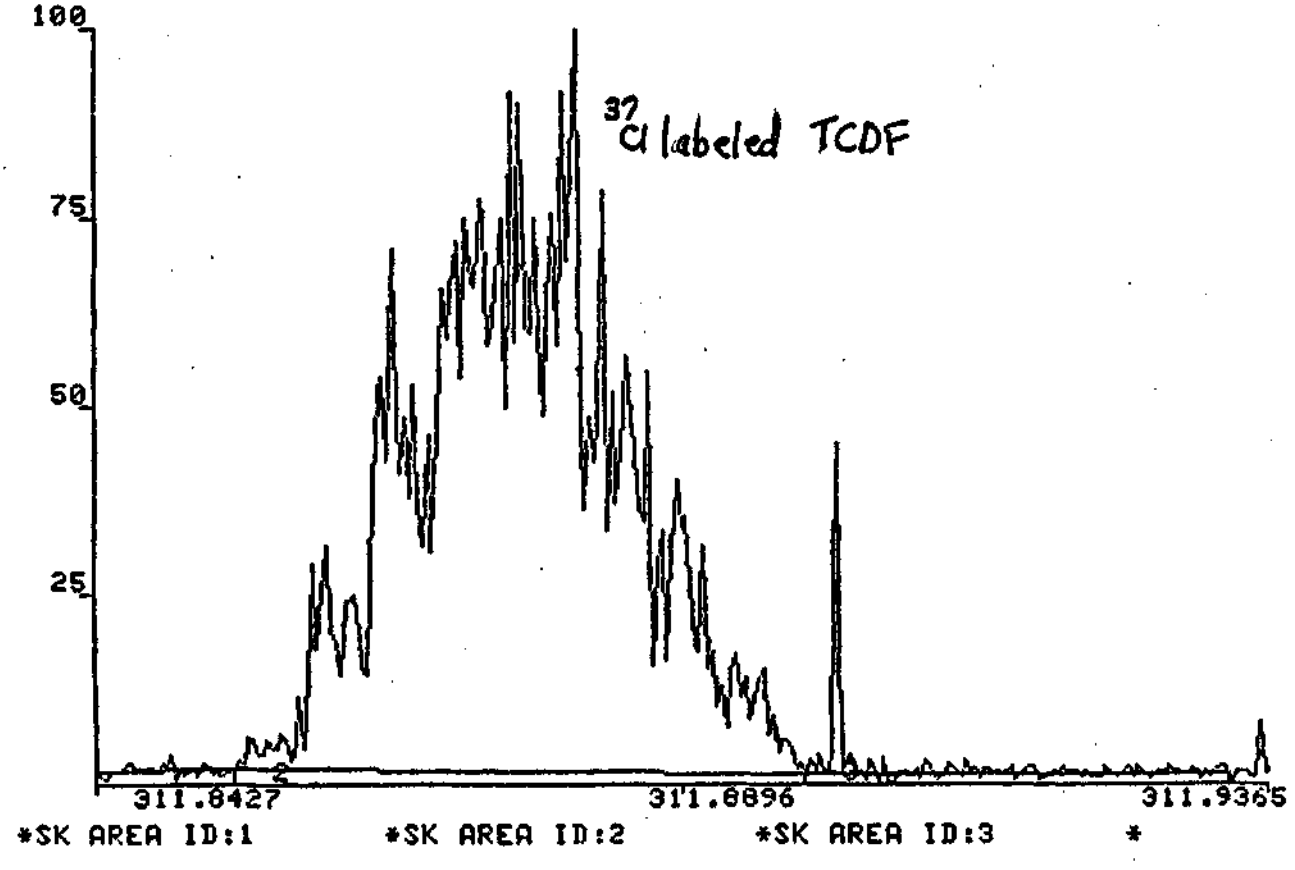
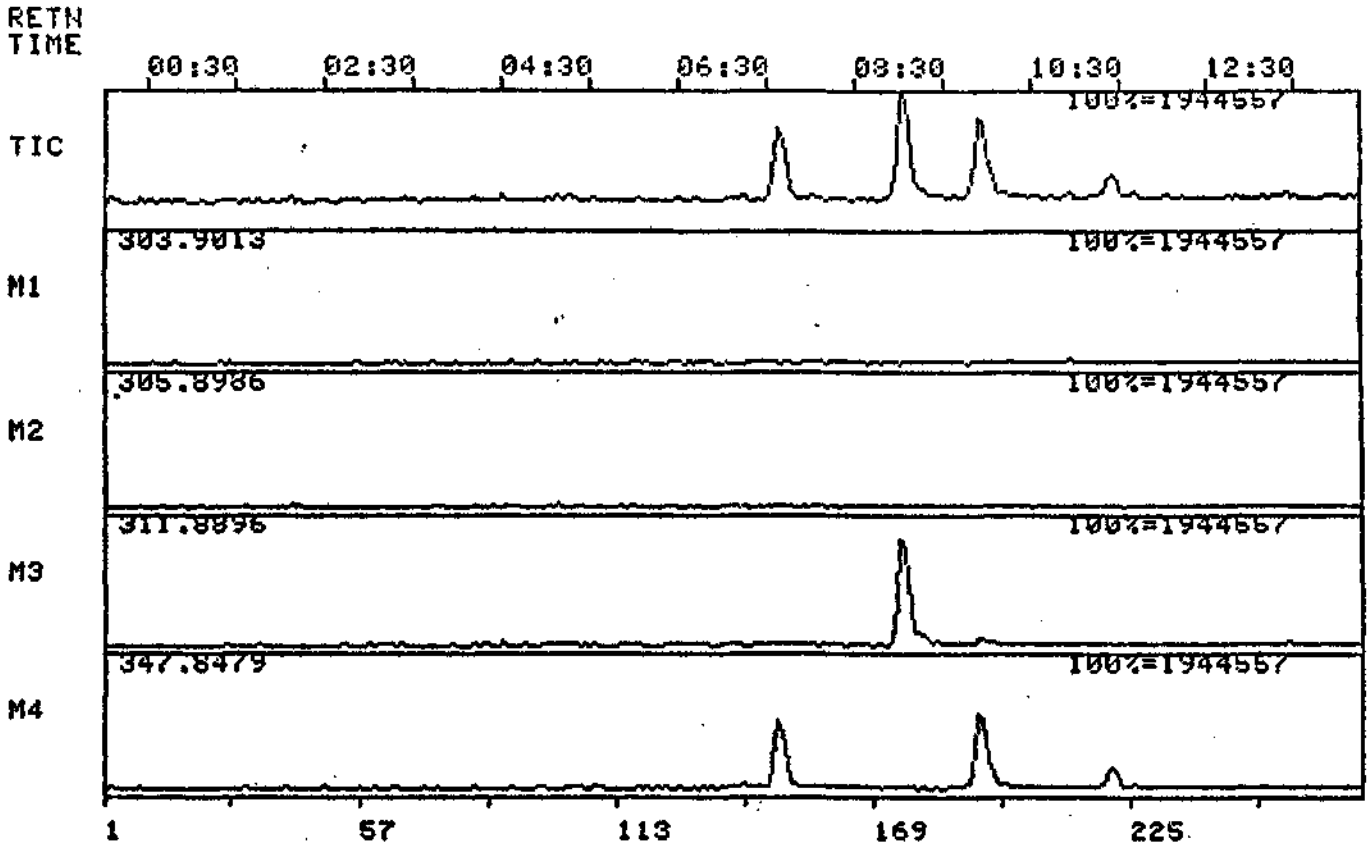



Figure 2 : mixed labeled standard ions for native tetra + penta CDF and labeled TCDF



DS55 HIGH RESOLUTION MPM
RUNNAME MDHM1 DATE 3/28/83 TIME Figure 3: mixed labeled Standard

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SCANS 94-231 100% INTENSITY 203331

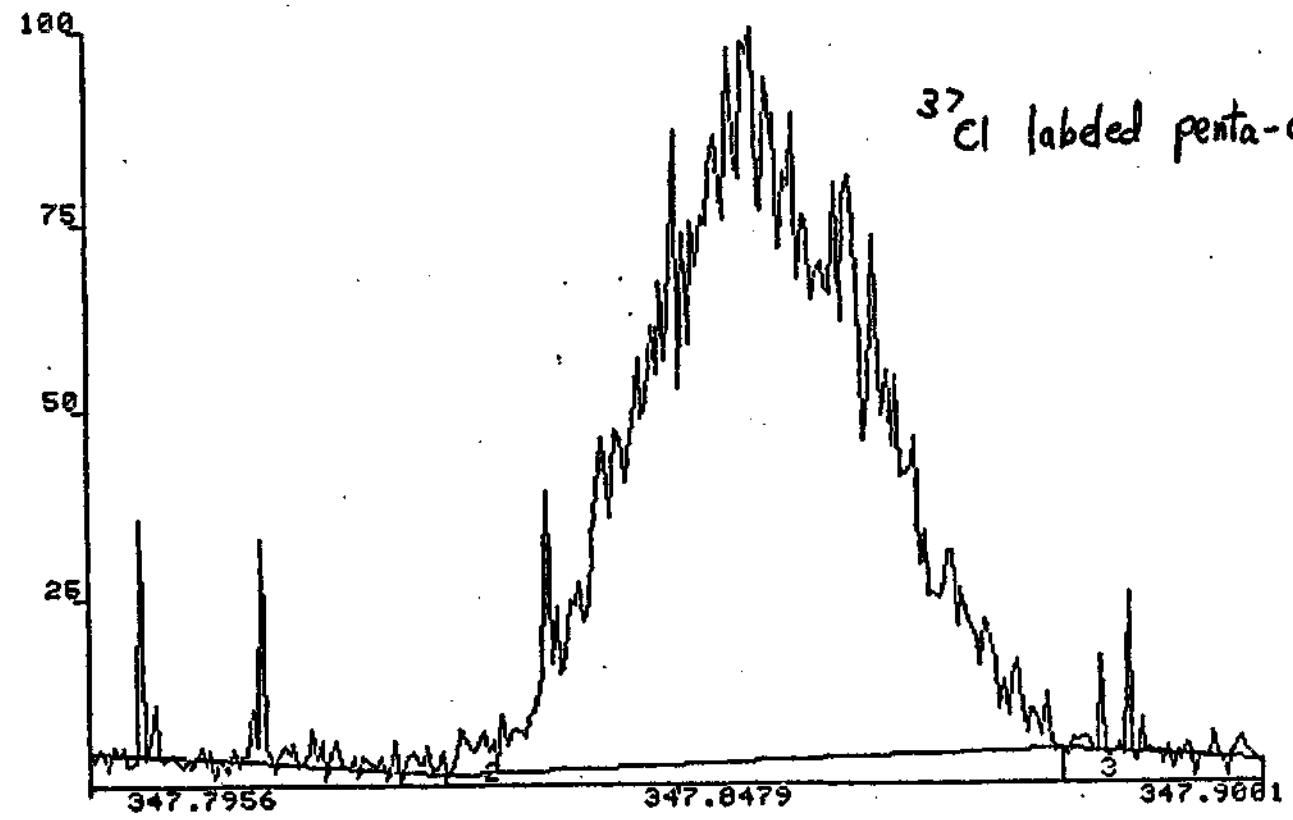


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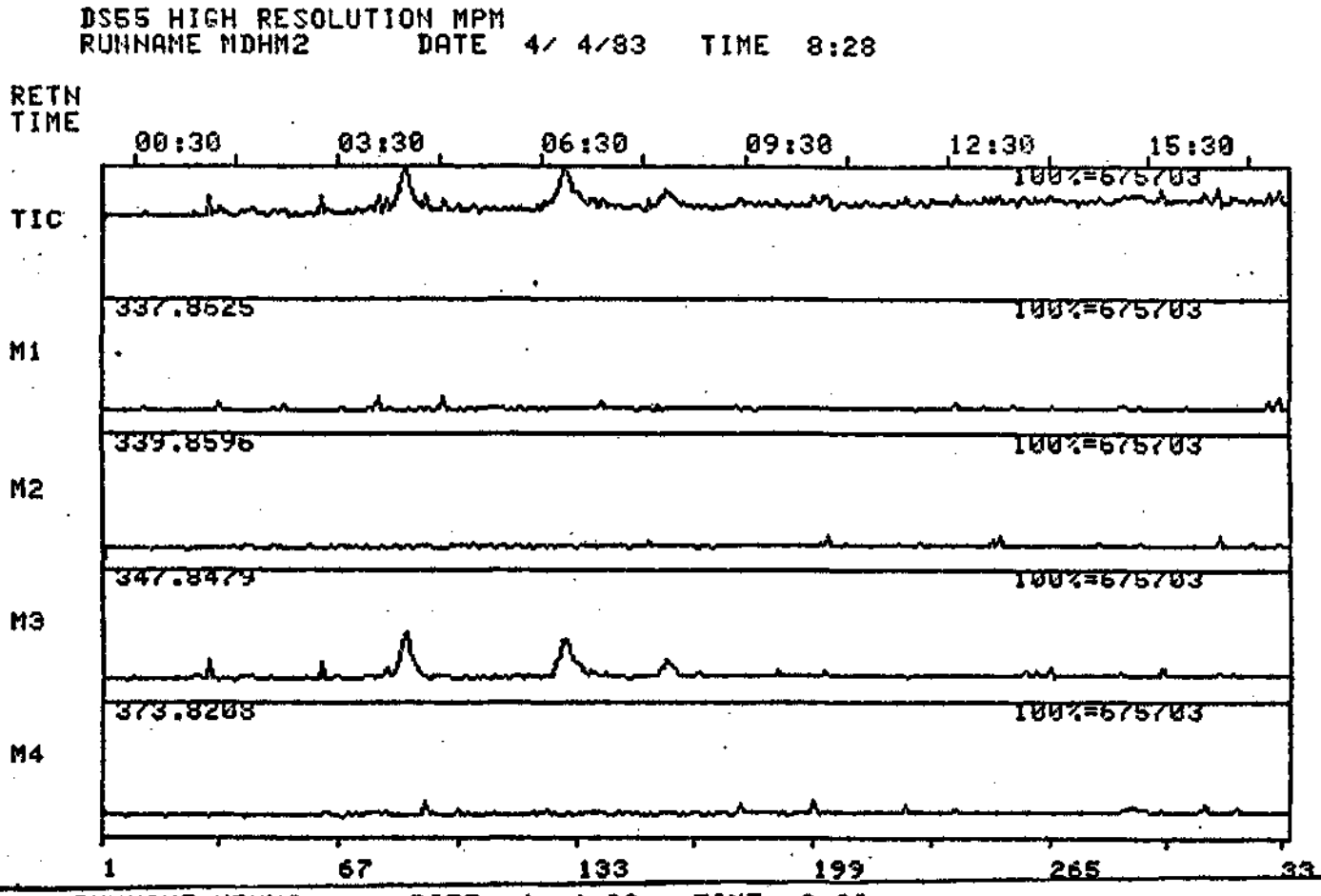
Figure 4: mixed labeled standard + native hexa CDF added



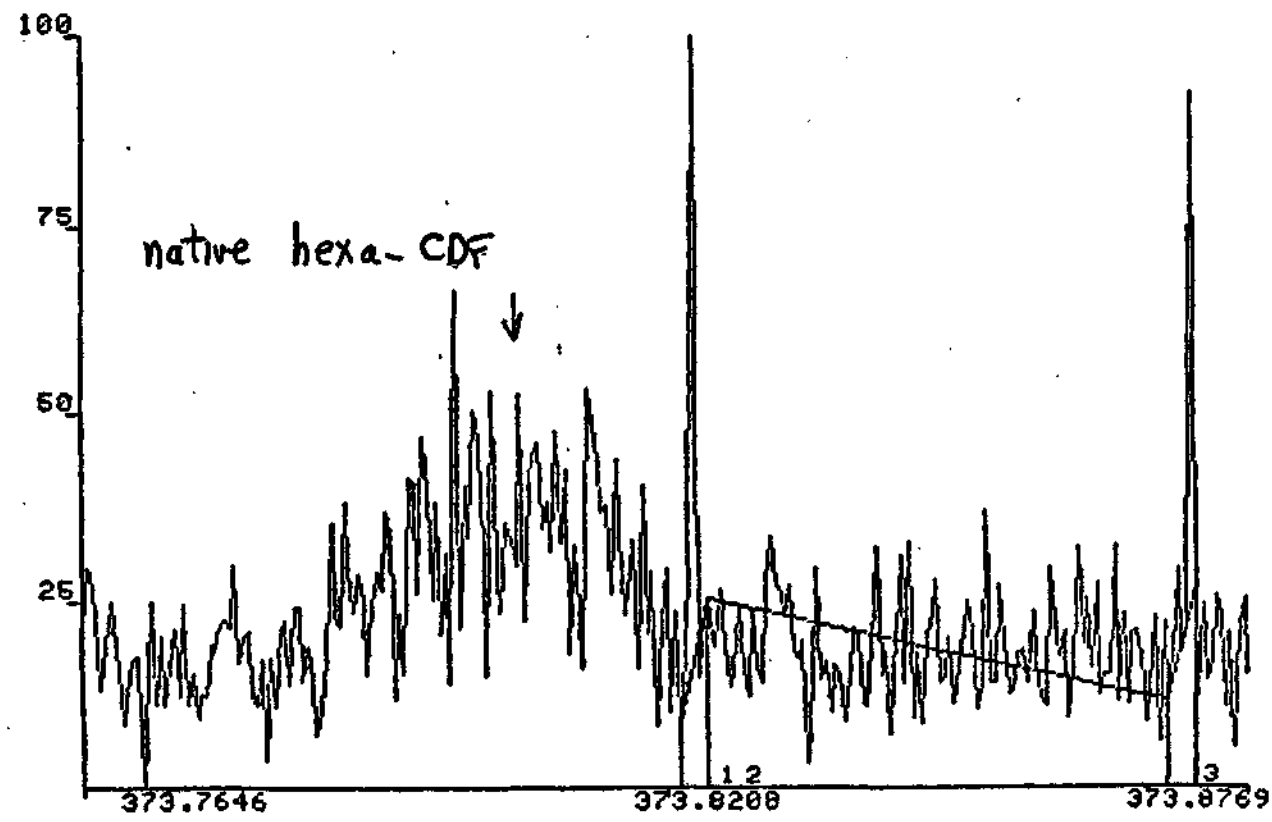




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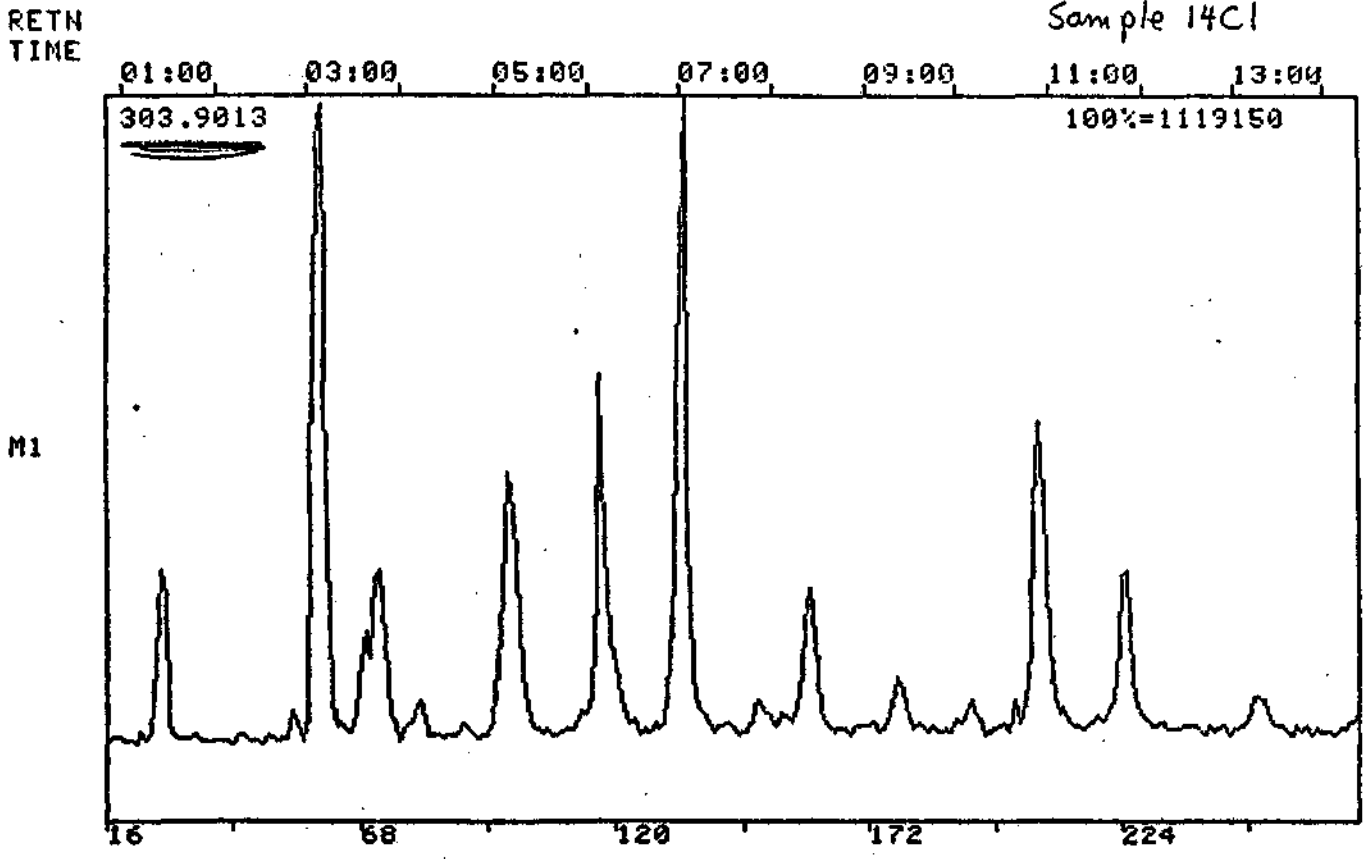
Figure 5: Tetra CDFs in Floor 14
Sample 14C1

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14C1

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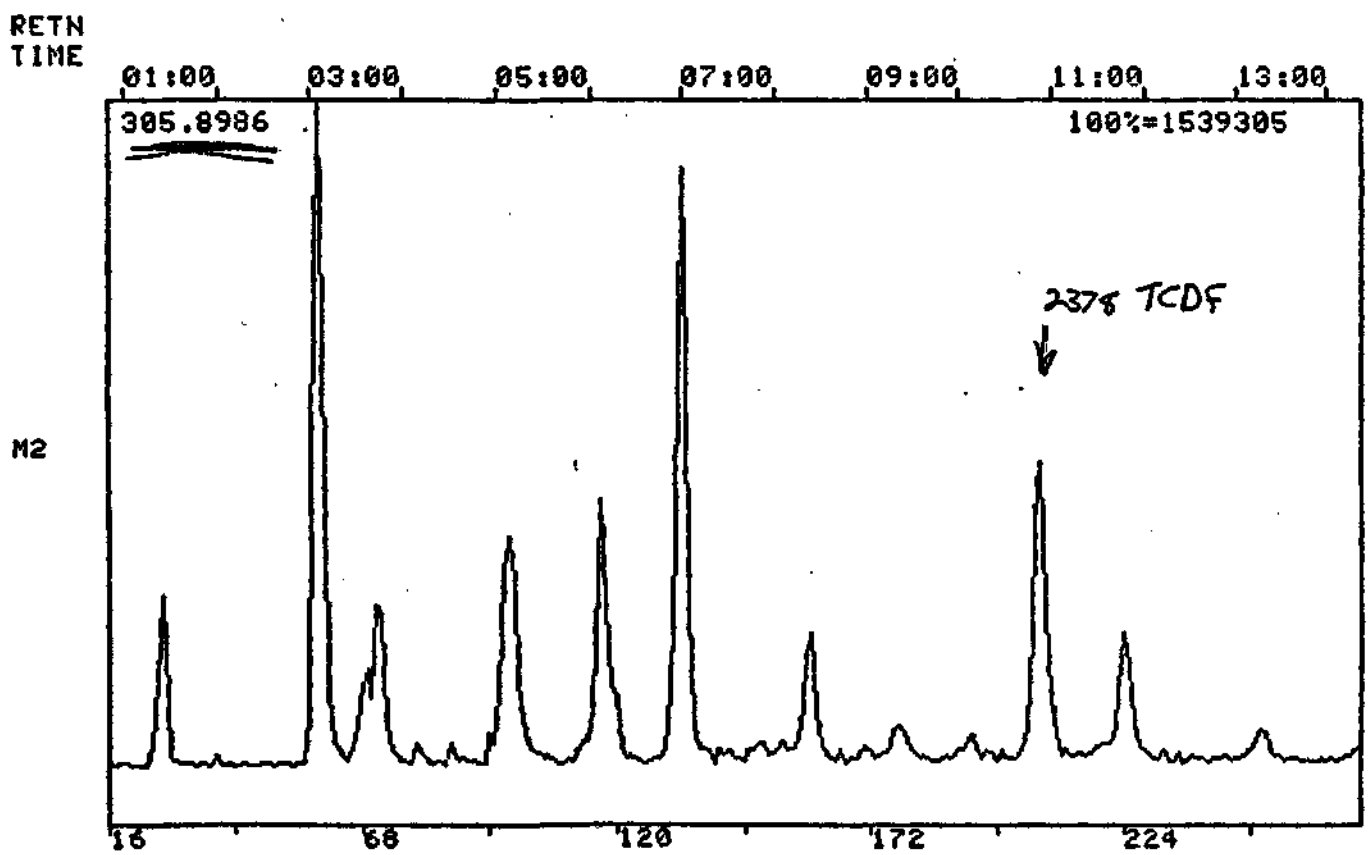


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DS55 HIGH RESOLUTION MPM
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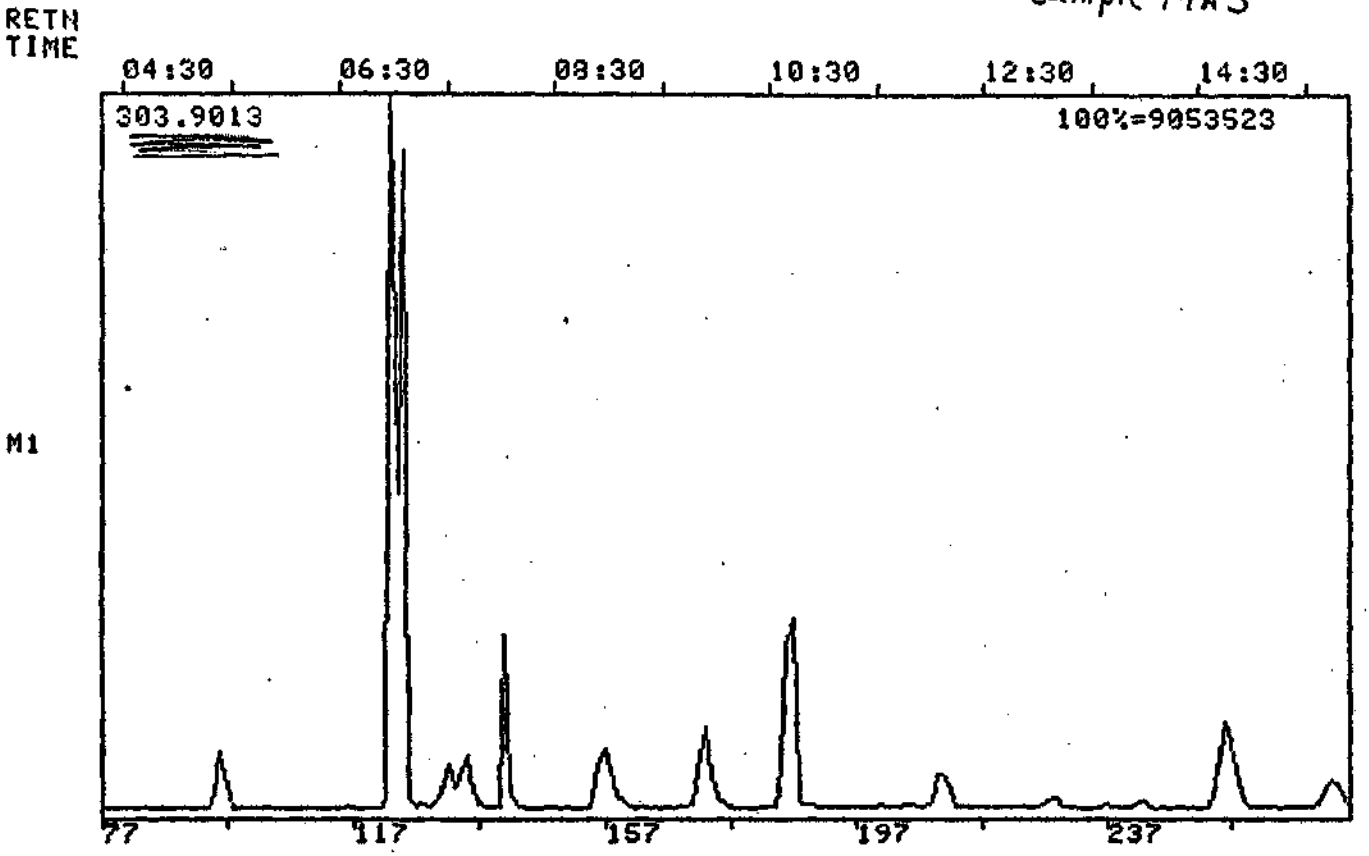
Figure 6: Tetra CDFs in Flour 14
Sample 14A3

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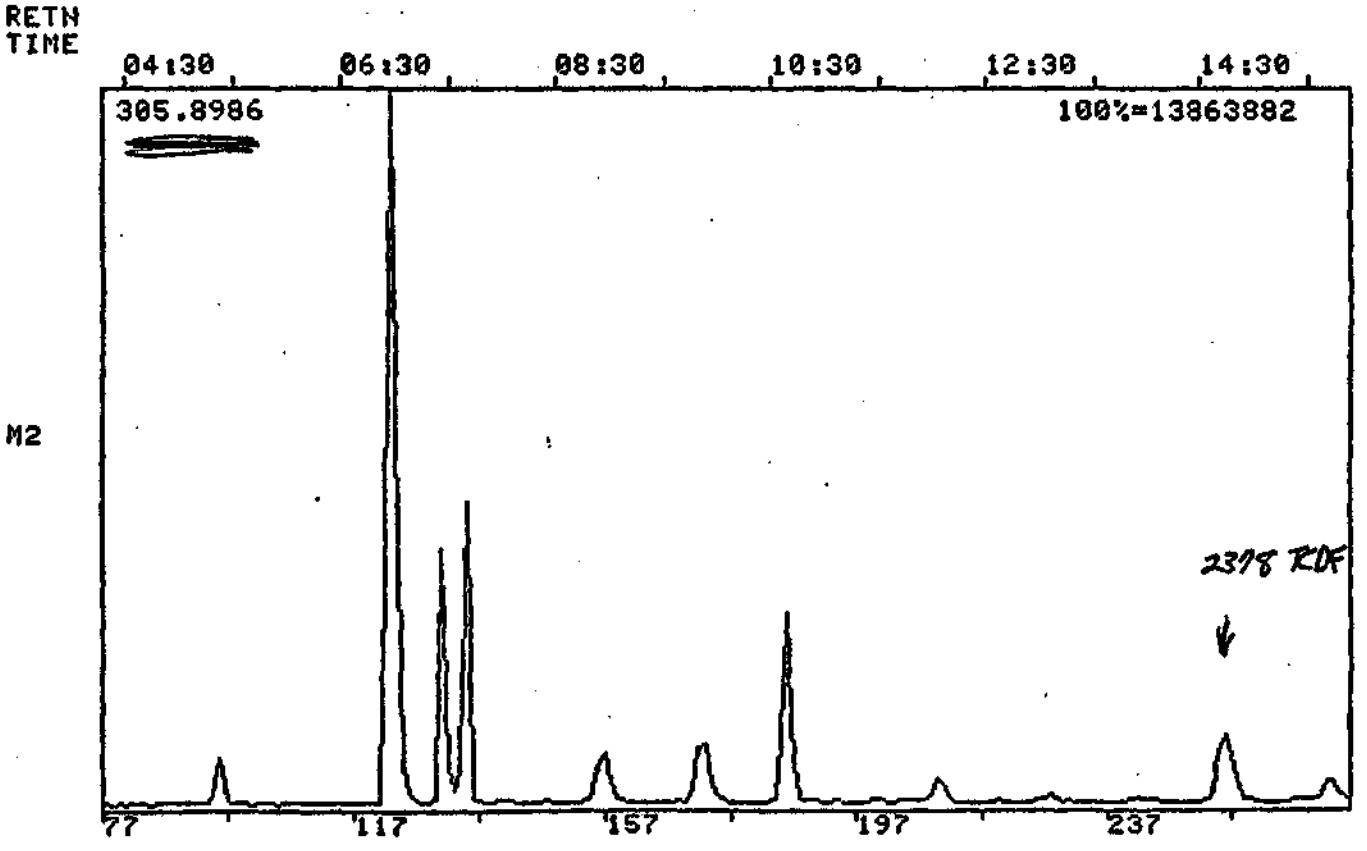


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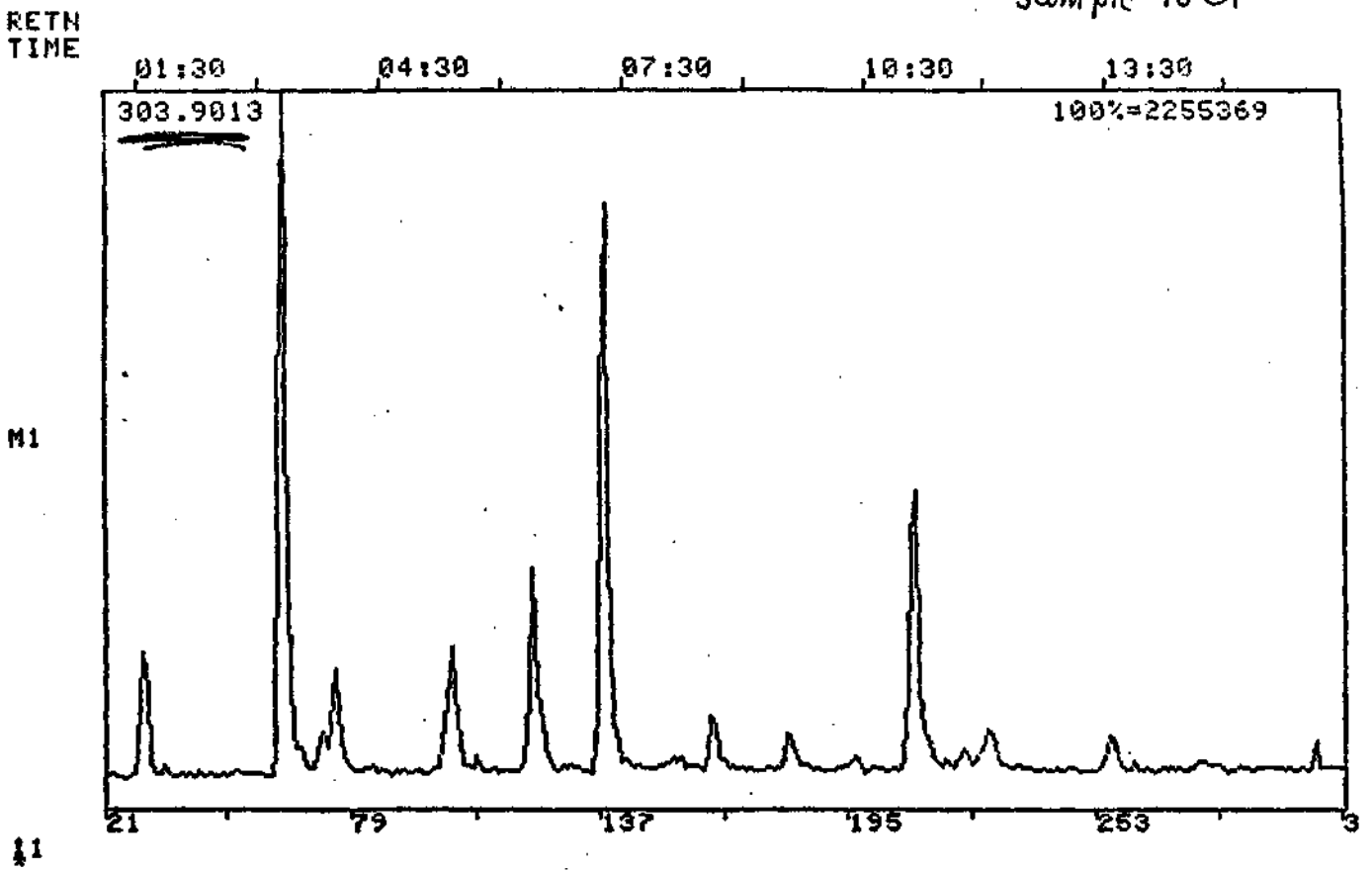
DS55 HIGH RESOLUTION MPM
RUNNAME MDHM3 DATE 3/28/83 TIME

Figure 7: Tetra CDFs in Floor 16 Sample 16 C1

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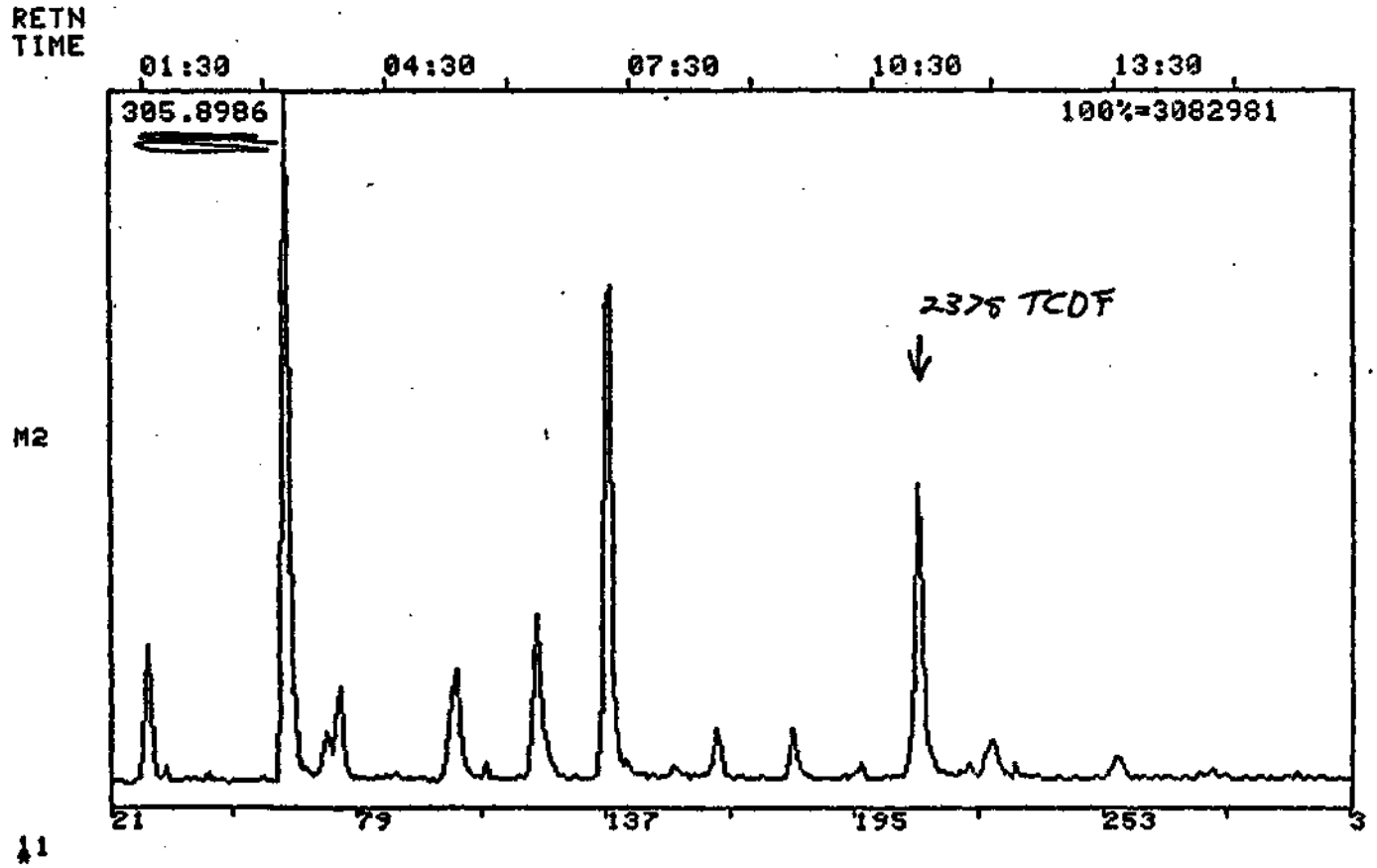

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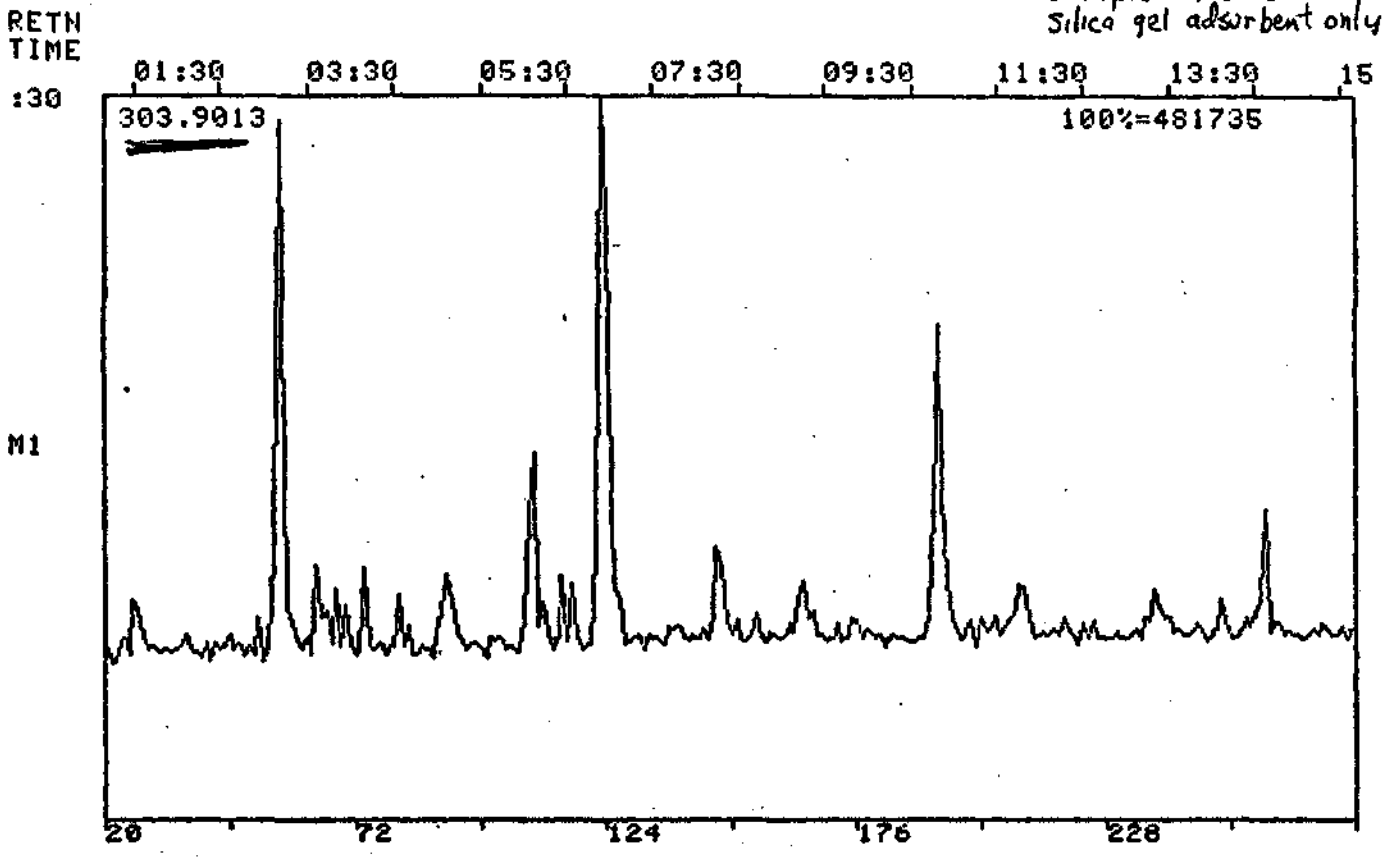
Figure 8: tetra CDFs in Floor 17
Sample 17C1G
Silica gel adsorbent only

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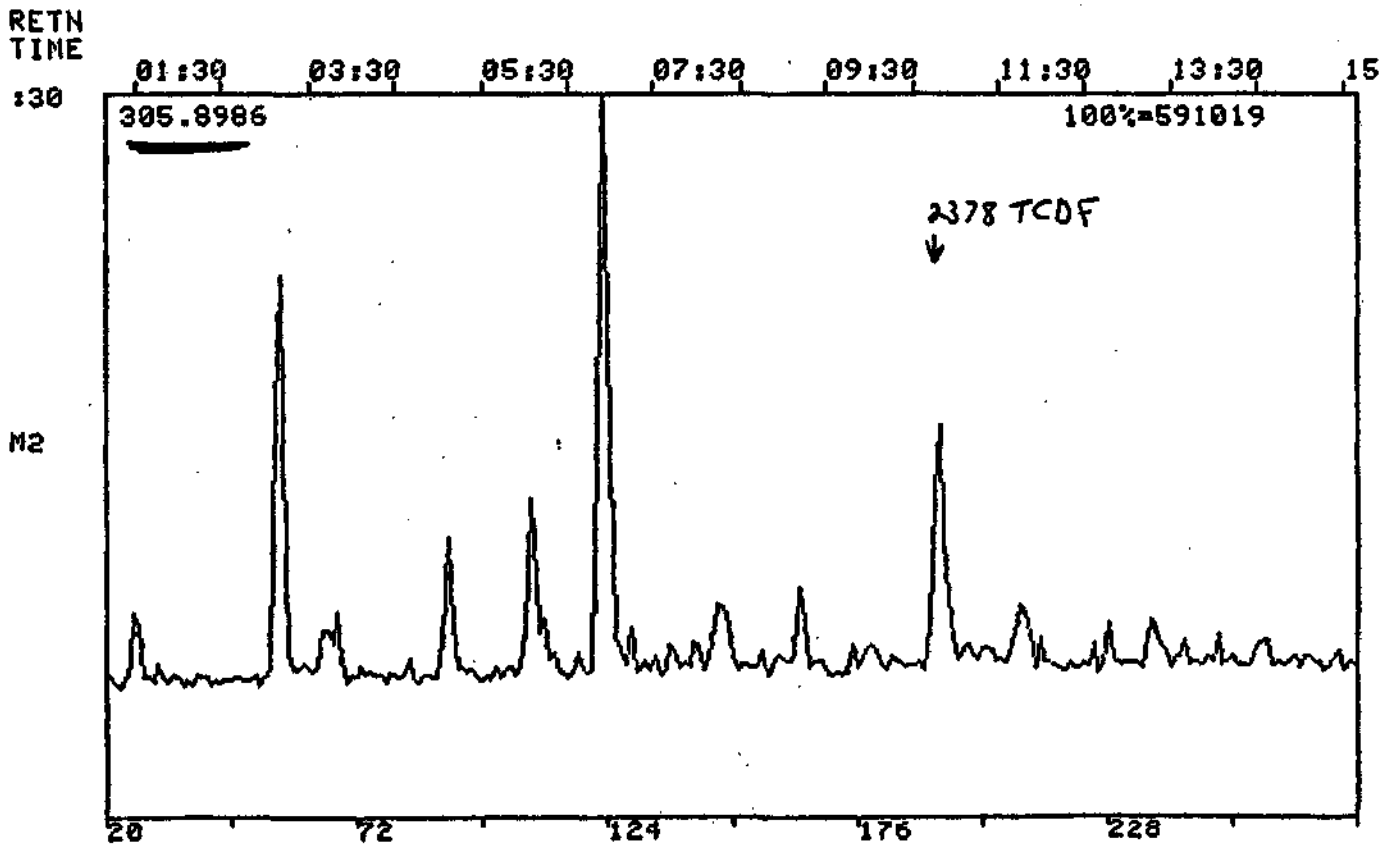


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DS55 HIGH RESOLUTION MPM
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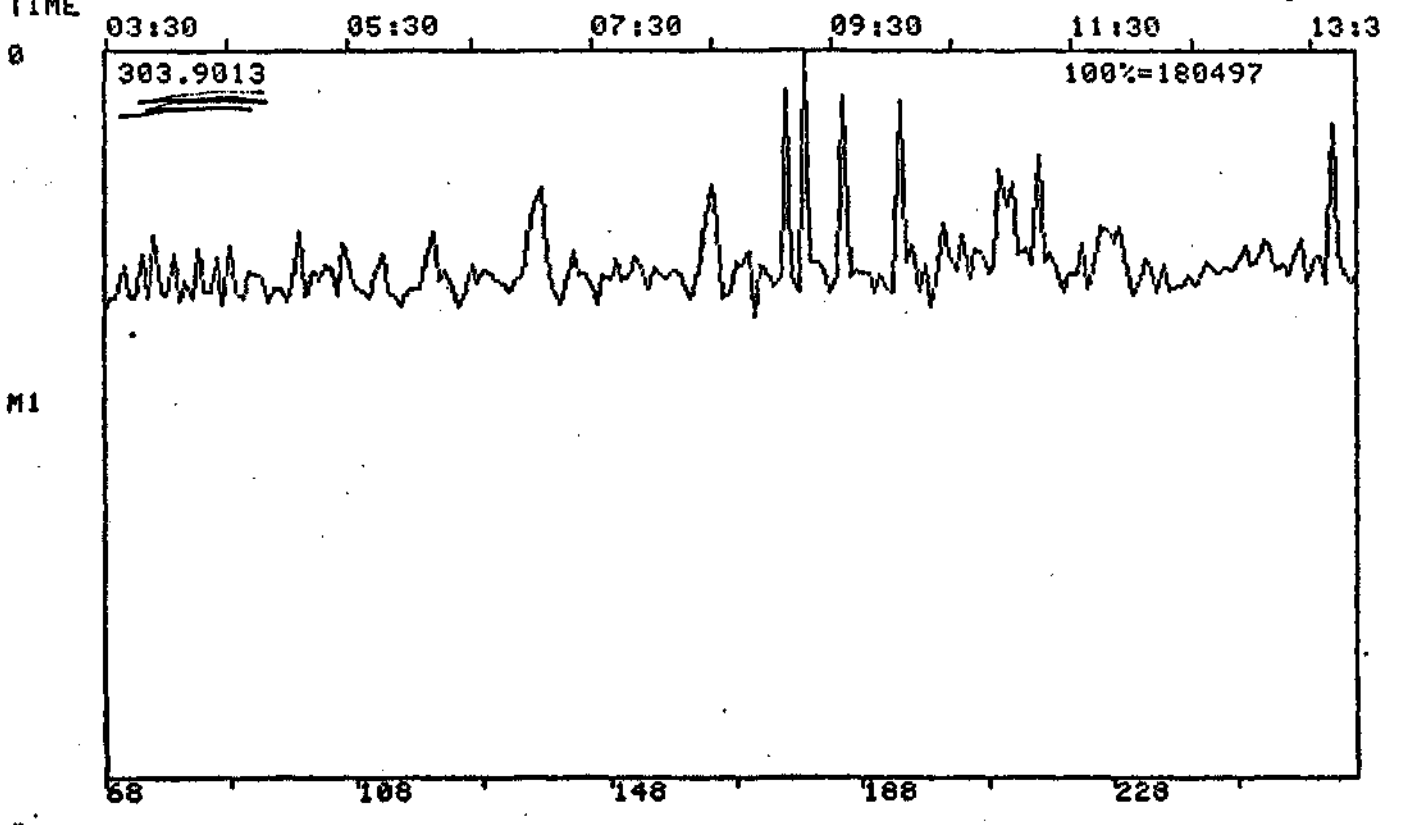
Figure 9: tetra CDFs in Floor 17
Sample 17C1P
Particulate only

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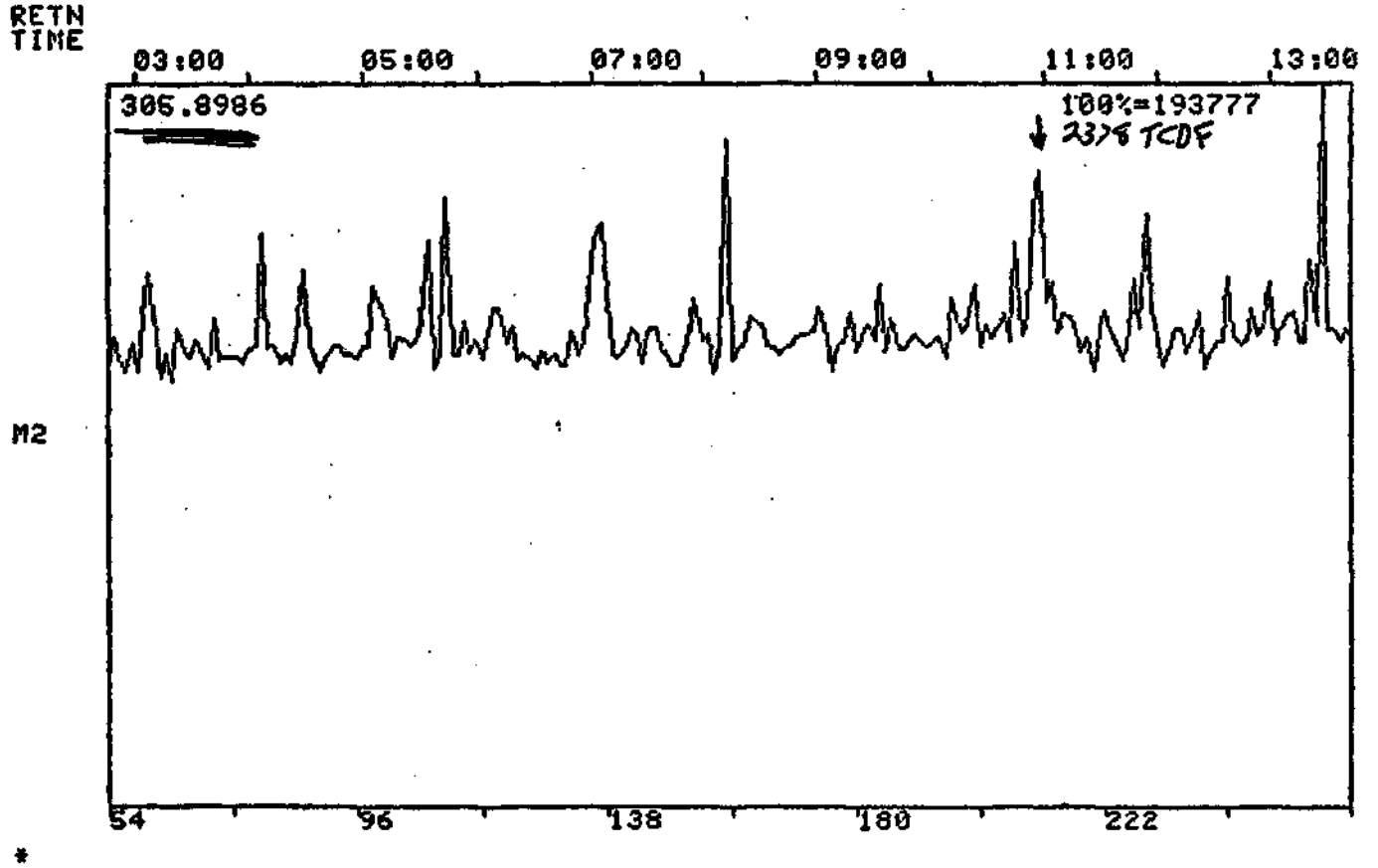


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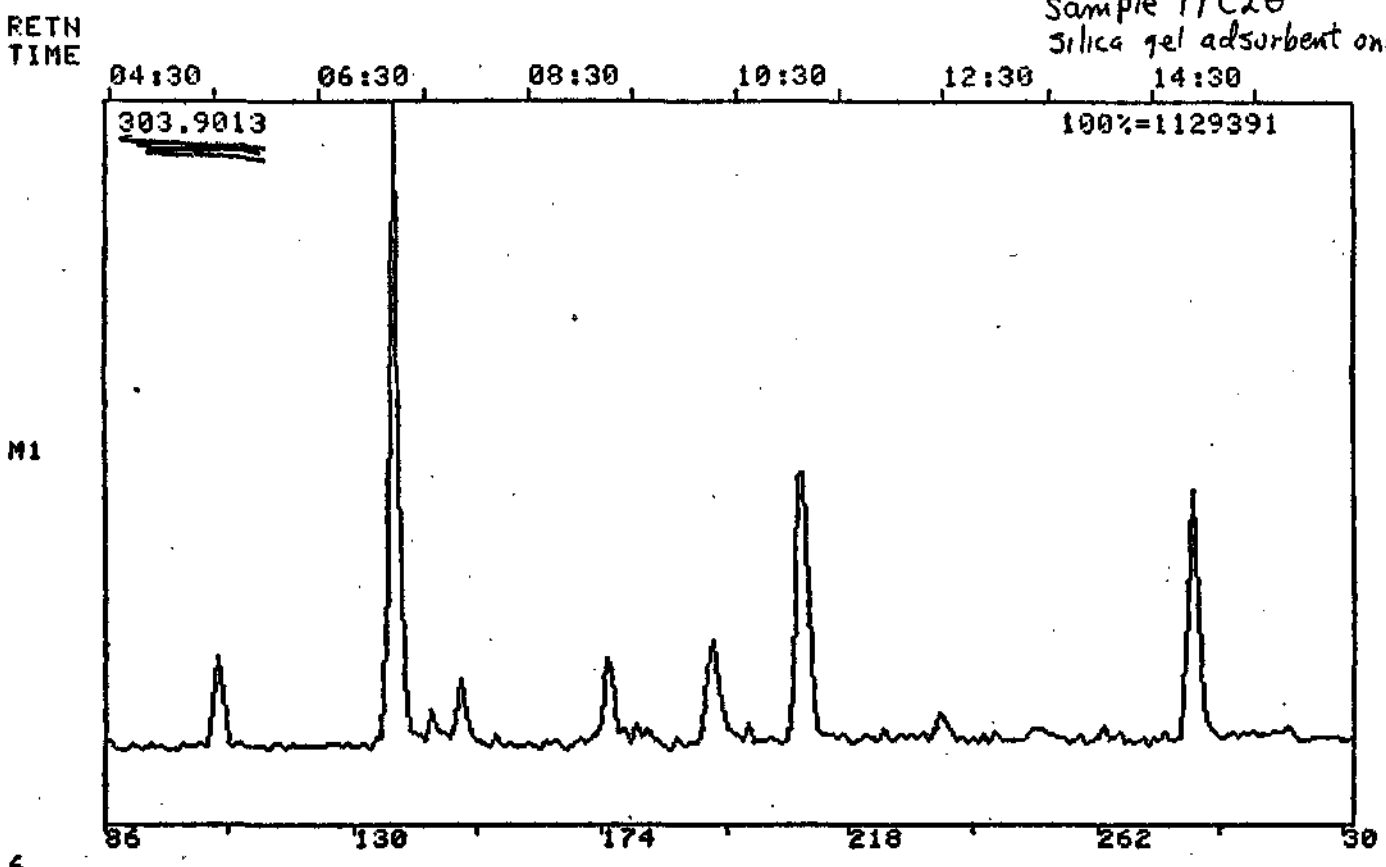
Figure 10: Tetra CDFs in Flour 17
Sample 17 C26
Silica gel adsorbent only

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RUNNAME TDHM10 DATE 3/29/83 TIME 16:57

17C1C

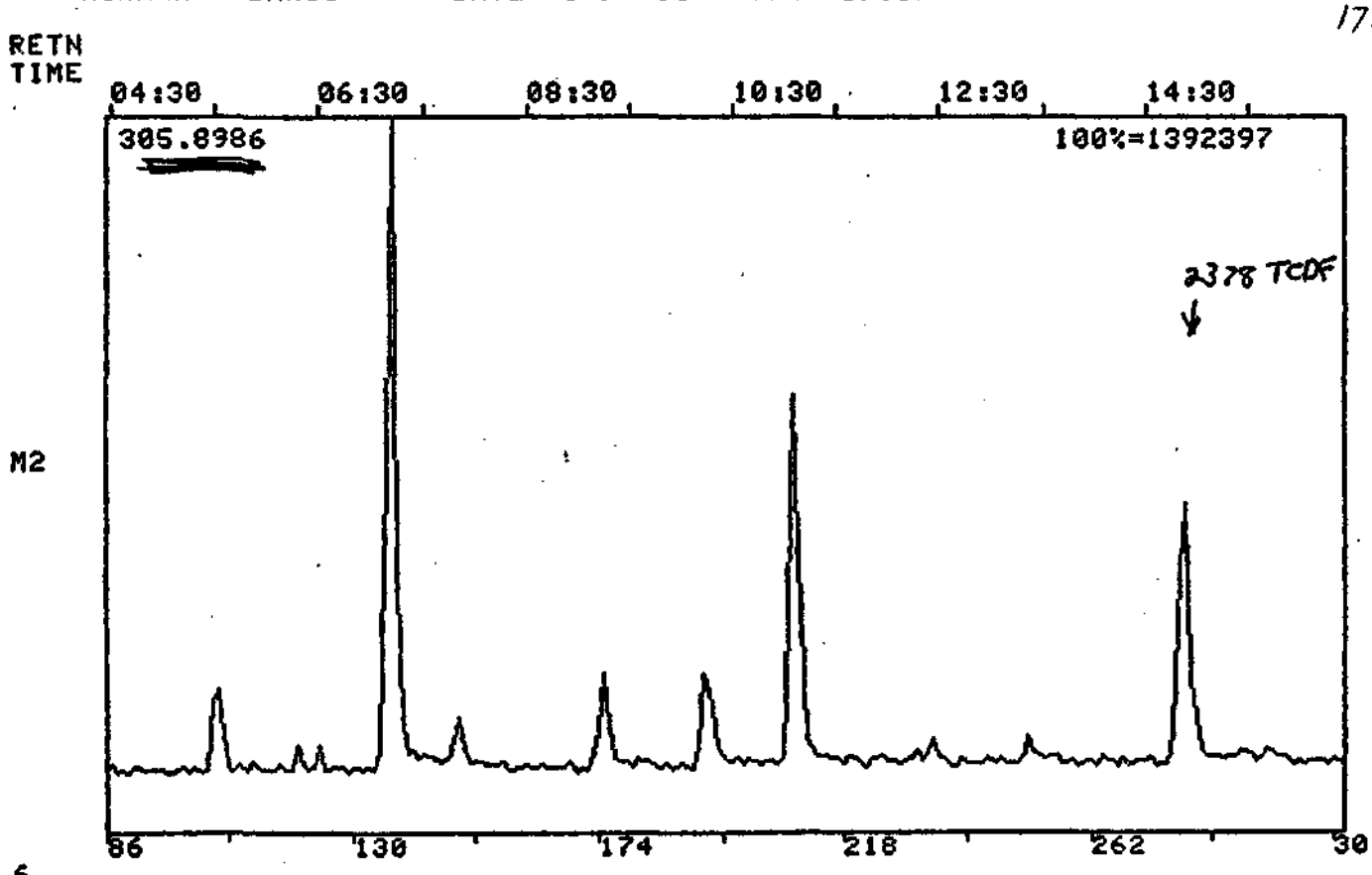
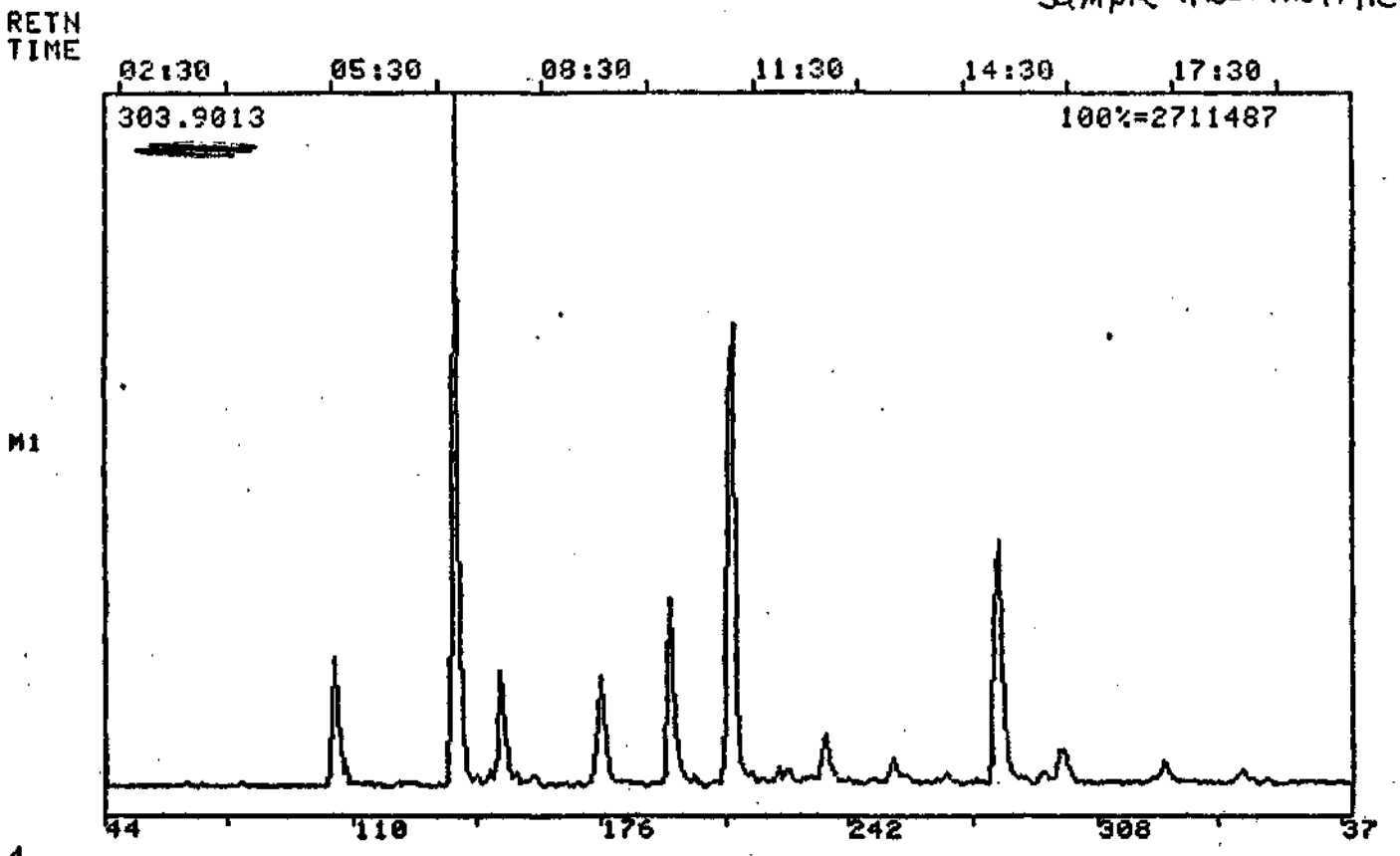
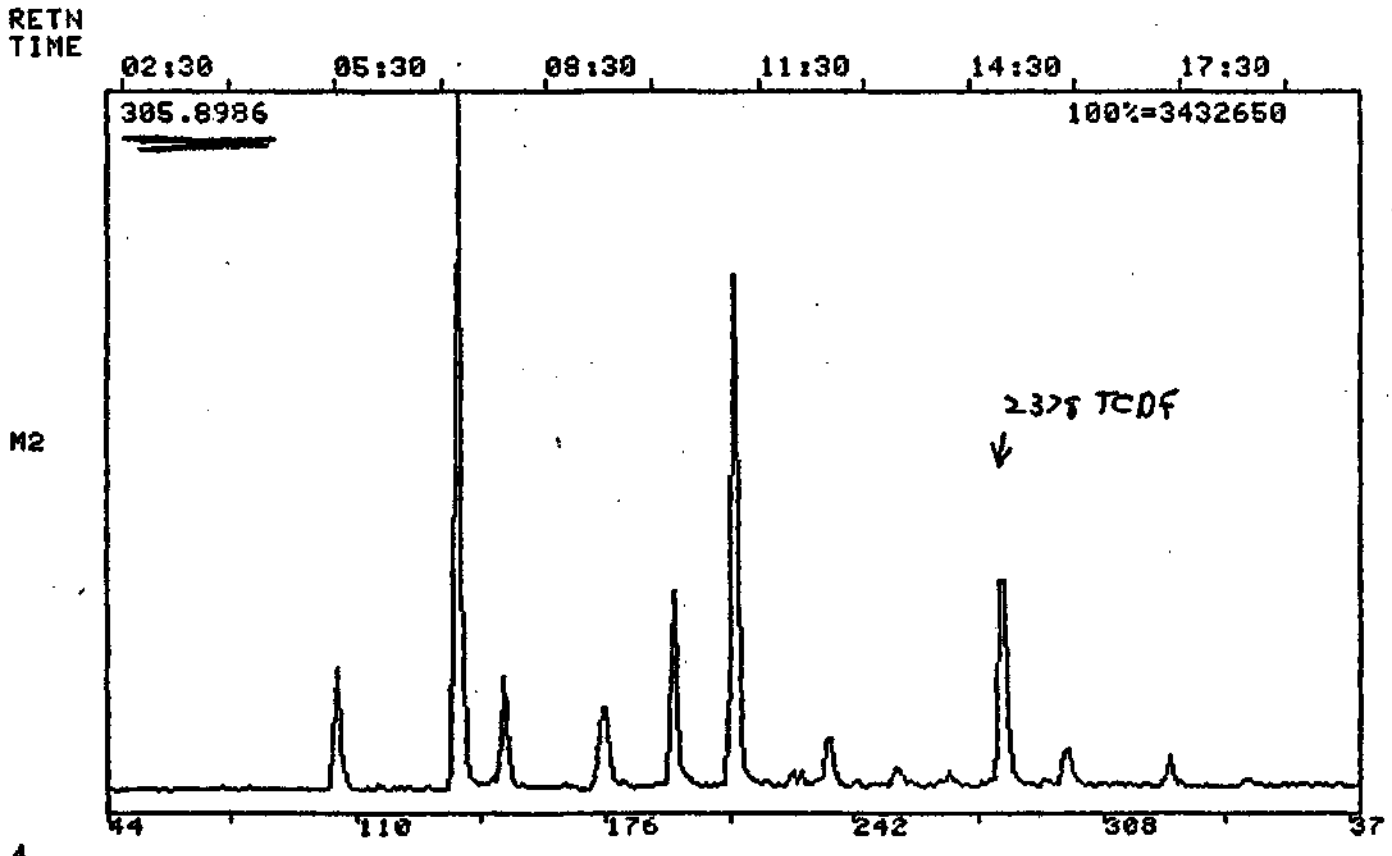


Figure 11: Tetra CDFs in Floor 11
Sample 11B3+11B4+11C1

DS5S HIGH RESOLUTION MPM
RUNNAME WDHM7 DATE 3/30/83 TIME

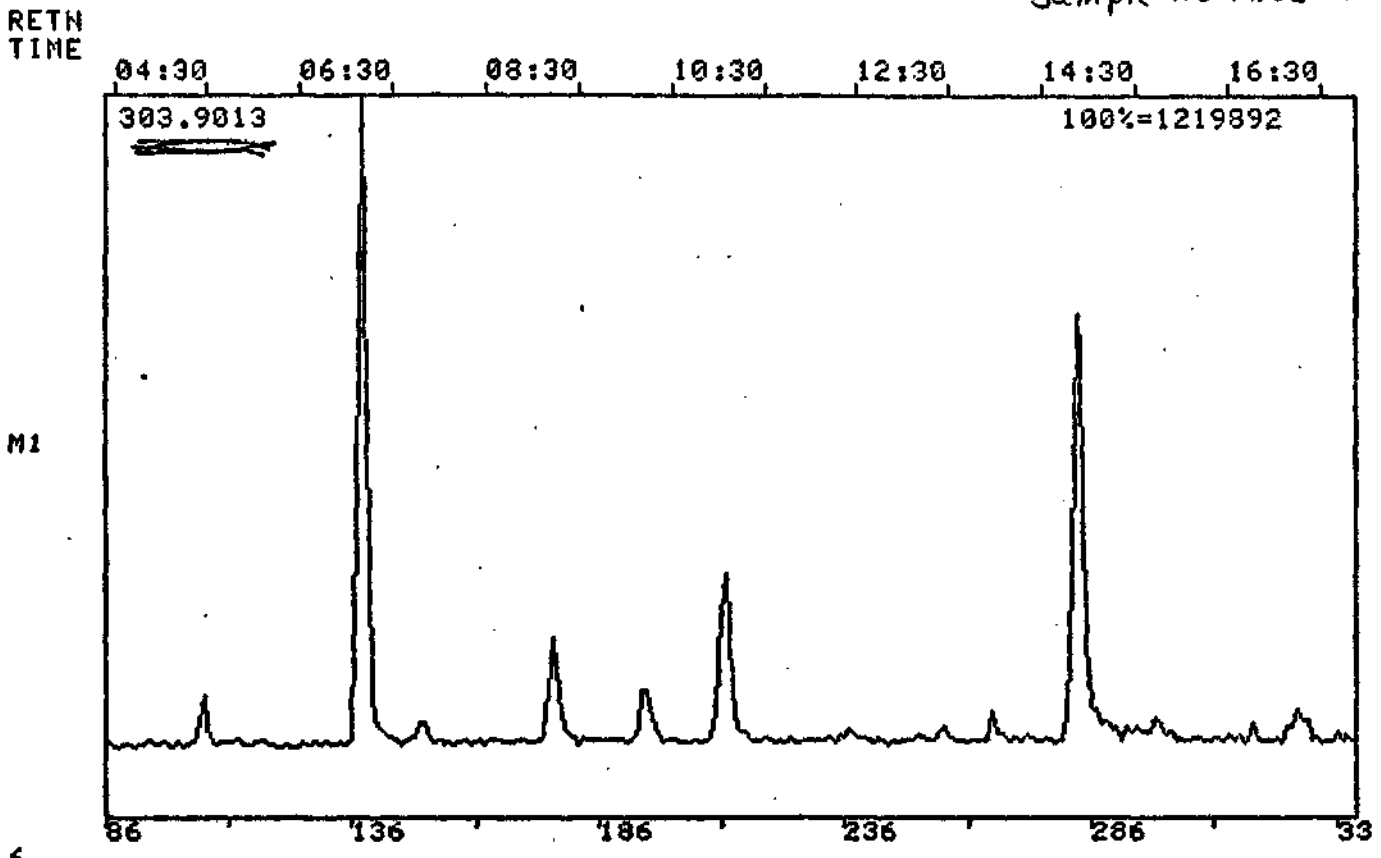



DS5S HIGH RESOLUTION MPM
RUNNAME WDHM7 DATE 3/30/83 TIME 12:57

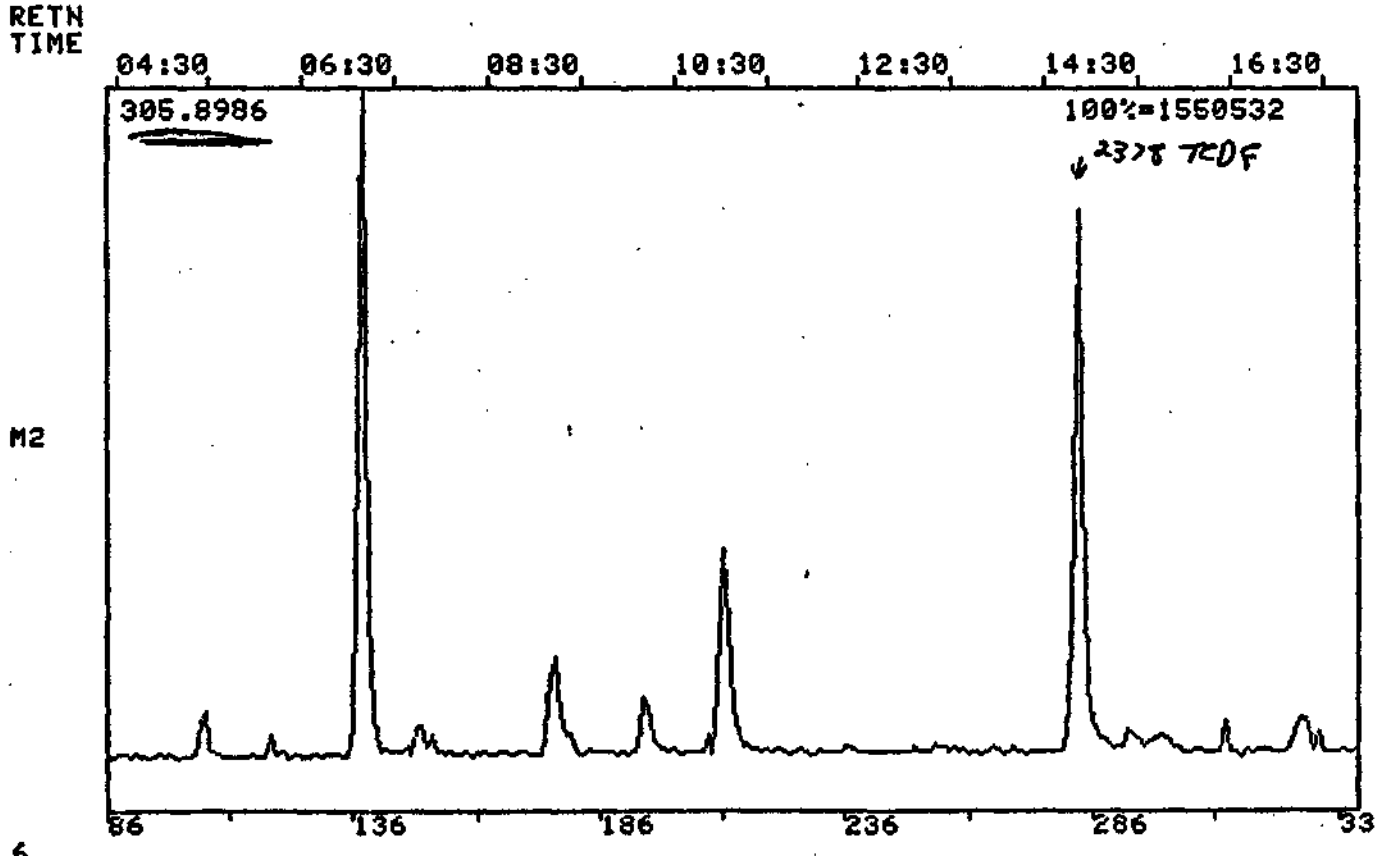


DS55 HIGH RESOLUTION MPM
RUNNAME TDHM5 DATE 3/31/83 TIME :

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

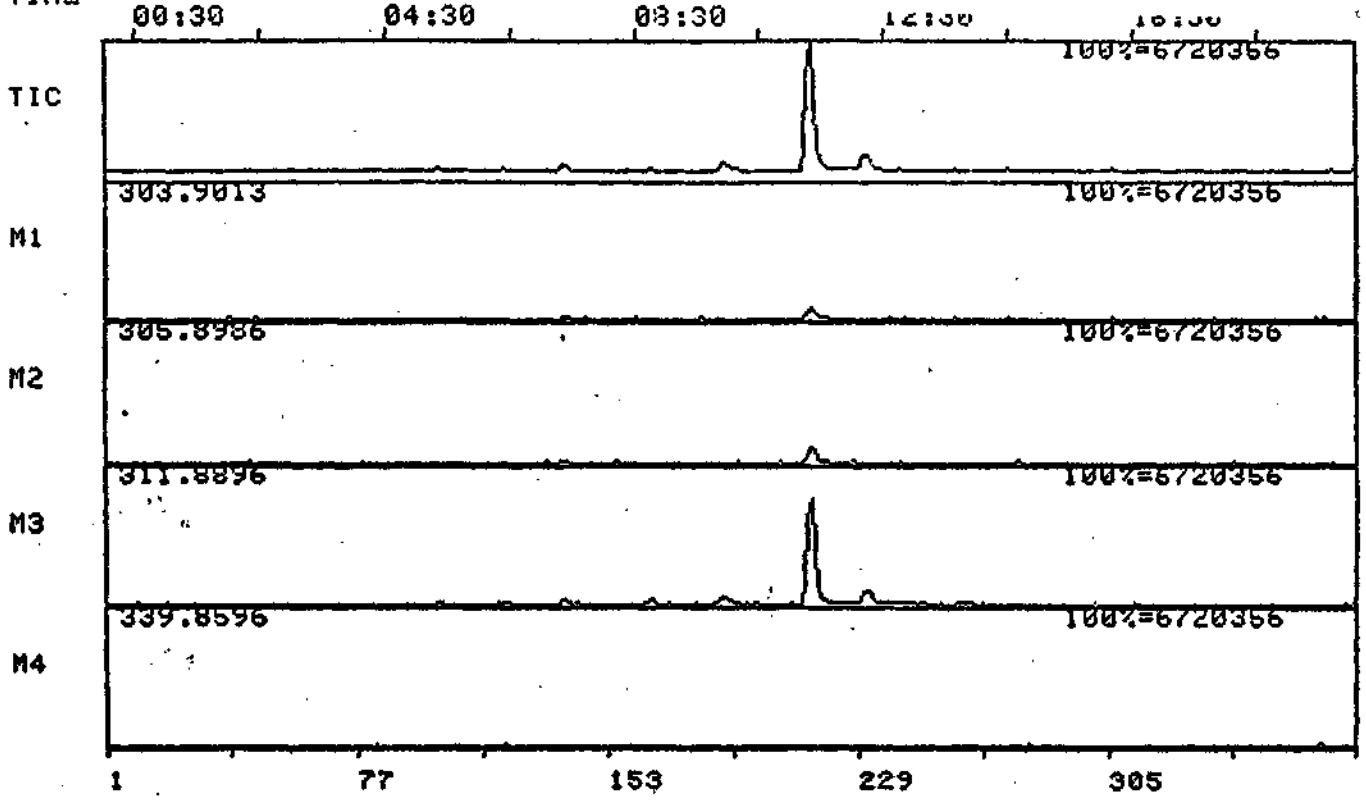



DS55 HIGH RESOLUTION MPM
RUNNAME TDHM5 DATE 3/31/83 TIME 11:31



RUNNAME MDHM2 DATE 3/28/83 TIME

Figure 13: QC Sample NSA65 Fortified with native TCDF




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

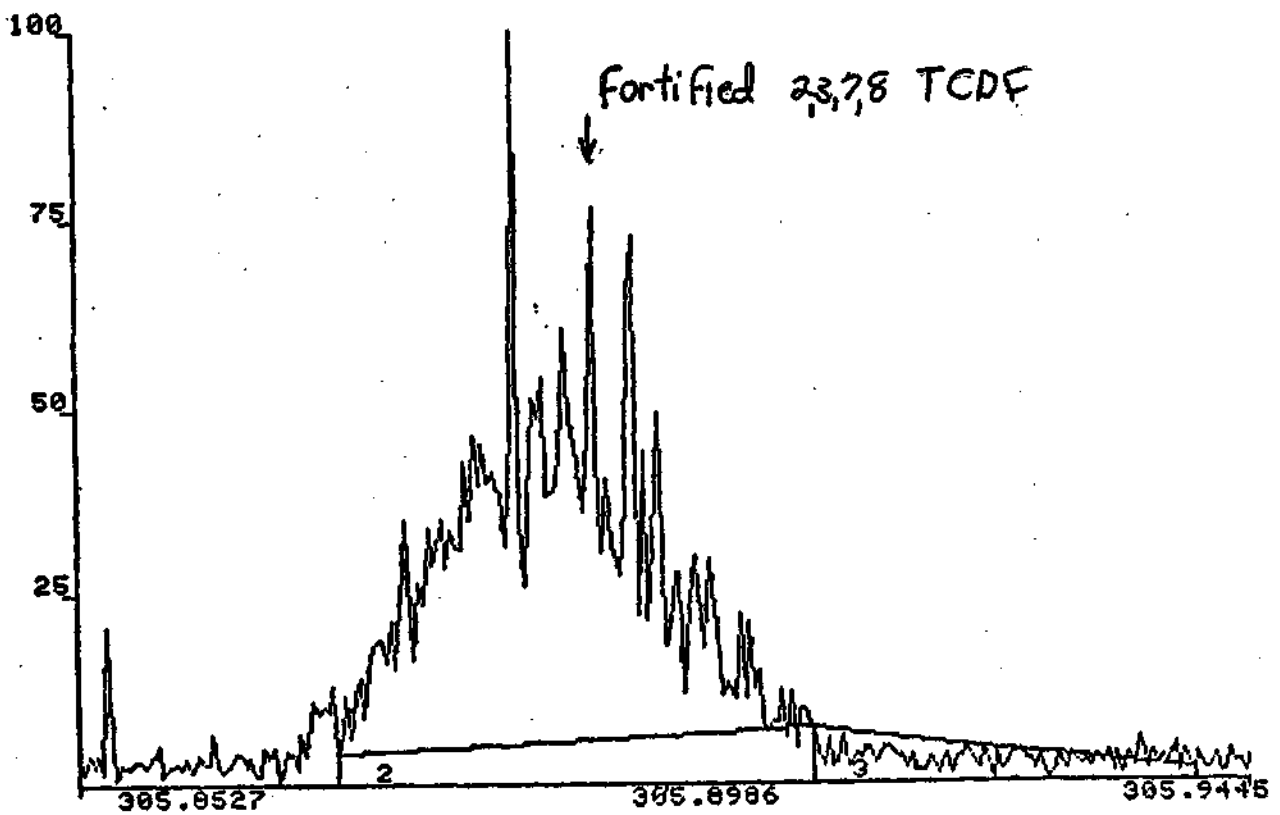
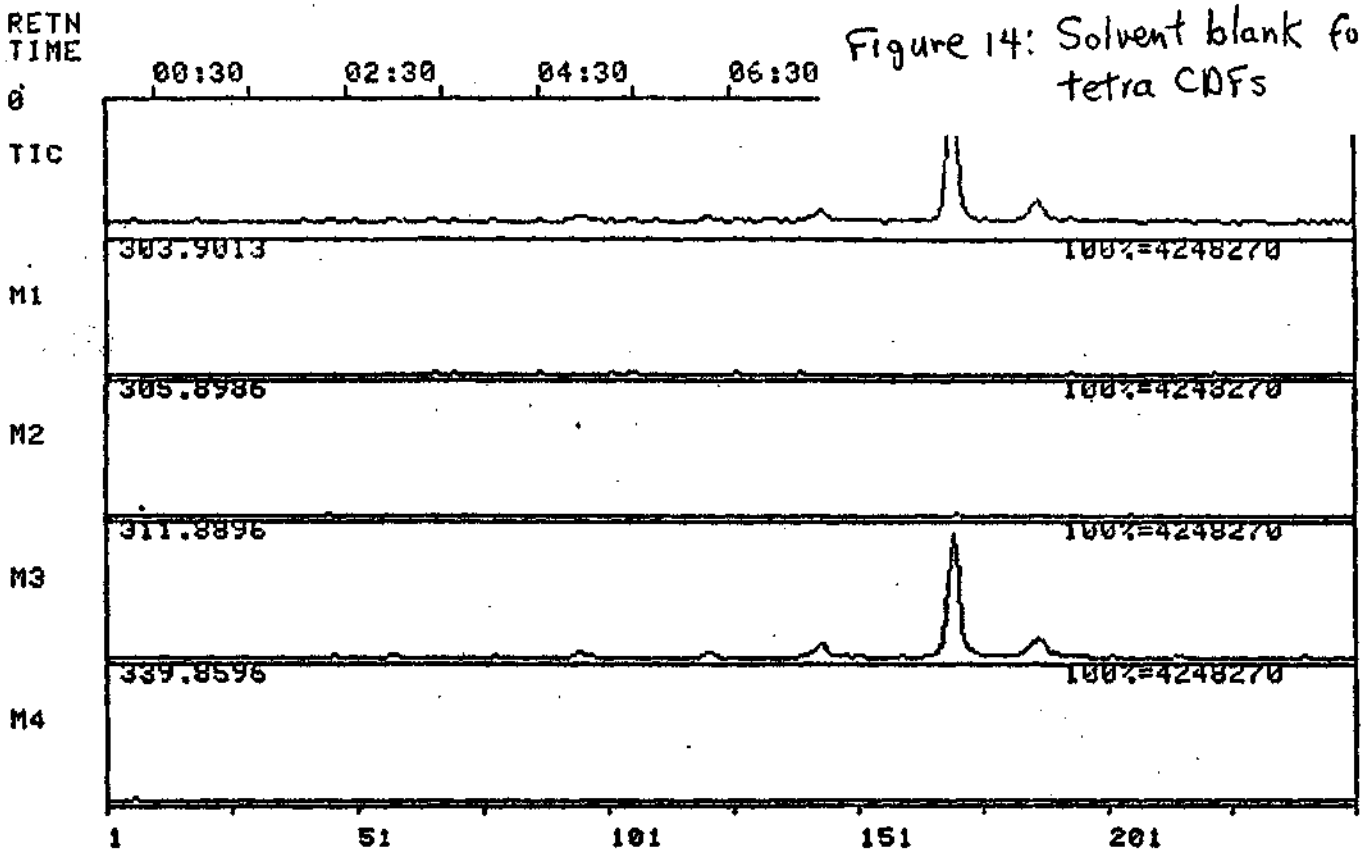


Figure 14: Solvent blank for tetra CDFs




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

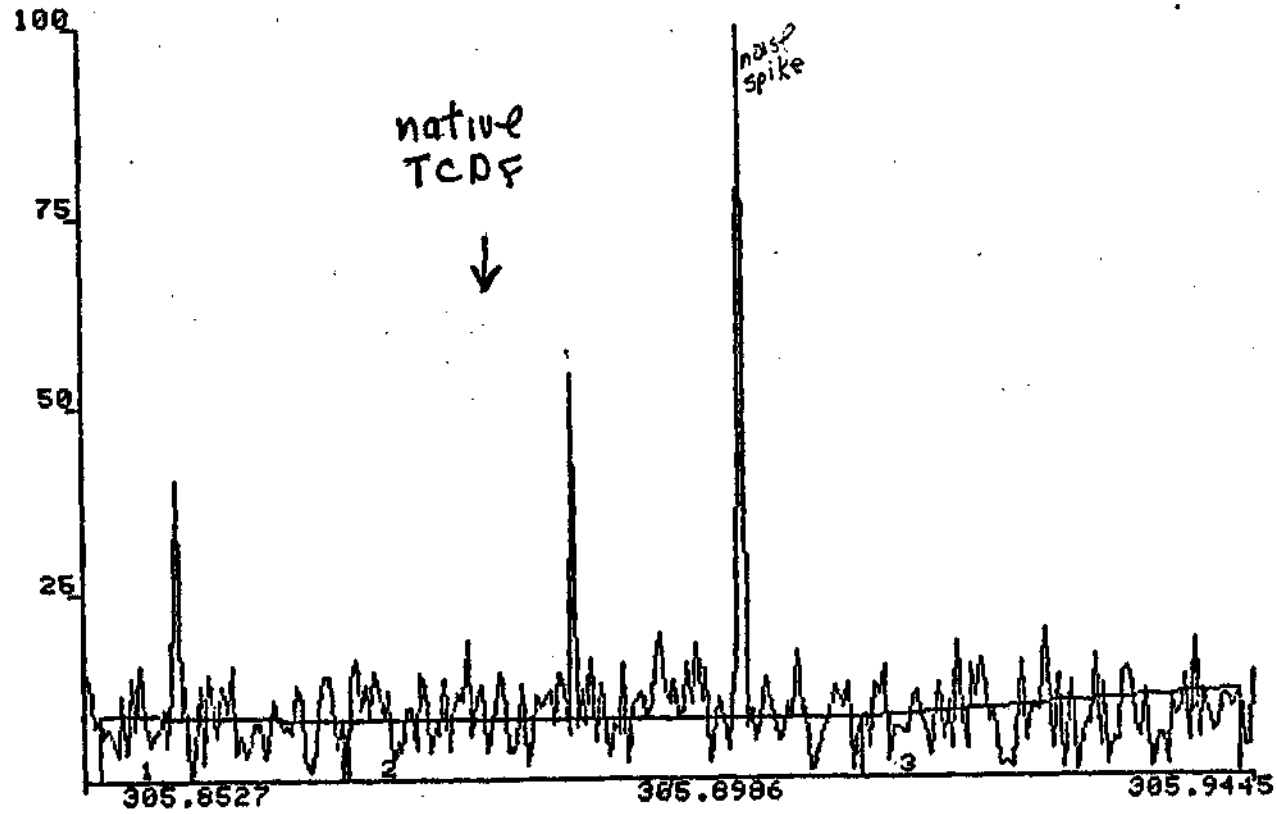
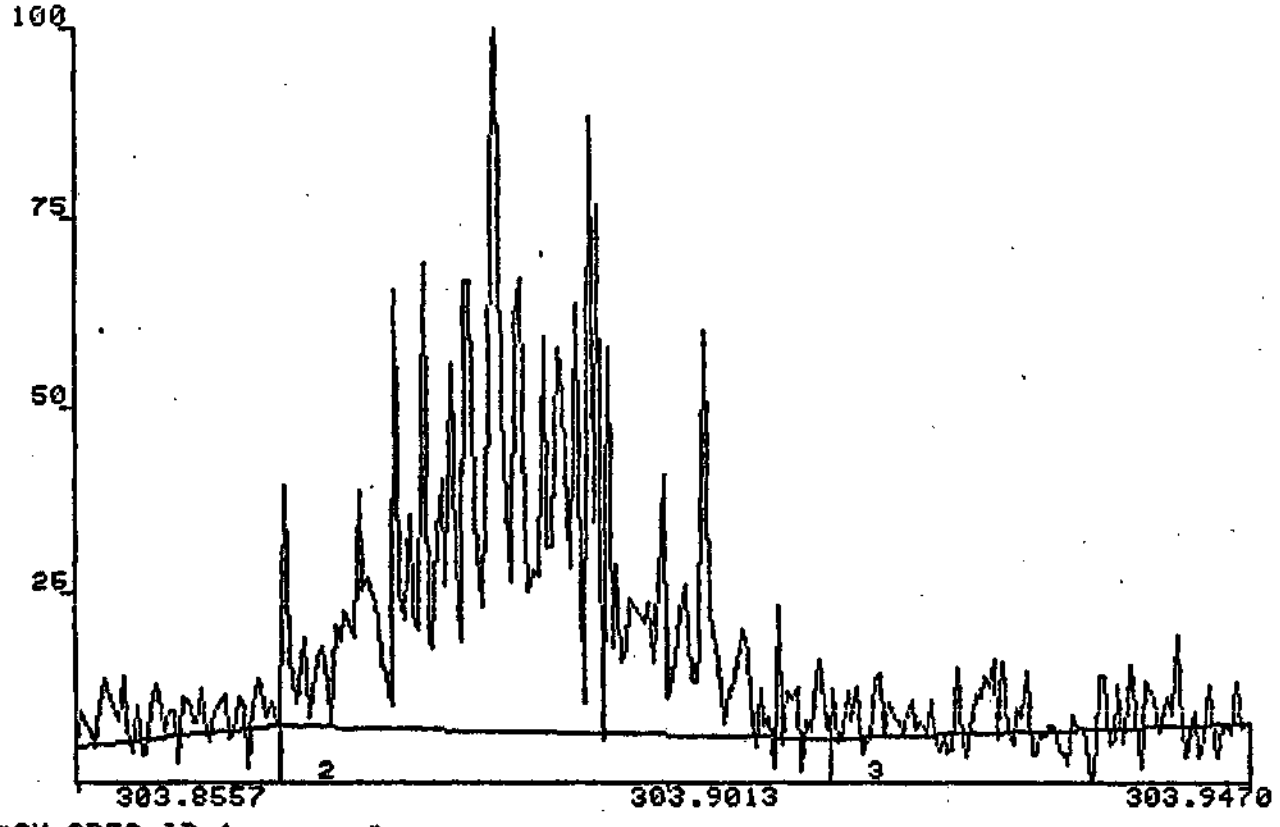
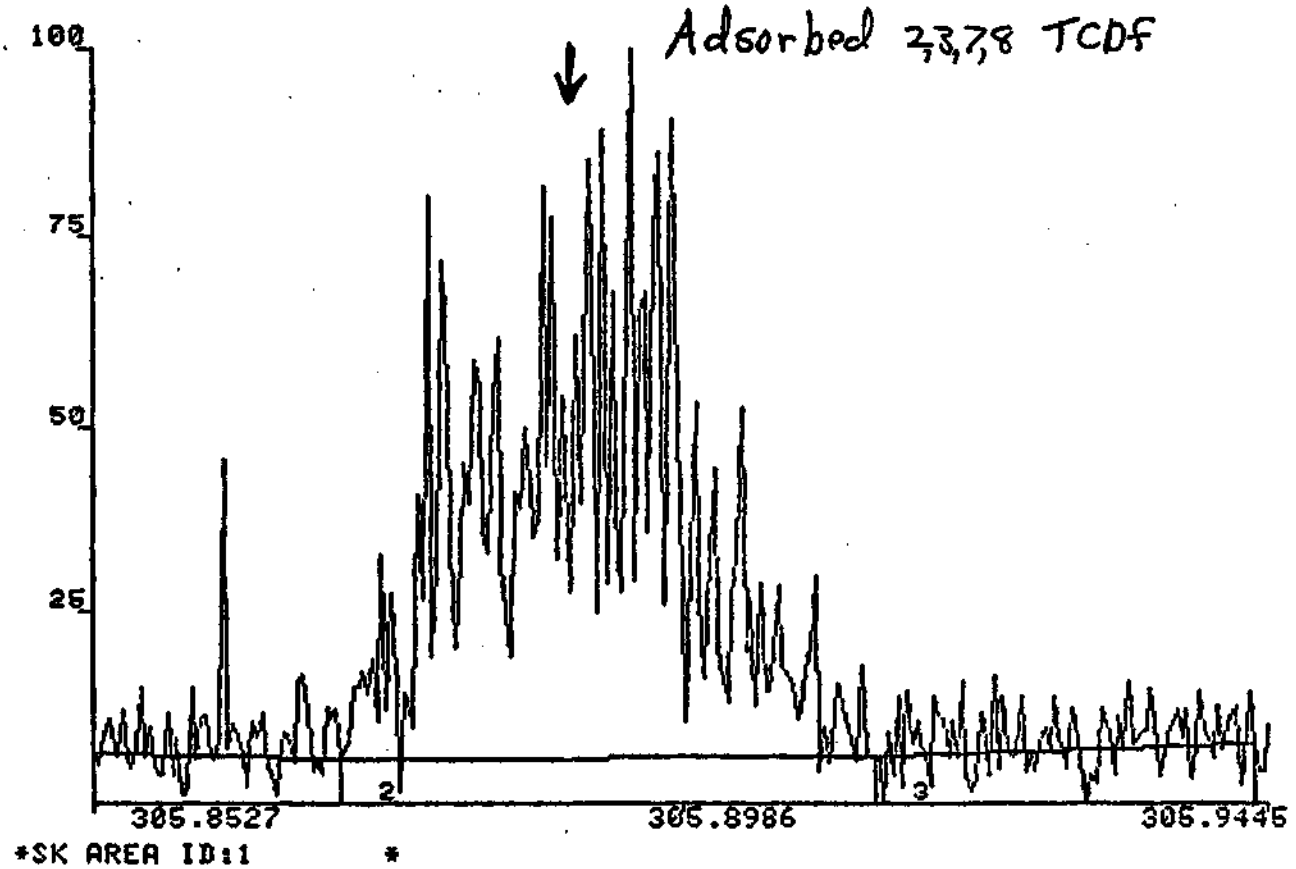


Figure 15: tetra CDFs in Sample 17C1G Silica Gel cartridge only

RUNNAME MDHM5 DATE 3/28/83 TIME
MASS 303.9013 SWEEP 300 (PPM)
SCANS 189-199 100% INTENSITY 24601



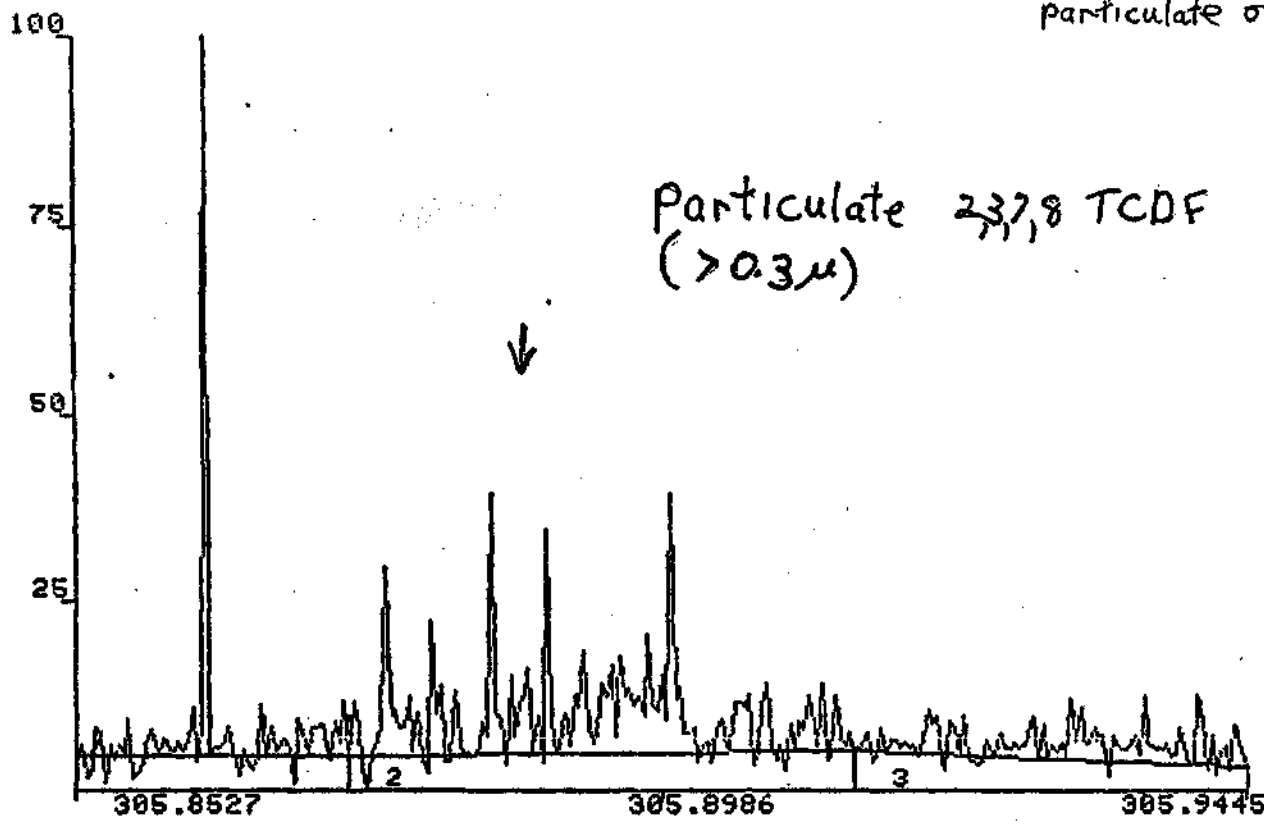

*SK AREA ID:1 *
RUNNAME MDHM5 DATE 3/28/83 TIME 18: 0
MASS 305.8986 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)
SCANS 189-199 100% INTENSITY 23146



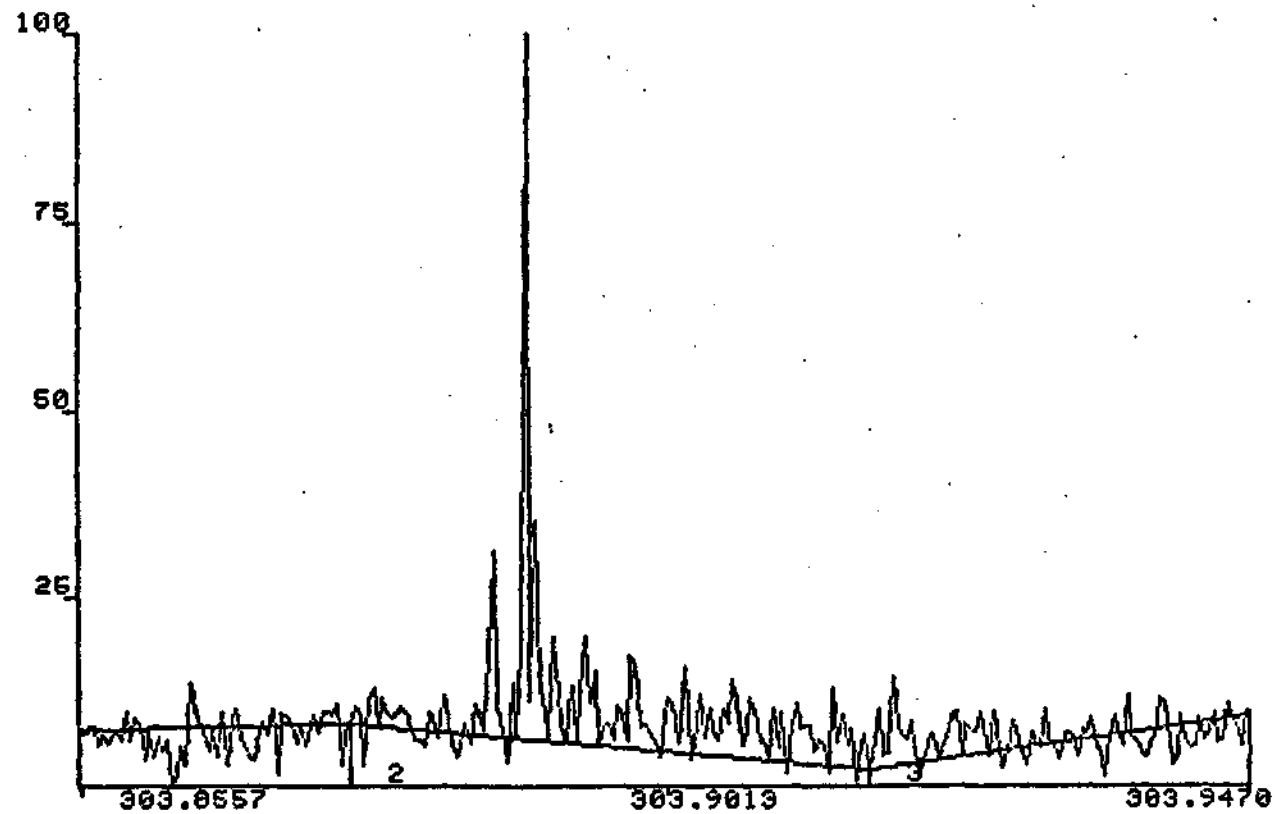

*SK AREA ID:1 *

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? C1 P 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 *

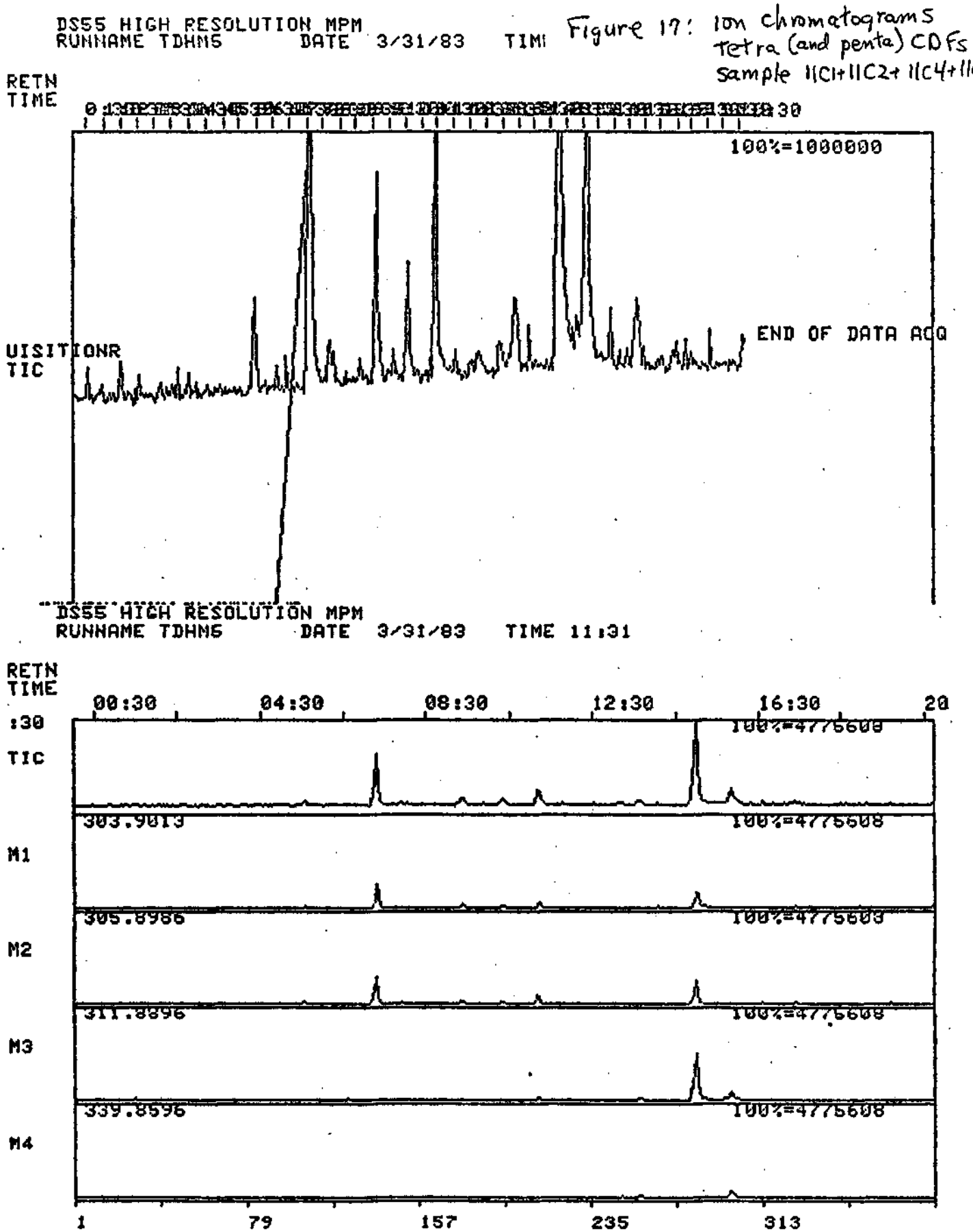



Figure 17: Ion chromatograms tetra (and penta) CDFs sample 11C1+11C2+11C4+11C5

*

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

DS55 HIGH RESOLUT

PEAK SUMMATION R

RUNNAME TDHM5 DATE 3/31/83 TIME 11:31

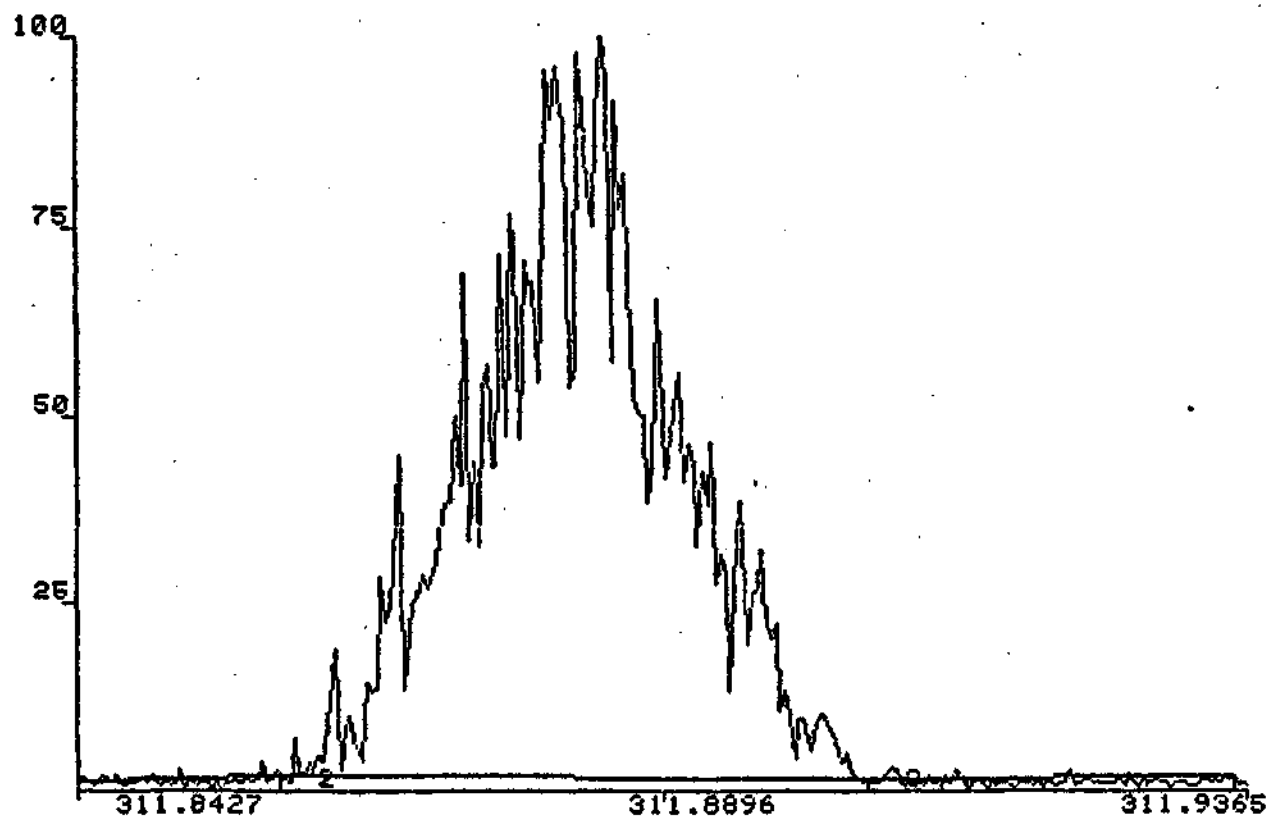
| | |
|---|---|
| MASS | 311.6897 |
| SCAN WIDTH | 300 PPM |
| SCAN TIME | 0.3 SECS |
| SCAN NUMBERS | 279- 287 |
| STANDARD | 0.0000 |
| FACTOR | 0 |

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

| MASS CENTROID | ITEM | AREA | BASELINE SUBTRACTED | BASELINE SKIMMED | %TOTAL AREA | RELATIVE TO STANDARD |
|---|---|---|---|---|---|---|
| 311.8836 | TOTAL | 5945755. | YES | NO | 88.00 | 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 TDHM5 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 Figure 19: areas
PEAK SUMMATION RI Sample 11C1+11C2+11C4+11C5

RUNNAME TDHM5 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 |
| 303.8629 | 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 TDHM5 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

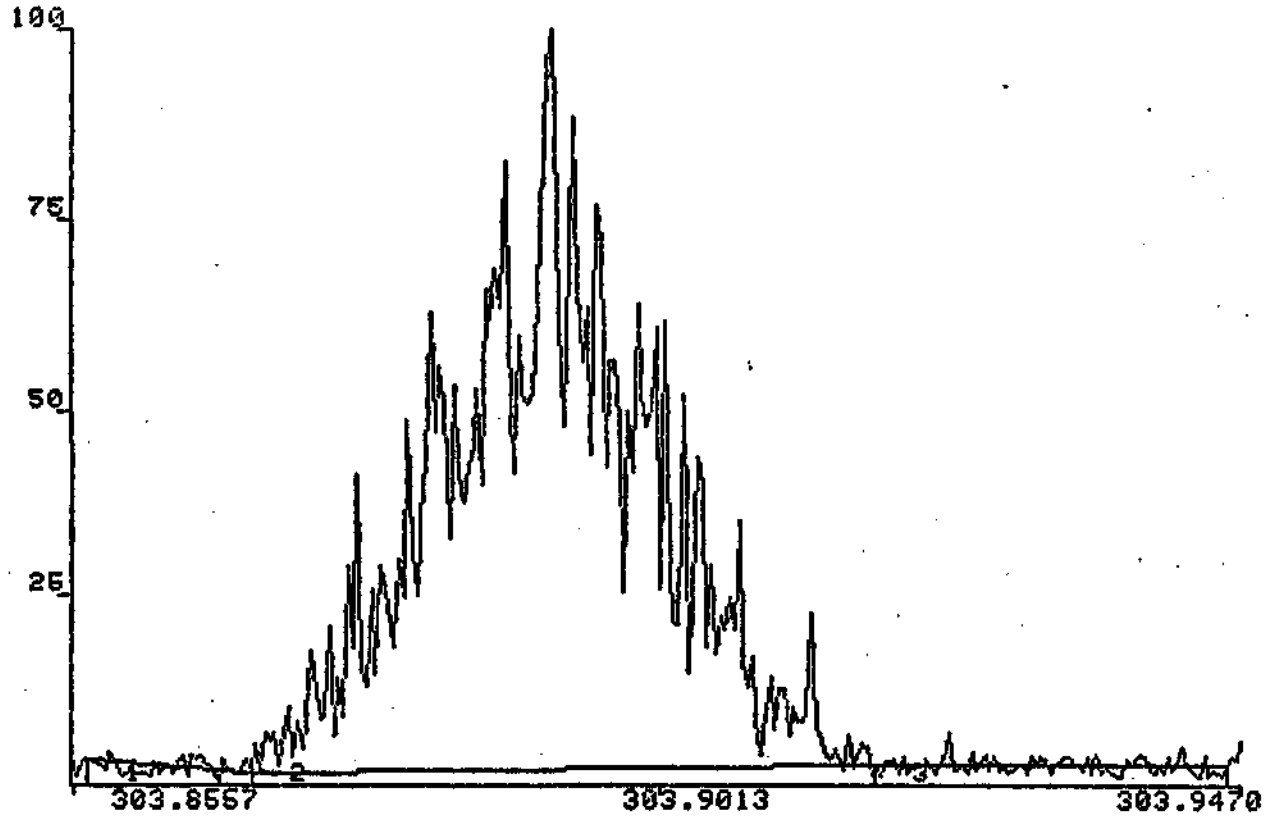
1.95 OF 7.6UL SAMPLE #C11(1+2+4+5)

| 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 |

*

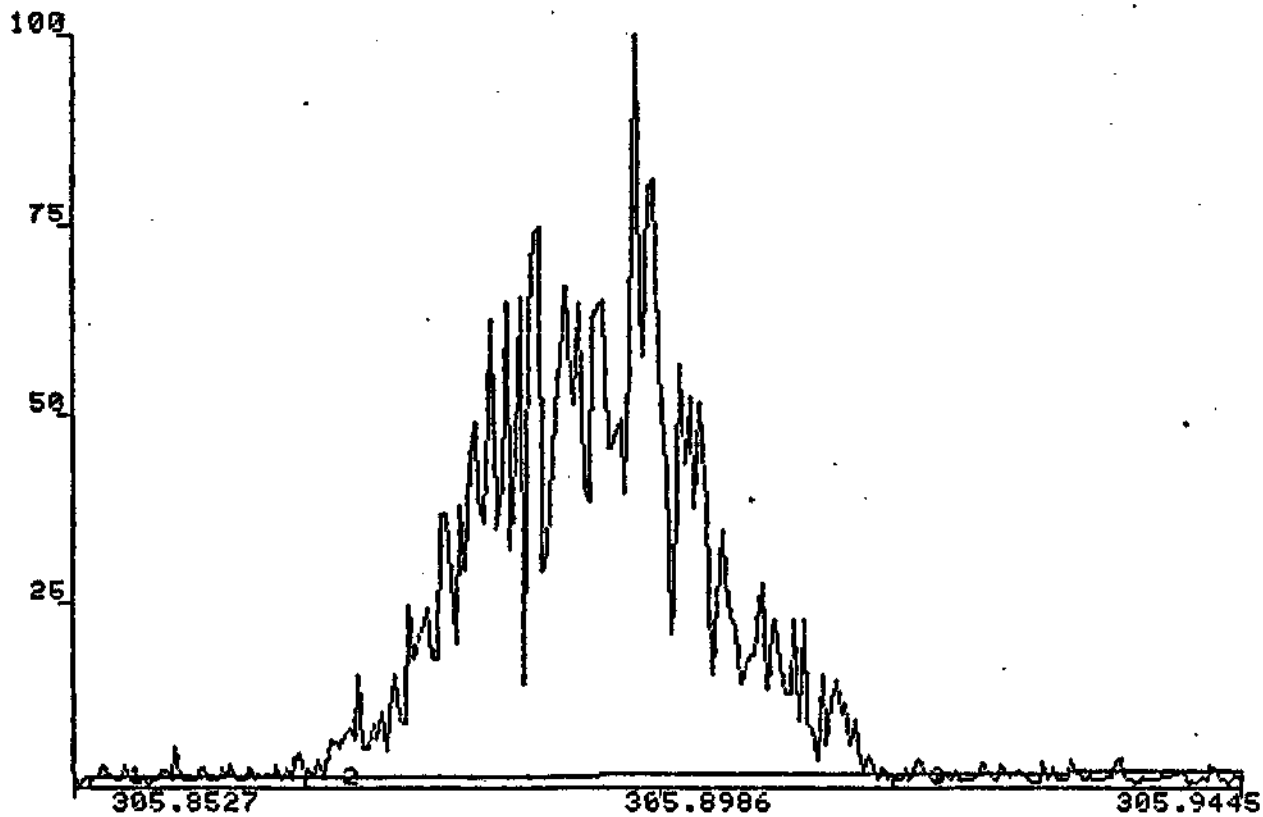
Figure 20: mass profiles
Sample 11C1+11C2+11C4+11C5

RUNNAME TDHM5 DATE 3/31/83 TIM
MASS 303.9013 SWEEP 300 (PPM)
SCANS 279-287 100% INTENSITY 54145



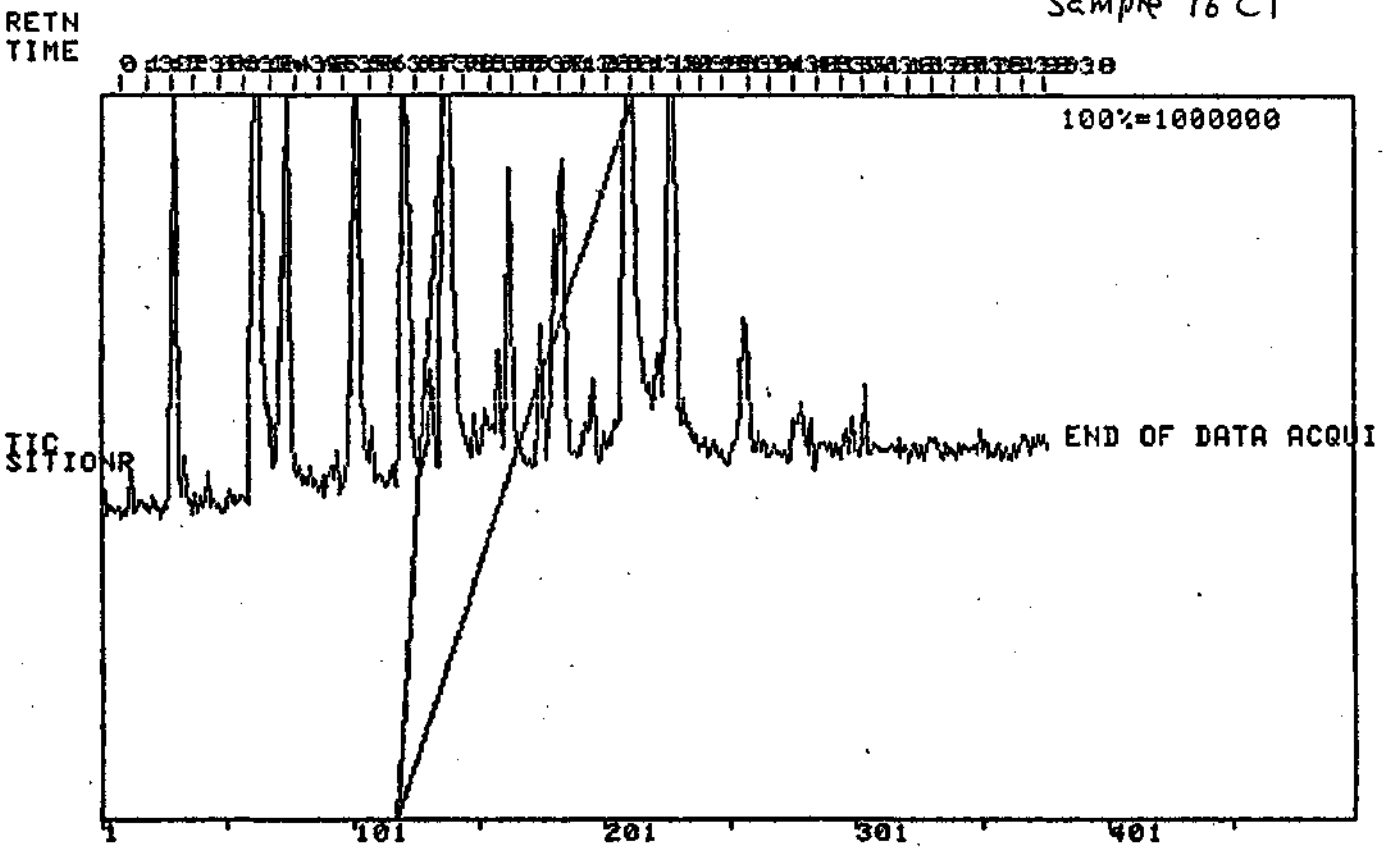

*

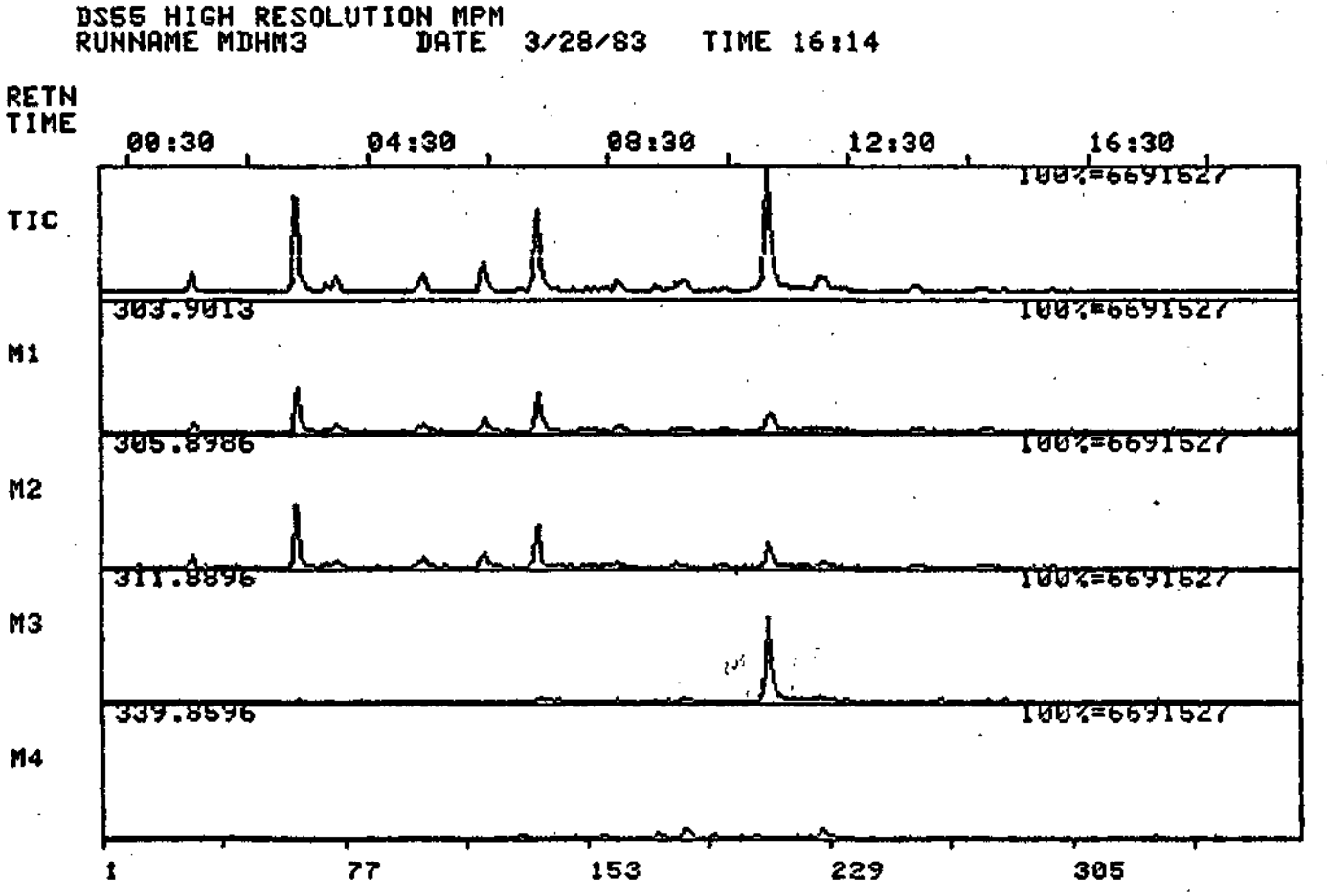
RUNNAME TDHM5 DATE 3/31/83 TIME 11:31
MASS 305.8986 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)
SCANS 279-287 100% INTENSITY 76709




*

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




*

Figure 22: Mass profile + areas
Sample 16 C1

PEAK SUMMATION R

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

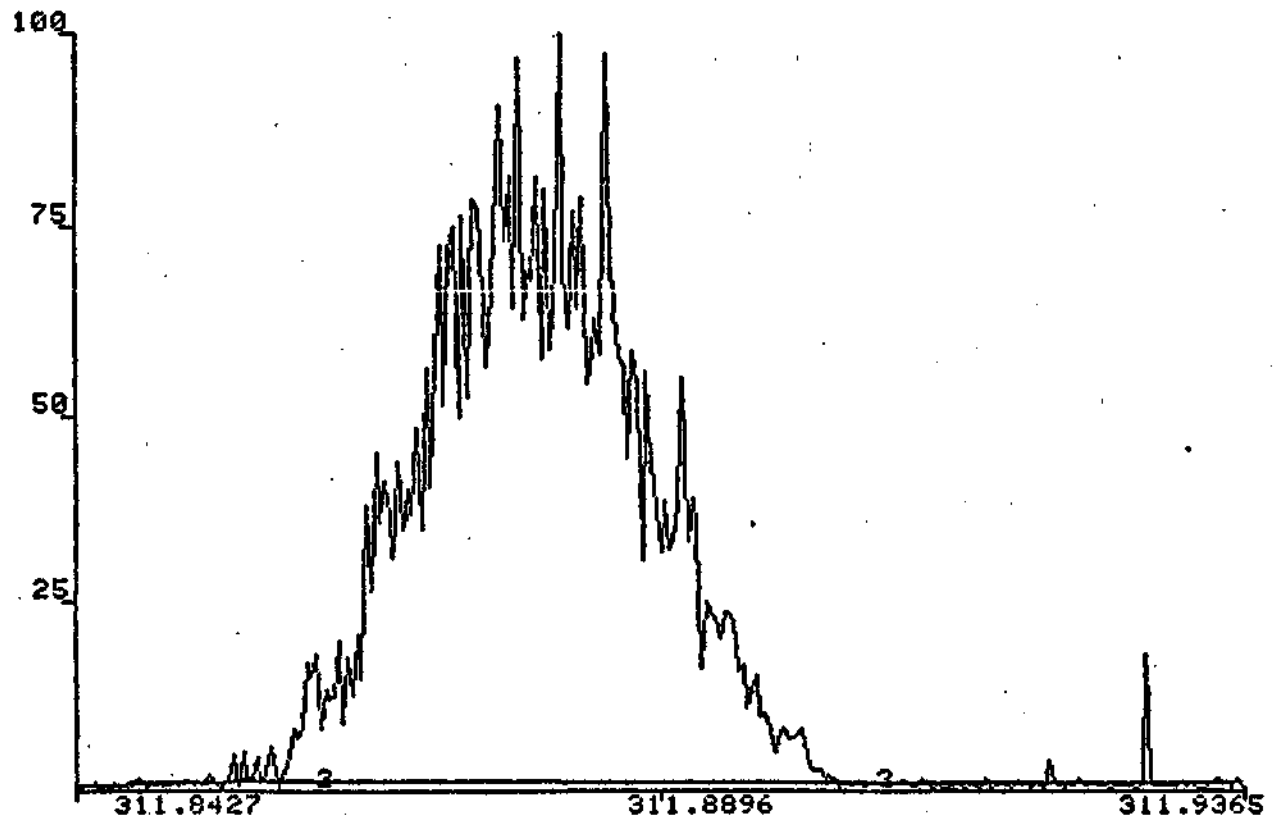
| | |
|---|---|
| MASS | 311.8897 |
| SCAN WIDTH | 300 PPM |
| SCAN TIME | 0.3 SECS |
| SCAN NUMBERS | 207- 215 |
| STANDARD | 0.0000 |
| FACTOR | 0 |

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

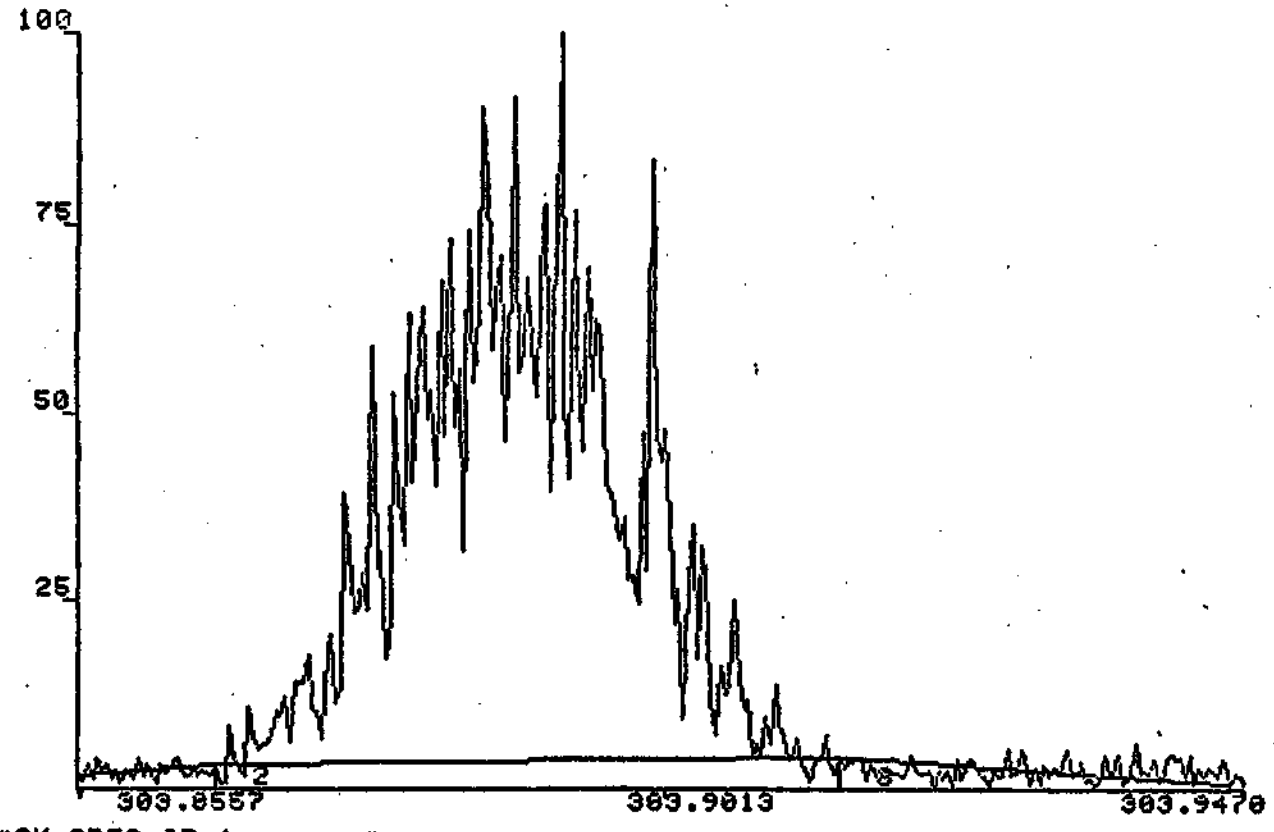
16C1 2UL OF 8UL TOTAL

| 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 |

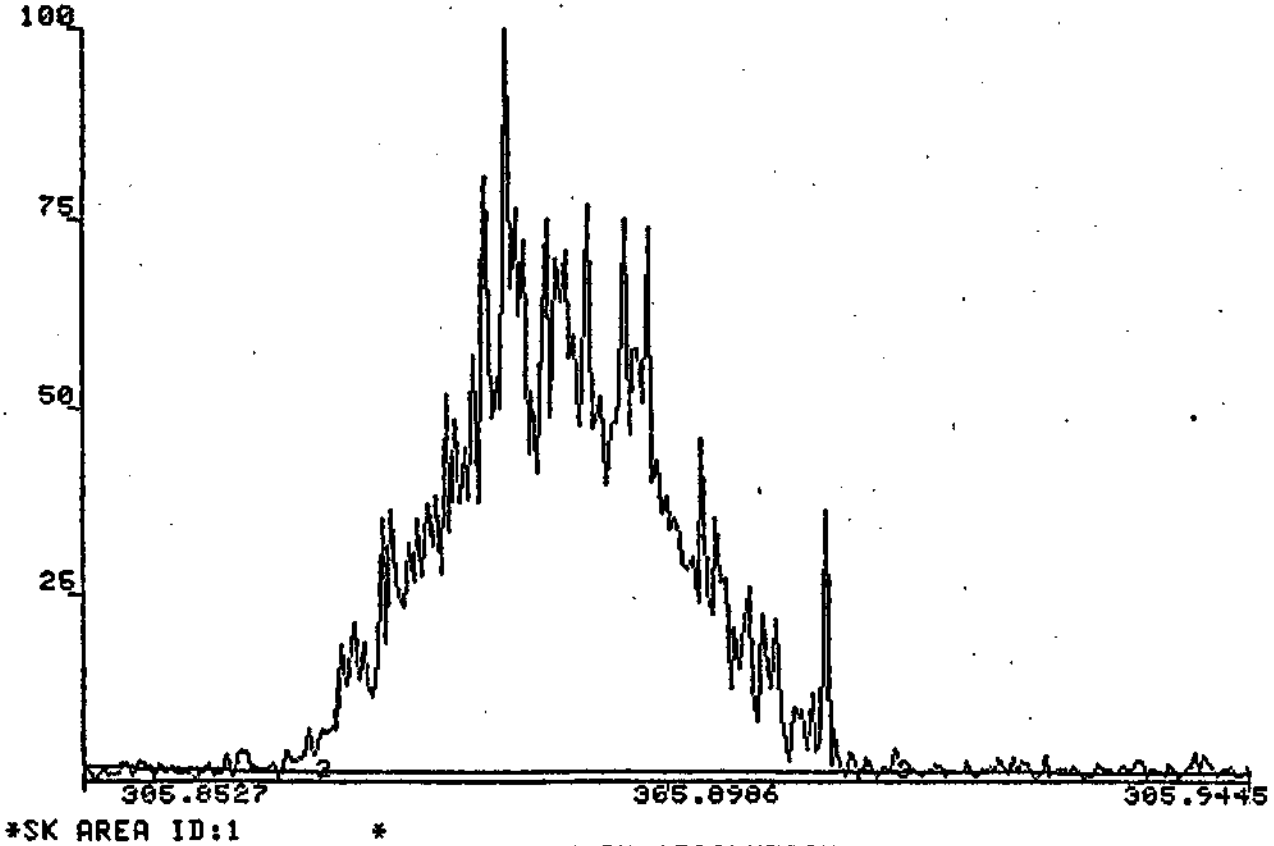
*

Figure 24: mass profiles sample 16 Cl

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




*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 *

DS55 HIGH RESOLUTION NPM

Figure 25: Calculations
Sample 16 C1

MDHM3 - 2.0 ul of 8.0 ul 16 C1
Acqui. @ 20 min.

Amt of $^{37}$Cl-TCDF = 20 x 60 pg/ul = 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%

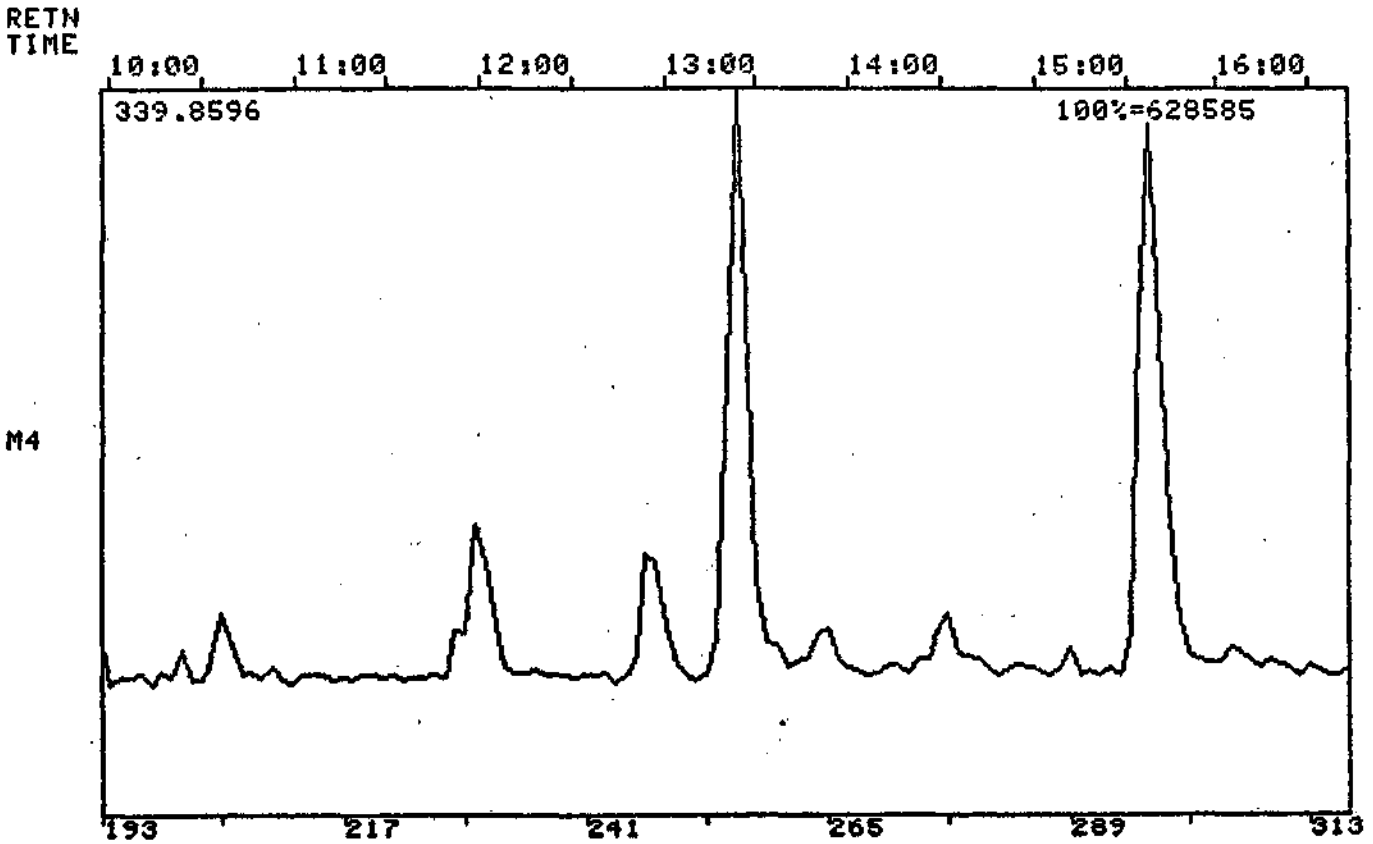
Recovery = (9403356/35793 counts/pg / (1200 x 1/4)) x 100 = 88%

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

RRT = 1.0

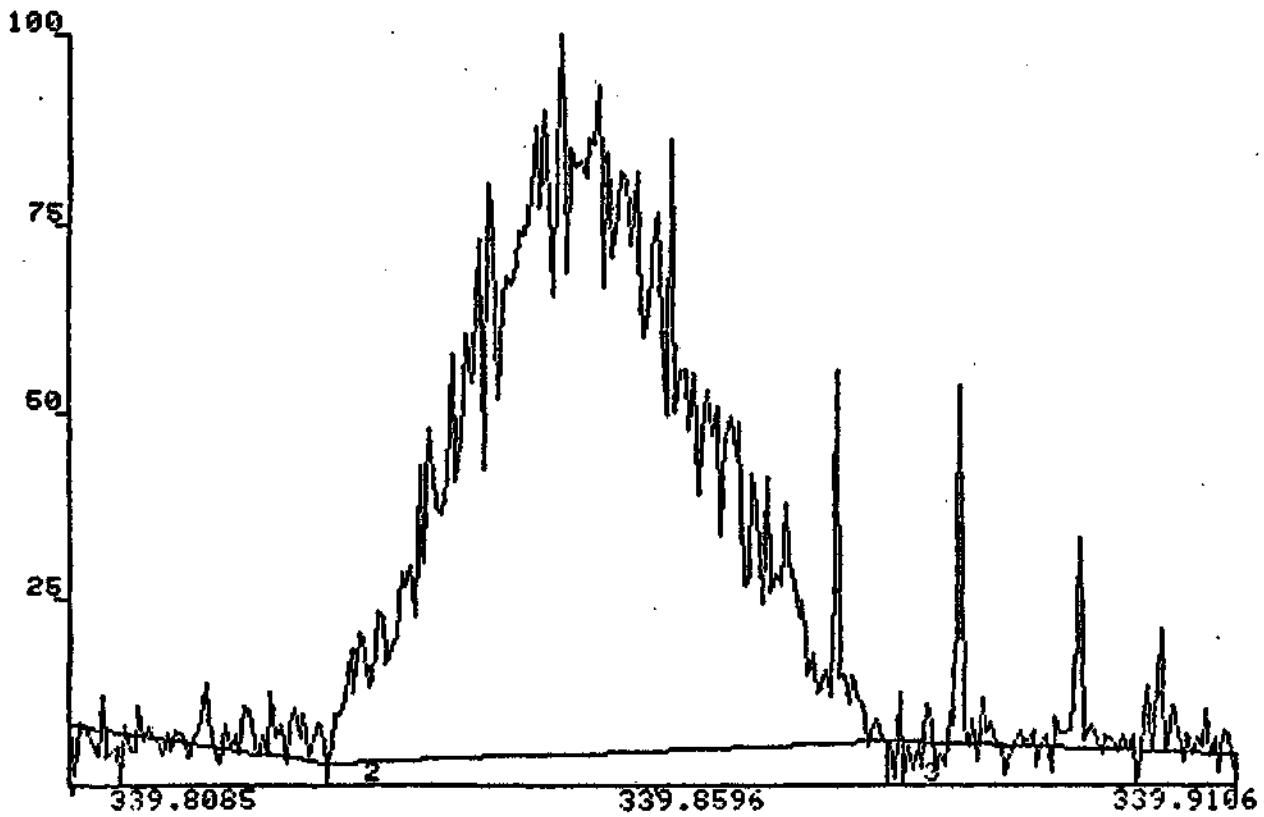
Figure 26: penta CDFs
Sample 11B3+11B4+11C1

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



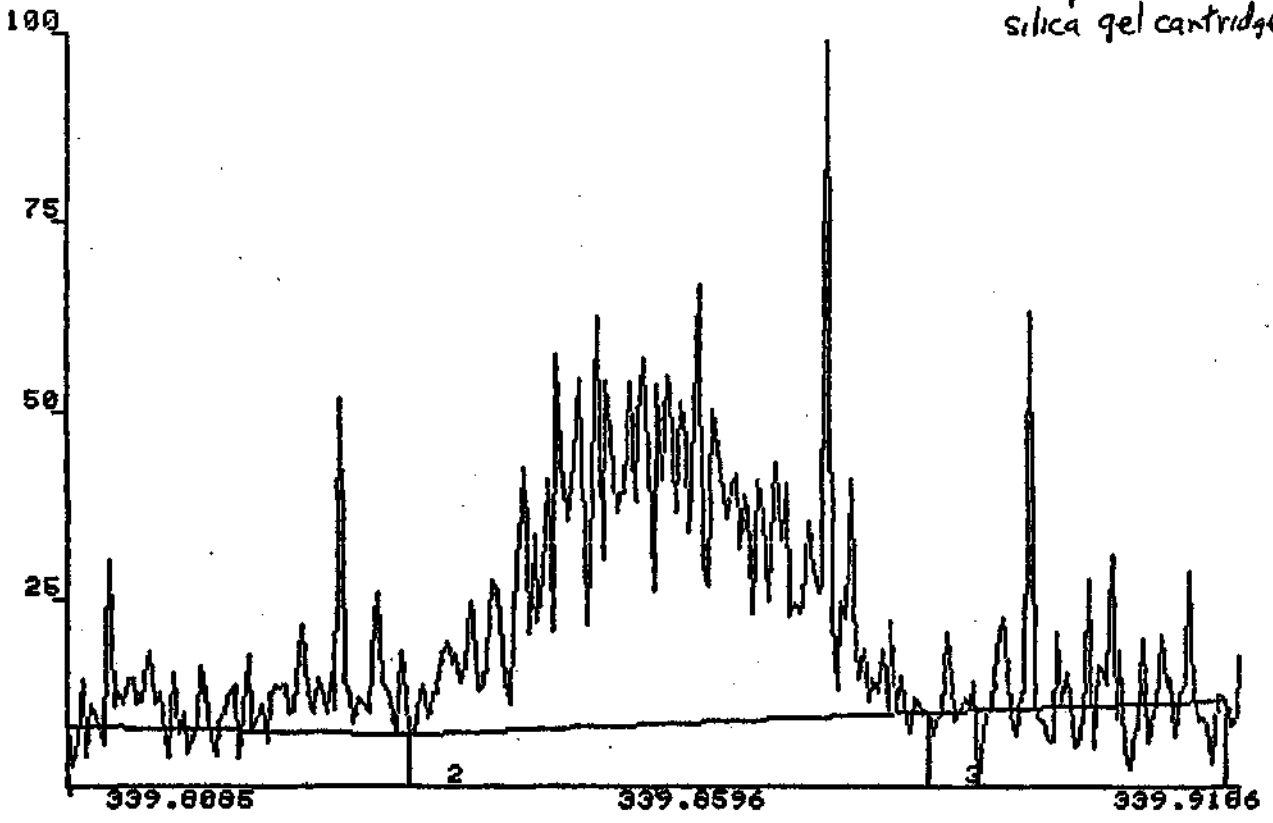

*

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



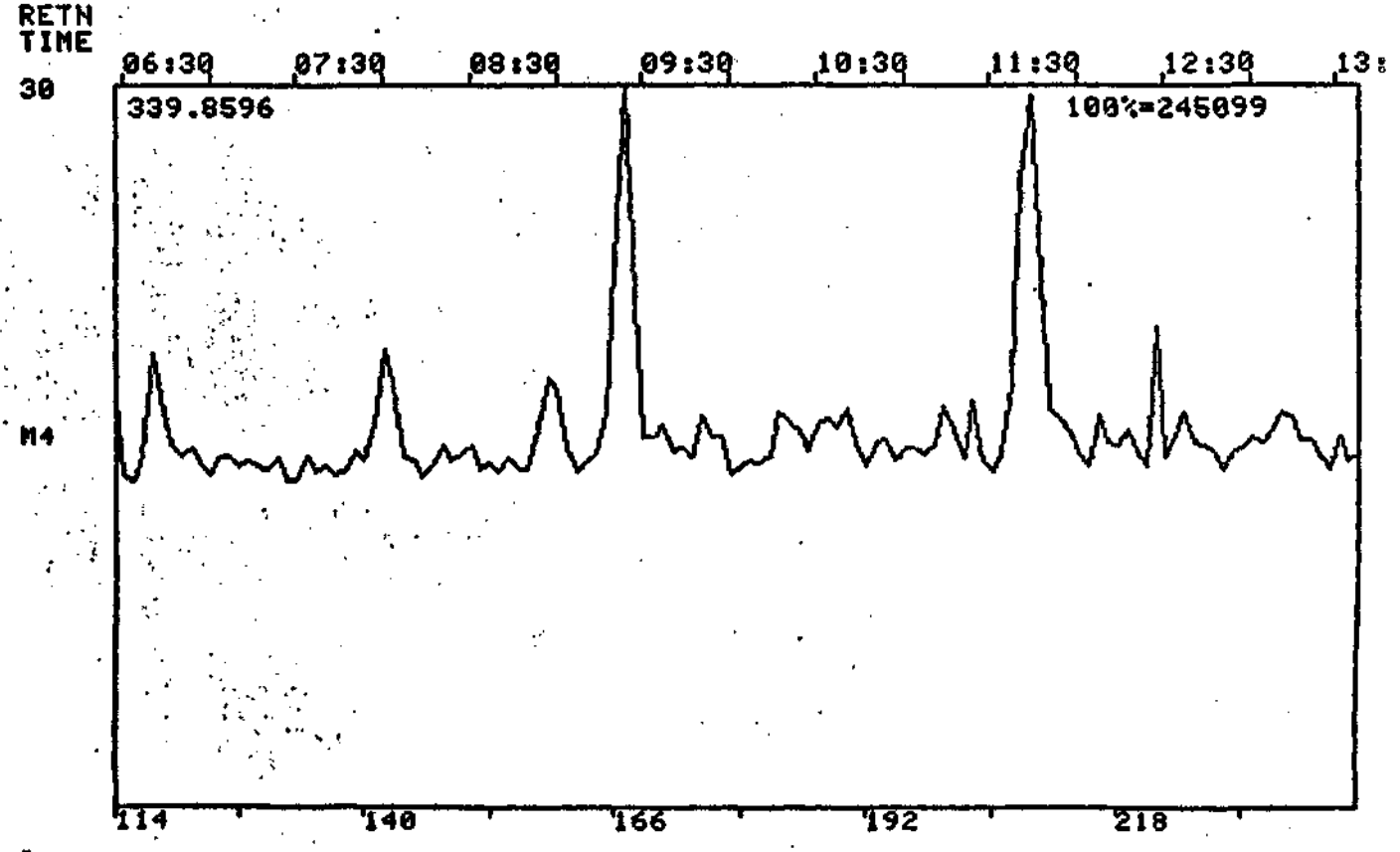
RUNNAME MDHMS DATE 3/28/83 TI
MASS 339.8596 SWEEP 300 (PPM)
SCANS 114-243 100% INTENSITY 106788

Figure 27: penta CDFs
Sample 17C1G
silica gel cartridge only




*SK AREA ID:1 *

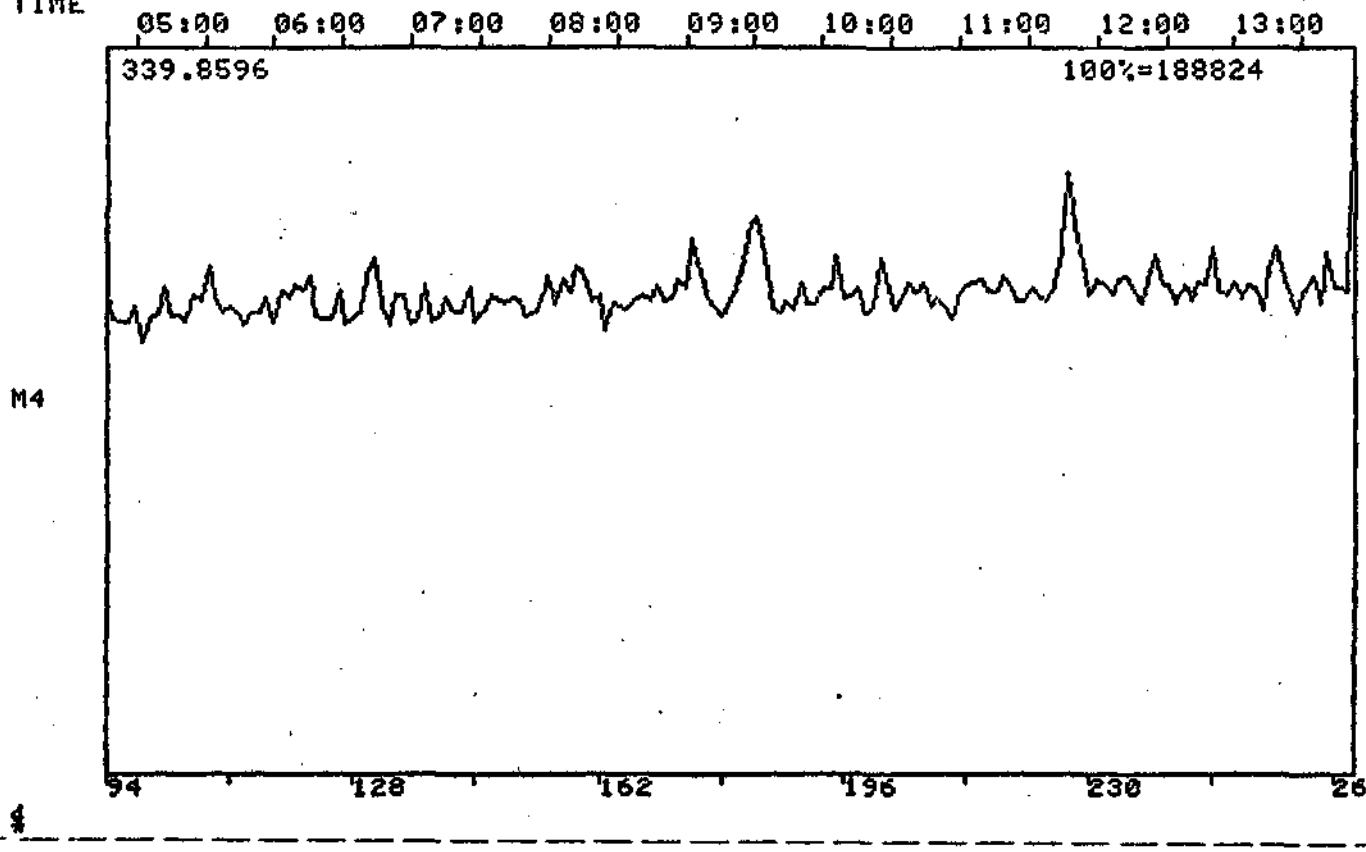
DS56 HIGH RESOLUTION MPM
RUNNAME MDHMS DATE 3/28/83 TIME 18: 0



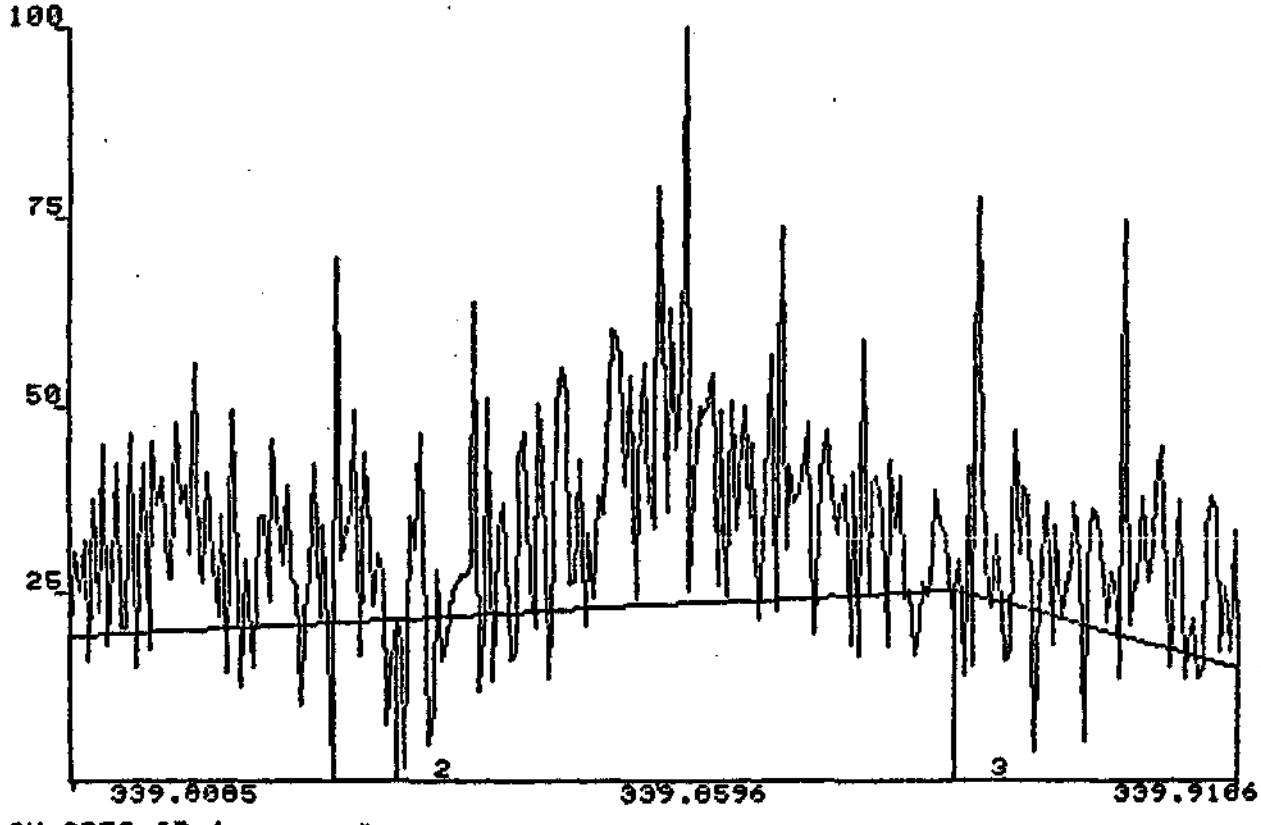

*

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

Figure 28: penta CDFs
Sample 17C1P
particulate only



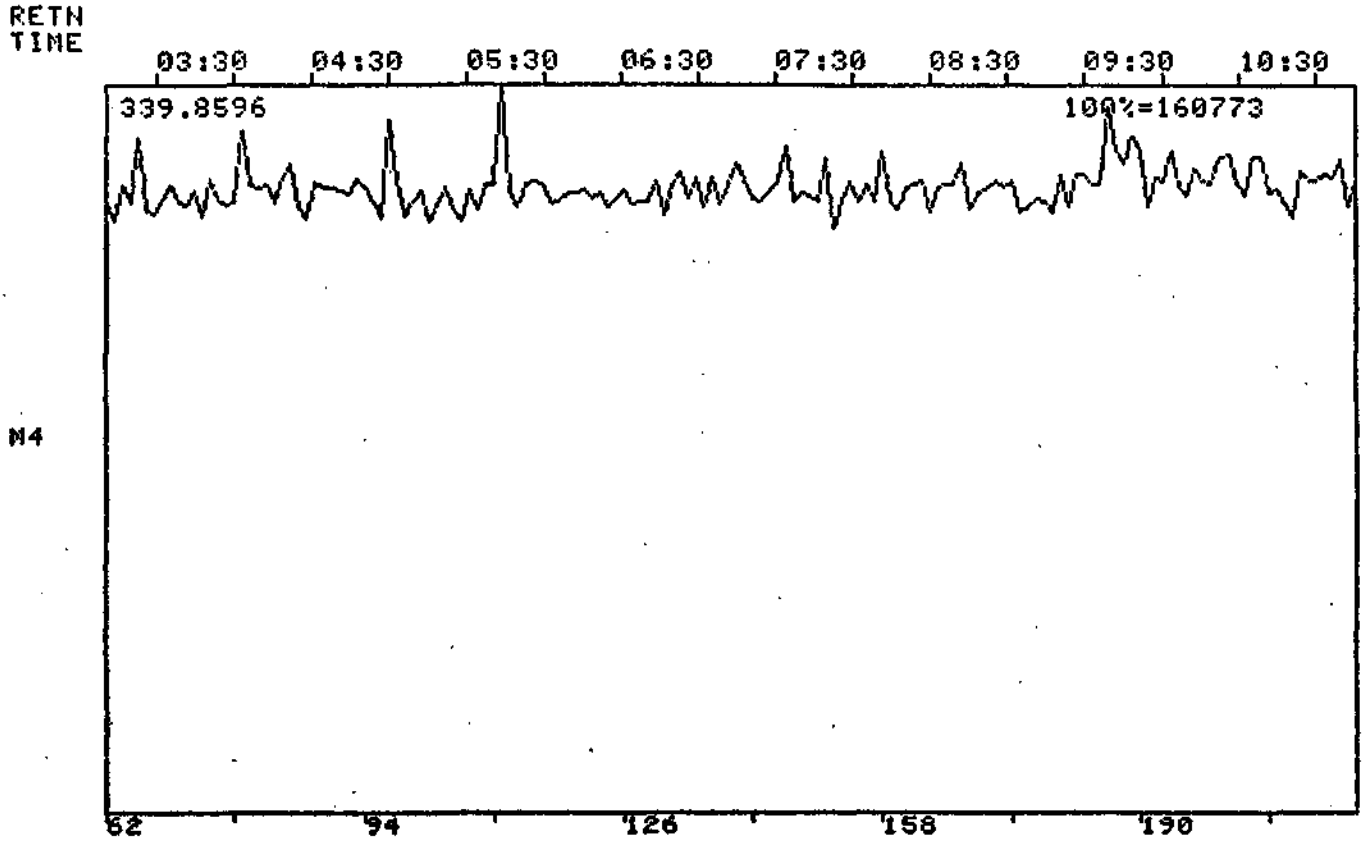

RUNNAME TUDHM4 DATE 3/29/83 TIME 10:29
MASS 339.8596 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)
SCANS 111-259 100% INTENSITY 88429




*SK AREA ID:1 *

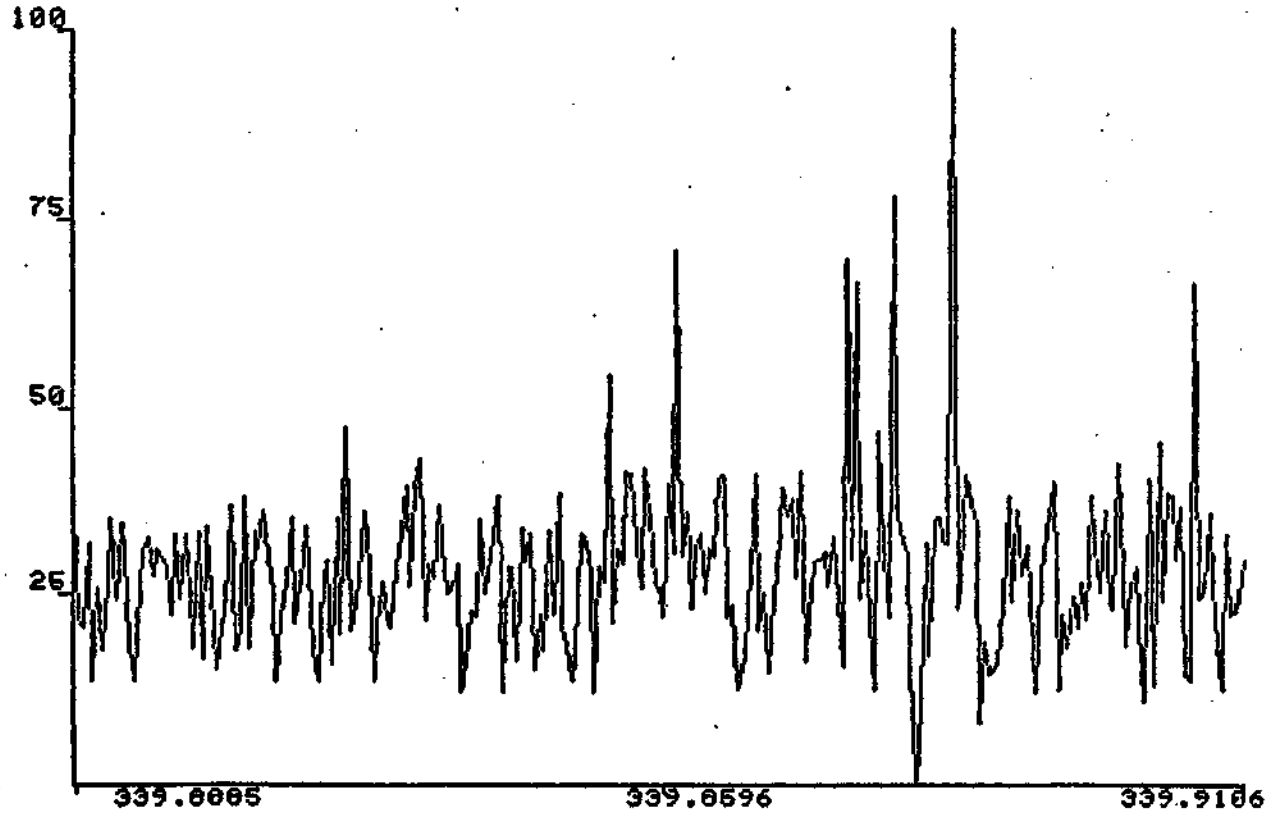
DS55 HIGH RESOLUTION MPM
RUNNAME TUDHM5 DATE 3/29/83 TIM

Figure 29: penta COFs
Solvent blank.




*

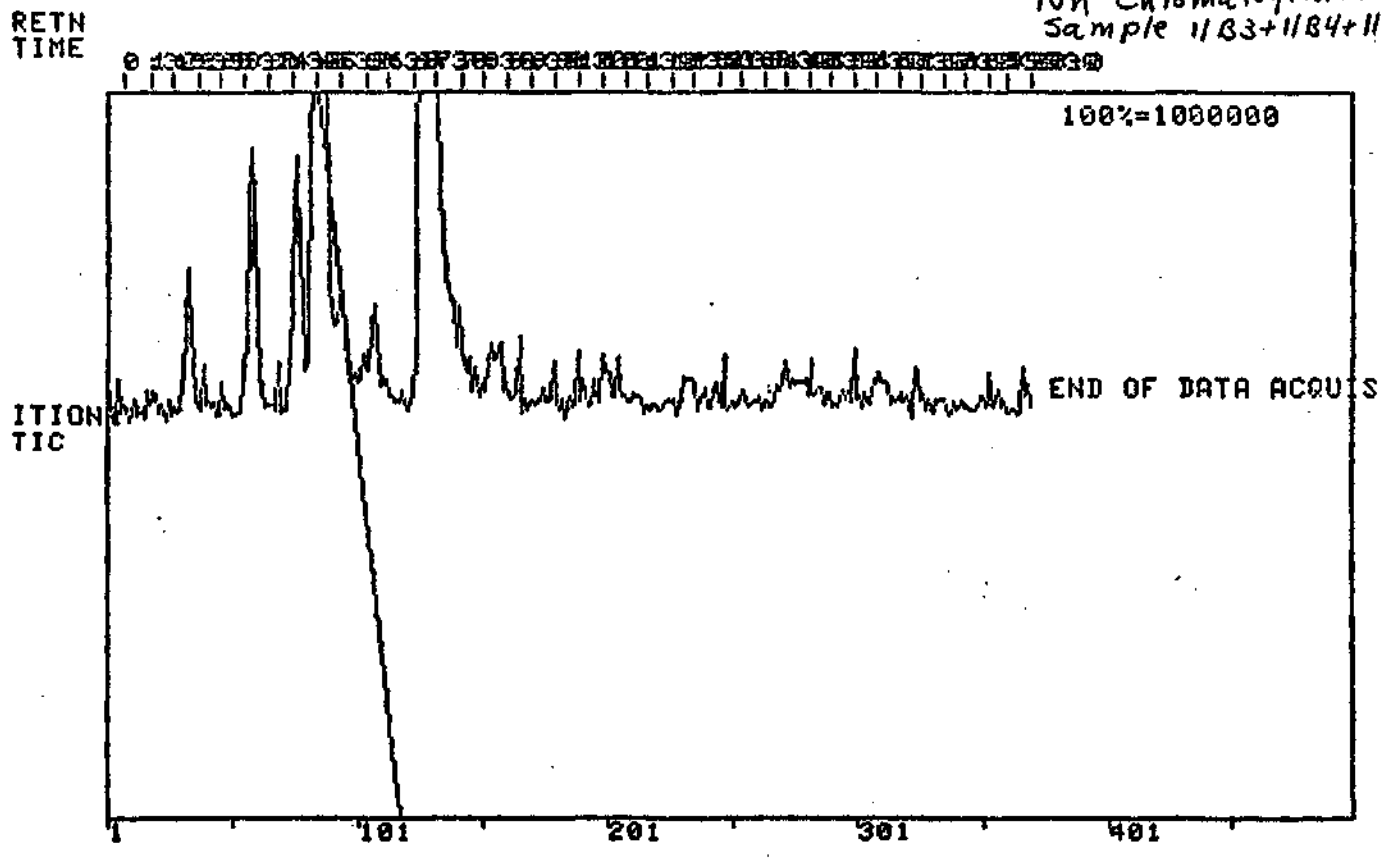
RUNNAME TUDHM5 DATE 3/29/83 TIME 11:40
MASS 339.8596 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)
SCANS 62-217 100% INTENSITY 108549



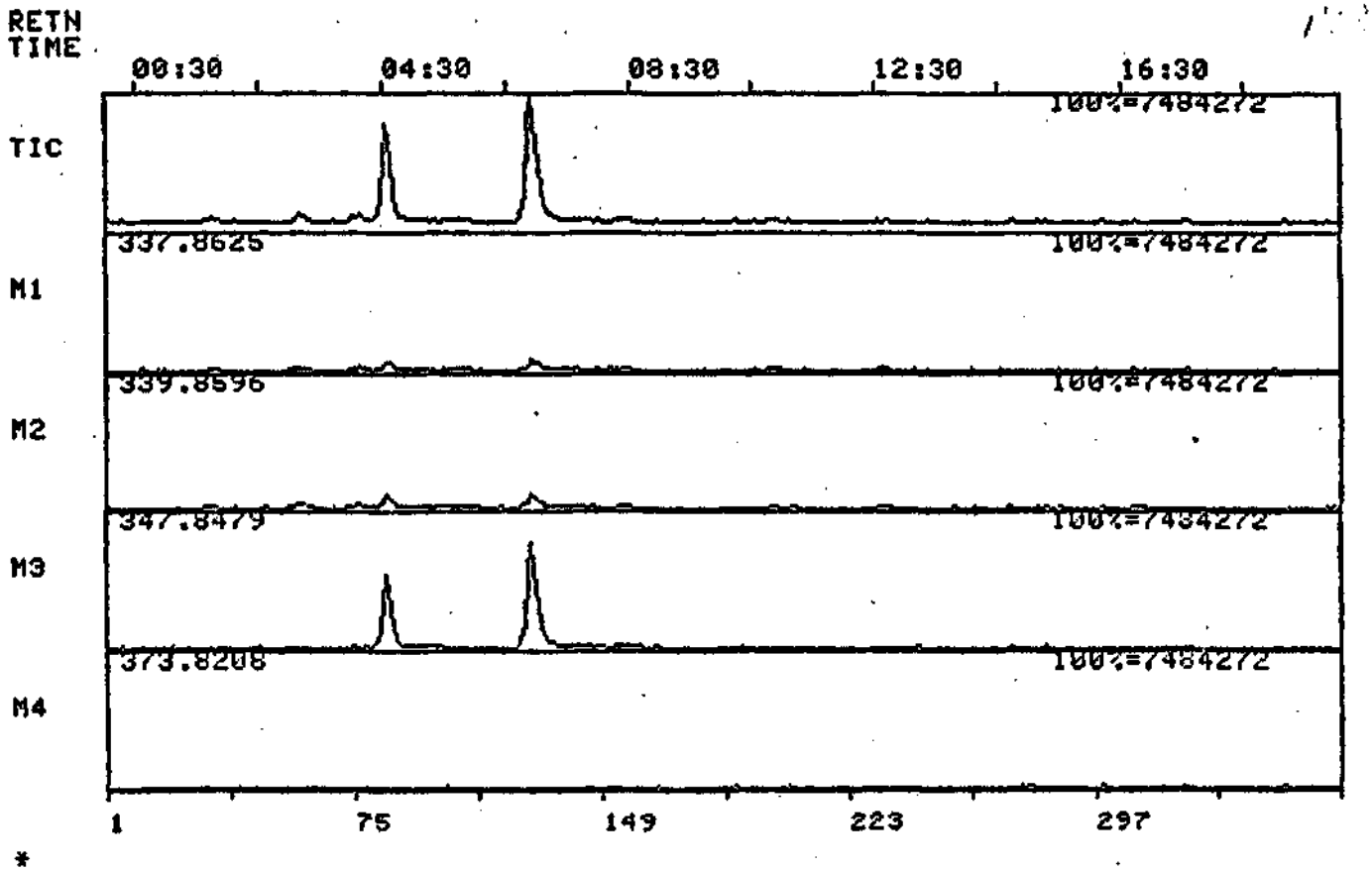

*

Figure 30: penta+hexa CDFs
2nd injection
ion chromatograms
Sample 11B3+11B4+11C1

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



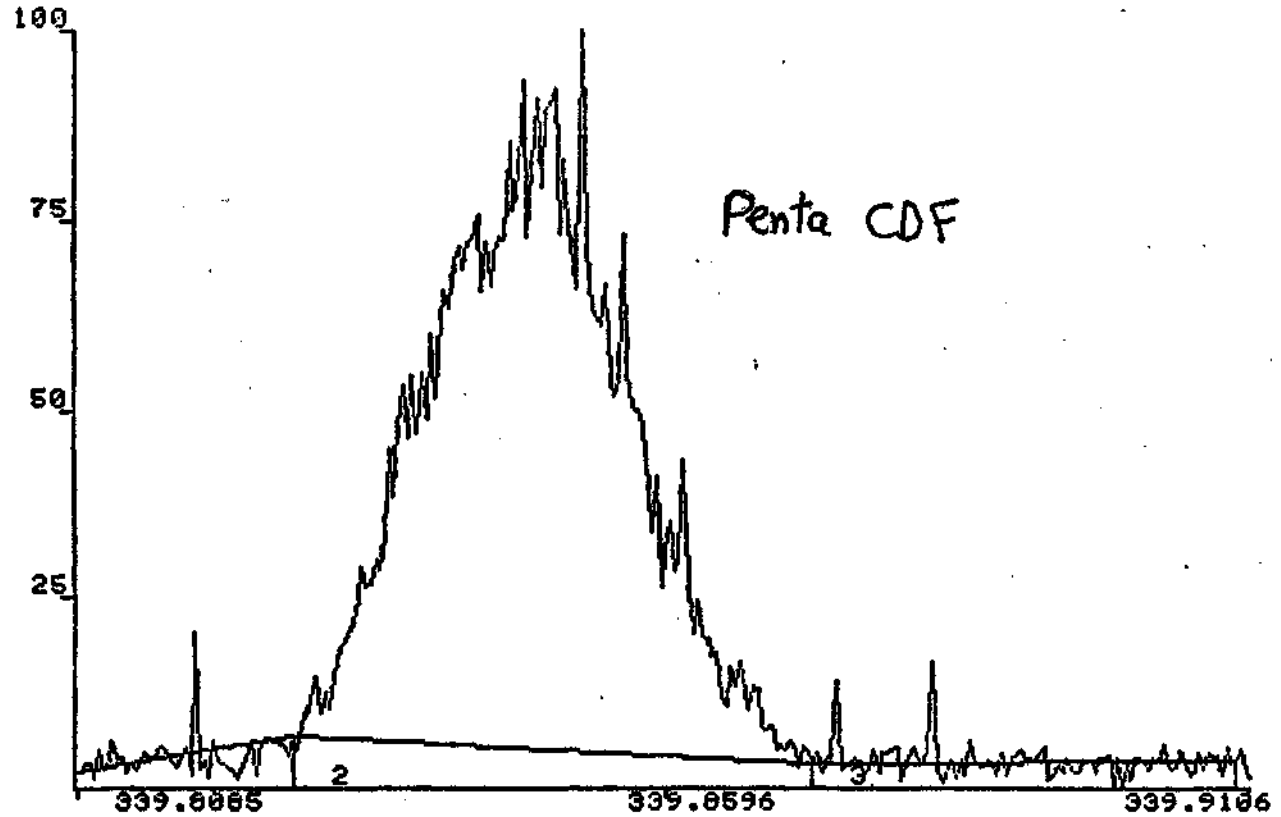

DS55 HIGH RESOLUTION MPM
RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11




*

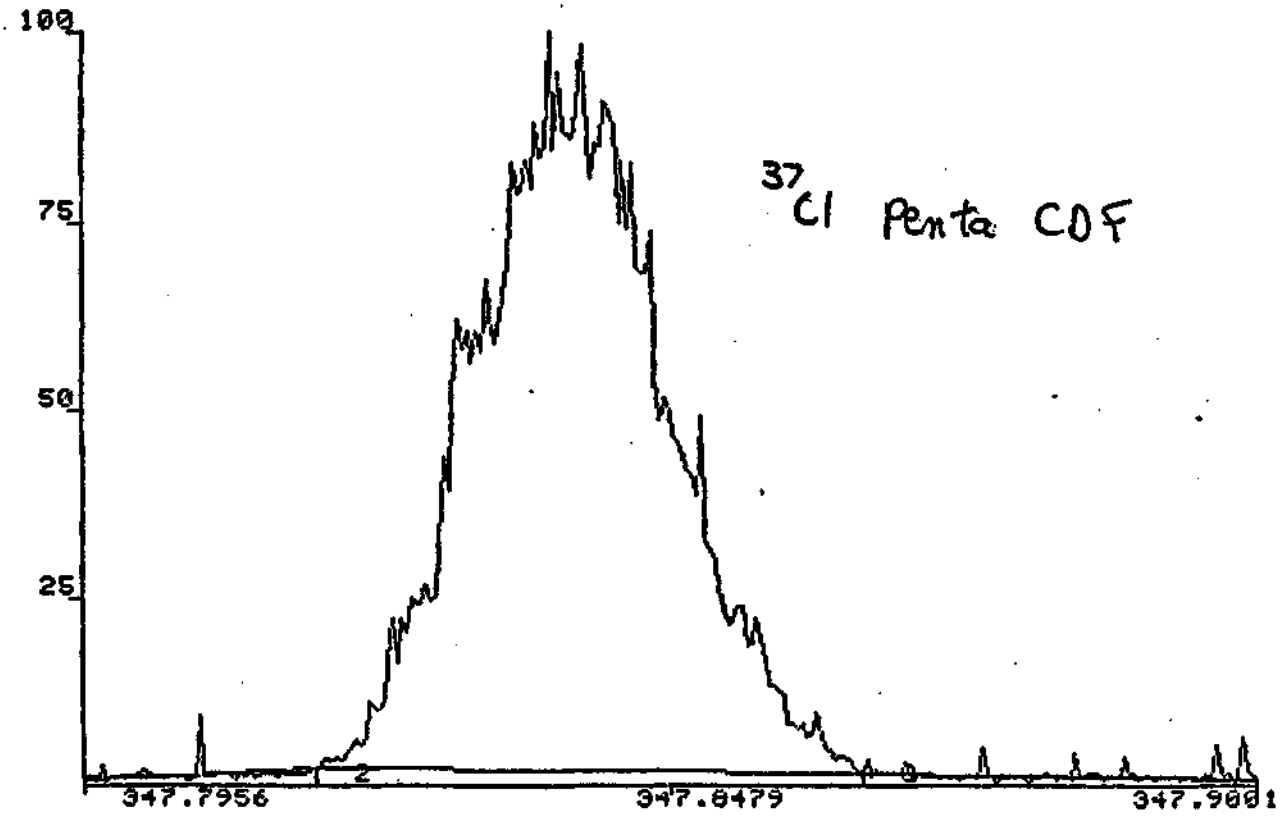
Figure 31: Mass profiles - penta CDF
2nd injection
Sample 11B3+11B4+11C1

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




*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 *

DS55 HIGH RESOLUTI Figure 32: mass profiles + area-percent
PEAK SUMMATION RE 2nd injection
Sample 11B3+11B4+11C1

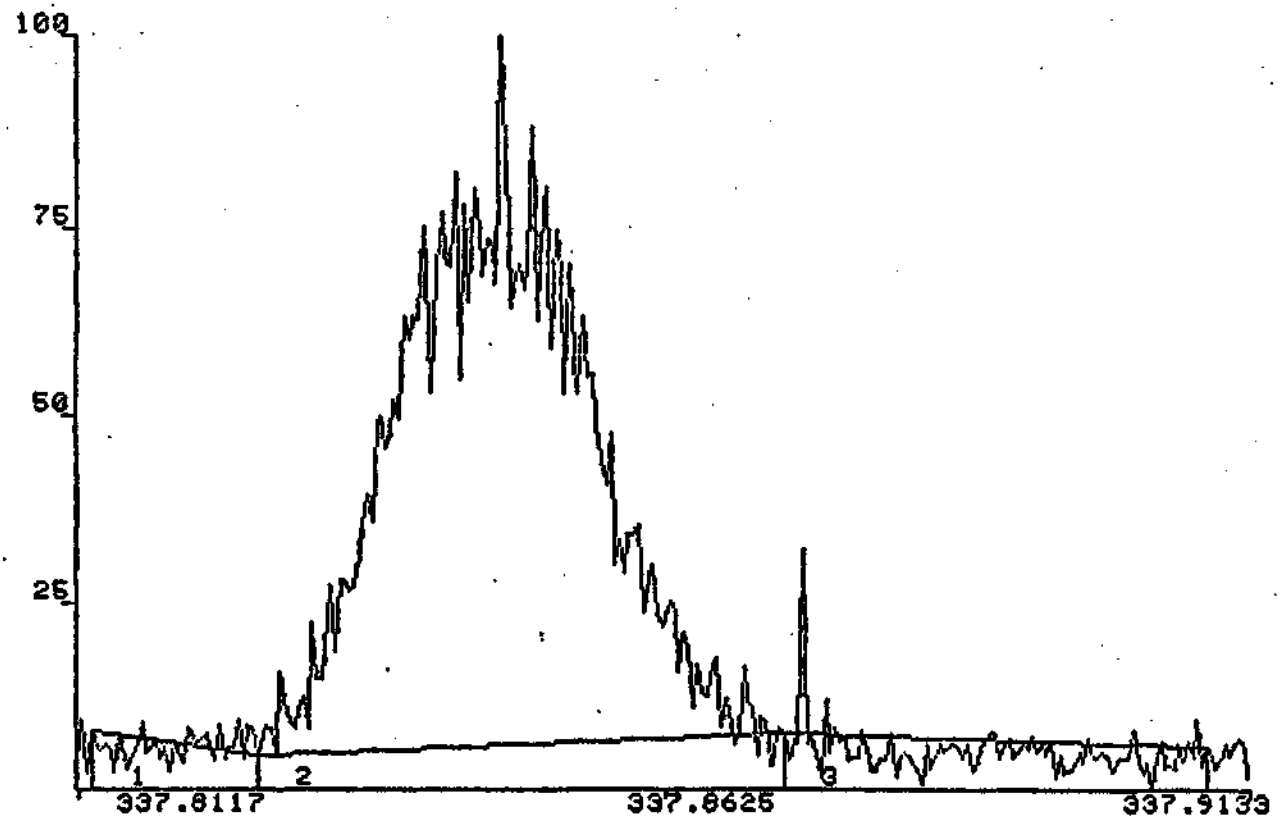
RUNNAME MDHM7 DATE 4/ 4/83 TIME 12:11

| | |
|---|---|
| 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 R[illegible]

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 | 161049. | YES | YES | 0.51 | 0.00 |

*

DS55 HIGH RESOLUTION MPM

PEAK SUMMATION REPORT

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

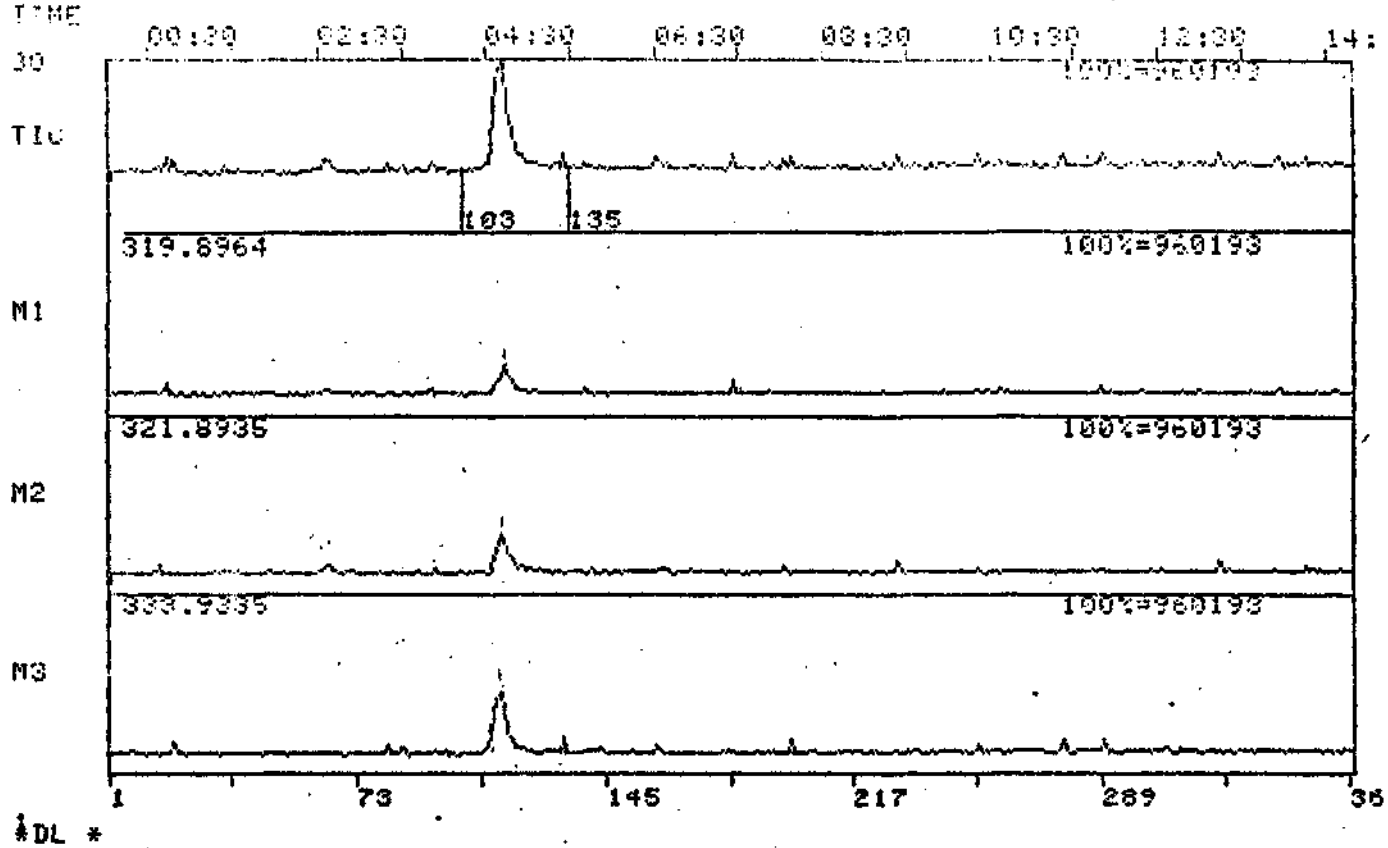
| | |
|---|---|
| MASS | 347.8479 |
| SCAN WIDTH | 300 PPM |
| SCAN TIME | 0.3 SECS |
| SCAN NUMBERS | 21- 166 |
| STANDARD | 0.0000 |
| FACTOR | 0 |

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

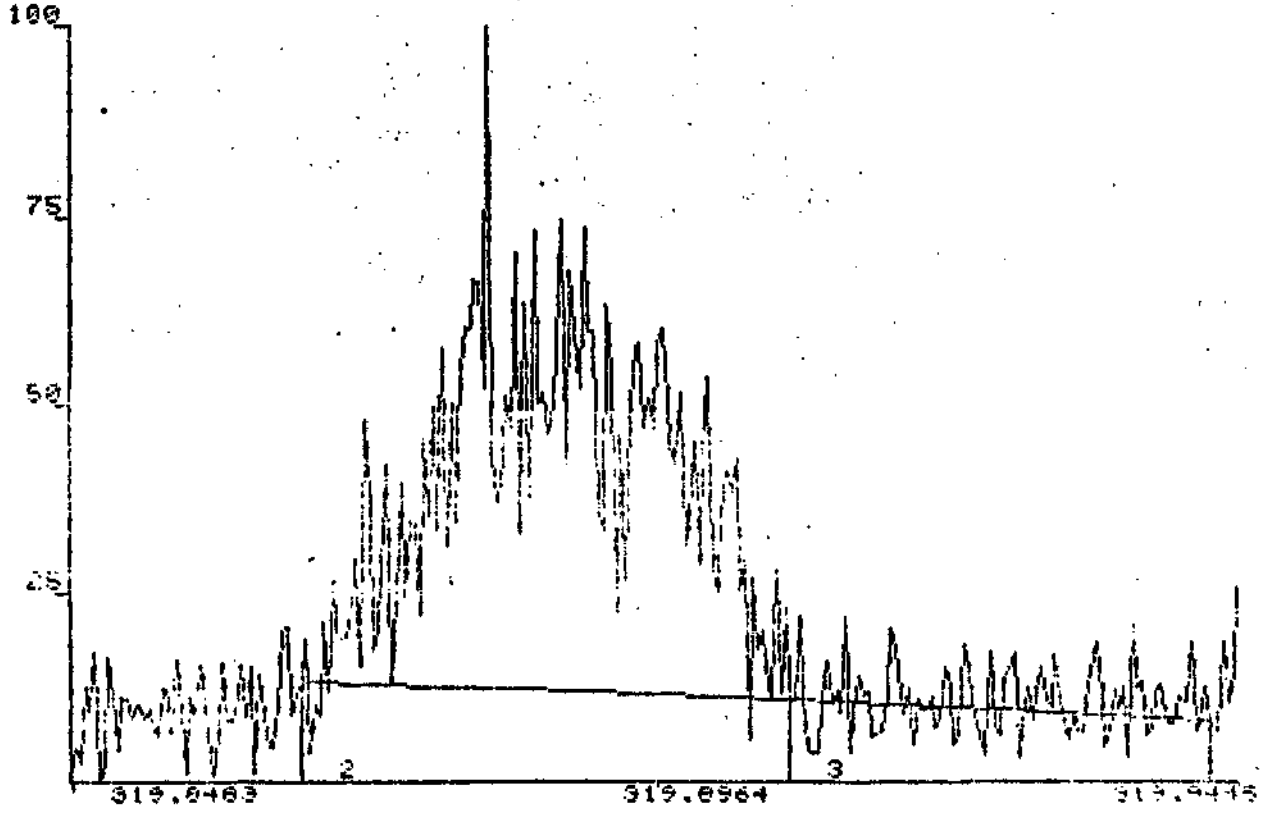
| MASS CENTROID | ITEM | AREA | BASELINE SUBTRACTED | BASELINE SKIMMED | %TOTAL AREA | RELATIVE TO STANDARD |
|---|---|---|---|---|---|---|
| 347.8428 | TOTAL | 35692510. | YES | NO | 65.95 | 0.00 |
| 347.8059 | 1 | 93650. | YES | YES | 0.17 | 0.00 |
| 347.8394 | 2 | 32597840. | YES | YES | 60.23 | 0.00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

*

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

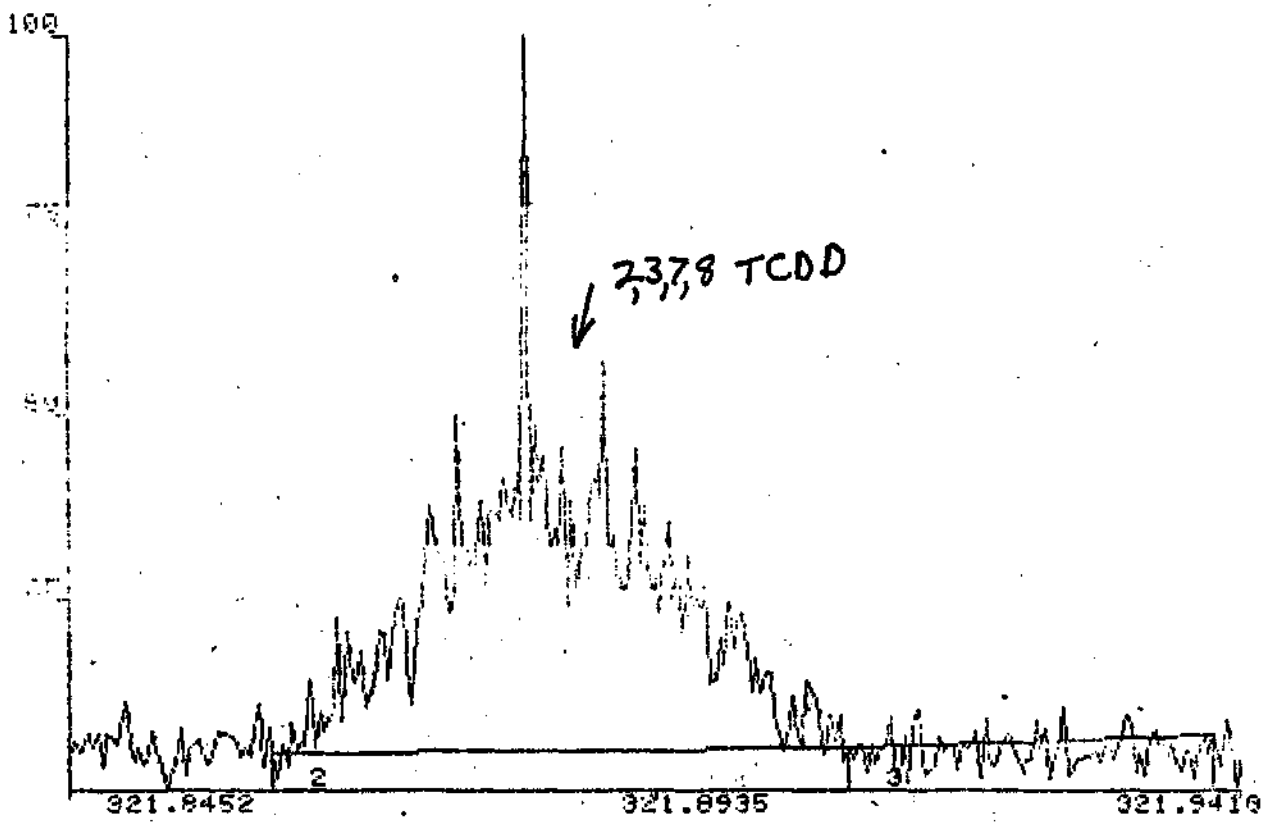



*DL *
RUNNAME FDHM9 DATE 3/25/83 TIME 16:49
MASS 319.8964 SWEEP 300 (PPM) SCANTIME 0.3 (SECS)
SCANS 103-135 100% INTENSITY 36335



RUNNAME FDHM9 DATE 3/25/83 TIME
MASS 321.8935 SWEEP 300 (PPM)
SCANS 103-135 100% INTENSITY 58332

Figure 35: Mass profile
Sample 11C1+11C2+11C4+11C5




*

DS5S HIGH RESOLUTION MFN

PEAK SUMMATION REPORT

RUNNAME FDHM9 DATE 3/25/83 TIME 16:49

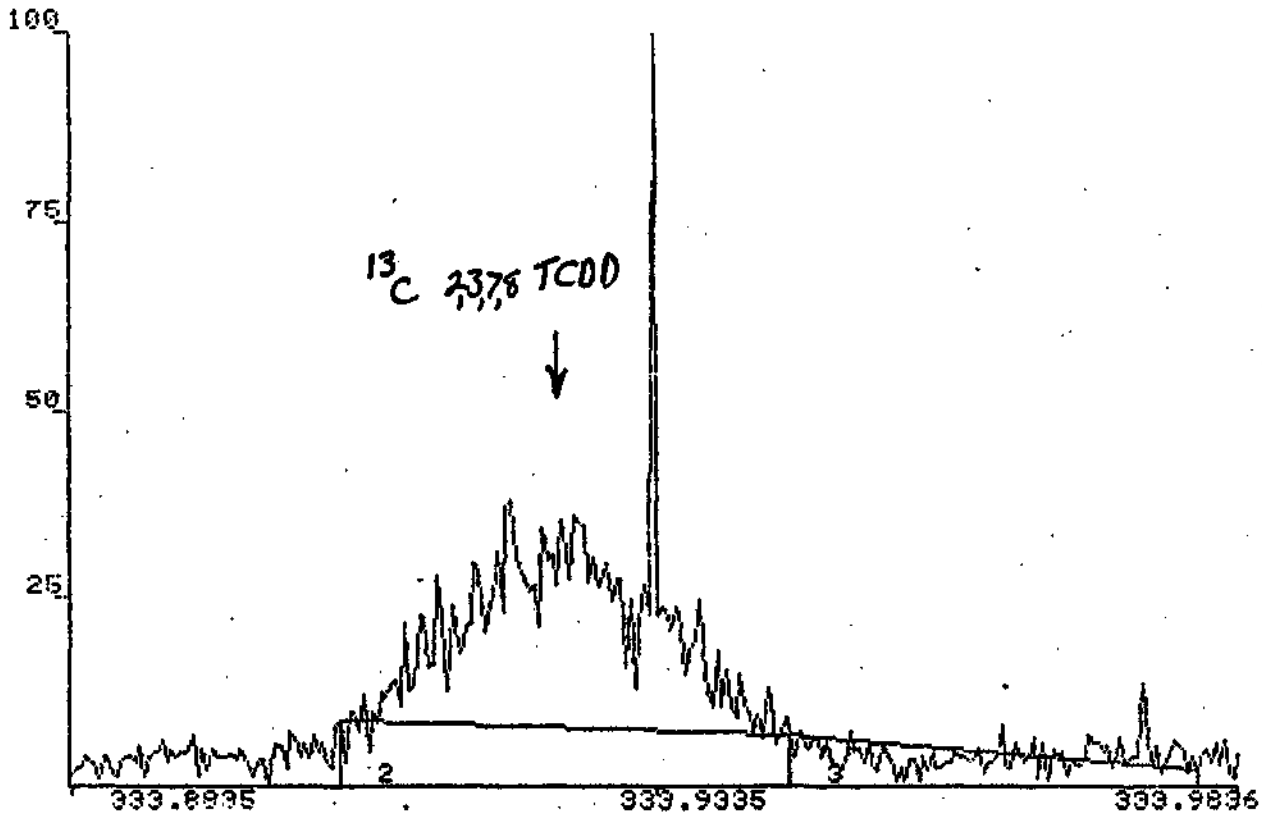
| | |
|---|---|
| MASS | 321.8935 |
| SCAN WIDTH | 300 PPM |
| SCAN TIME | 0.3 SECS |
| SCAN NUMBERS | 103- 135 |
| STANDARD | 0.0000 |
| FACTOR | 0 |

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

| MASS CENTROID | ITEM | AREA | BASELINE SUBTRACTED | BASELINE SKIMMED | %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 |
| 321.8953 | 2 | 1040165. | YES | YES | 21.19 | 0.00 |
| 321.9248 | 3 | 15910. | YES | YES | 0.32 | 0.00 |

RUNNAME FDHM9 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




DB55 HIGH RESOLUTION MPM

PEAK SUMMATION REPORT

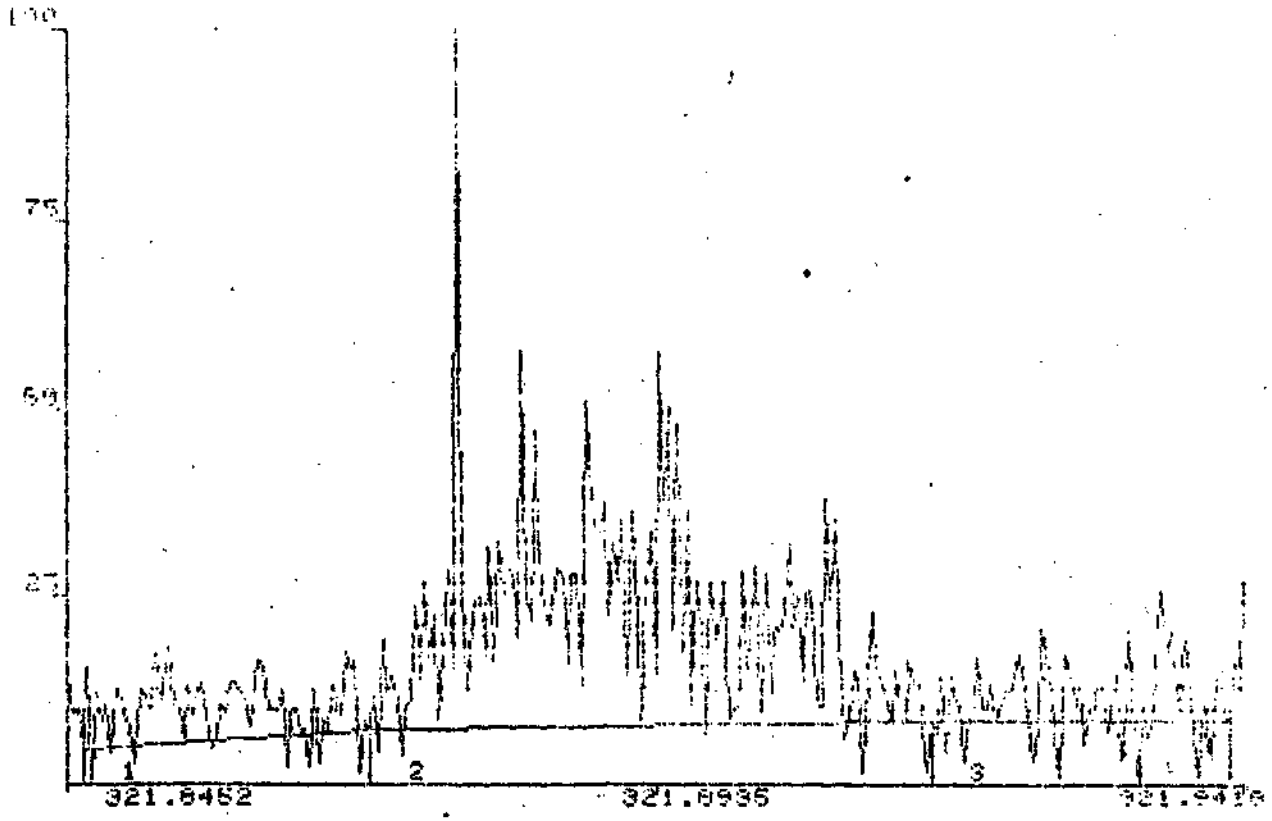
RUNNAME FDHM9 DATE 3/25/83 TIME 16:49

| | |
|---|---|
| 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 | 1106685. | YES | YES | 20.62 | 0.00 |
| 333.9629 | 3 | 53728. | YES | YES | 1.00 | 0.00 |

Figure 37: Mass profile for Solvent blank TCDDs




DS55 HIGH RESOLUTION MPM

PEAK SUMMATION REPORT

RUNNAME FDHM5 DATE 3/25/83 TIME 14:28

| | |
|---|---|
| MASS | 321.8935 |
| SCAN WIDTH | 300 PPM |
| SCAN TIME | 0.3 SECS |
| SCAN NUMBERS | 103- 123 |
| STANDARD | 0.0000 |
| FACTOR | 0 |

2.1 OF 2.3UL SOLVENT BLANK (2+3)

| MASS CENTROID | ITEM | AREA | BASELINE SUBTRACTED | BASELINE SKIMMED | %TOTAL AREA | RELATIVE TO STANDARD |
|---|---|---|---|---|---|---|
| 321.8933 | TOTAL | 659269. | YES | NO | 31.94 | 0.00 |
| 321.8582 | 1 | 45967. | YES | YES | 2.13 | 0.00 |
| 321.8931 | 2 | 393261. | YES | YES | 18.05 | 0.00 |
| 321.9295 | 3 | 99542. | YES | YES | 4.57 | 0.00 |