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Determination of Polychlorinated Dibenzofurans in Soot Samples from a Contaminated Office Building

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March 1982

ABSTRACT

The identification and quantitation of polychlorinated dibenzofurans (PCDF) in soot samples from the Binghamton State Office Building is reported. The analytical techniques and sample clean-up procedures are discussed and the identification of other major chlorinated combustion products given.

INTRODUCTION

On February 5, 1981, a soot-producing fire involving a transformer occurred in an office building in Binghamton, New York. The transformer contained a dielectric fluid with the trade name "Pyranol" consisting of polychlorinated biphenyl (PCB) Aroclor 1254 (65%) and chlorinated benzenes (35%) together with some trace additives. Preliminary analyses of a soot sample showed high levels of PCB and the presence of 3 ppm 2,3,7,8 TCDD and 100 ppm 2,3,7,8 TCDF (1). TCDFs and PCDFs are commonly found as contaminants in PCB formulations (2,3) and have also been shown to occur when PCB is heated under certain conditions (4).

Subsequent samples analyzed in our laboratory (5,6) include a soot homogenate used for animal toxicity studies and an air particulate sample taken inside the building using a high-volume air filter. Both samples were found to contain a mixture of TCDFs. The analysis of soot sample by two other dioxin laboratories verified the presence of Tetra Chlorodibenzodioxin (TCDD), TCDF and heavier chlorinated PCDDs and PCDFs (7,8). Several of the polychlorinated biphenylene compounds were also identified.

The present report describes the analysis of a representative soot sample from each of the 17 floors of the building and a soot homogenate used in animal toxicology experiments for PCDFs. Provision has been made in the clean-up and in data aquisition for the analysis of PCDDs but this will be discussed in a subsequent report.

METHOD

Sampling

Because a large portion of the contaminated building had already been cleaned to some degree, the particulate matter which had accumulatd undisturbed on the upper surface of suspended ceiling panels was sampled on each level. One half (2 ft. x 2 ft.) of a ceiling panel was wiped clean using a dry cellulose filter paper. Both the obtained particulates and the filter paper were stored inside a screw-capped glass test tube for analysis. Often adjacent samples were combined in the laboratory to provide enough weighable material for testing. A group of 8 samples was collected in the same building area for each of 16 floors. A sample of soot homogenate from several floors which was used in animal toxicology experiments was also included.

Extraction

Six to eighty milligrams of particulate were weighed out and placed into a glass extraction thimble containing 5 mm of silica gel (Bio Rad). Internal PCDD or PCDF standard in benzene was added to only 2 samples prior to extraction (added to all other samples after extraction) so other extracts could also be used for animal and cellular testing experiments. A glass soxhlet extraction apparatus was then charged with 100 ml benzene and the particles were continuously extracted for 16 hours.

A solvent blank, a carbon blank (active coconut charcoal), and four recovery control samples were similarly extracted and analyzed. All extracts were stored in the dark.

Collaborative Studies

Separate portions of each crude extract were provided for PCB analyses and for use in cell keratinization studies.

Sample Clean-up

The benzene extract was concentratd to 5 ml using a boiling water bath. An aliquot of the sample was spiked with 80 1 of mixed, labeled internal standard (^{13}C 2,3,7,8 TCDD, ^{37}Cl TCDF, ^{37}Cl OCDD) and cleaned-up using three sequential liquid chromatographic columns as follows:

Each sample was diluted with acetone to 20% benzene and injected onto a low pressure LC system using a column of 50 mg PX-21 adsorptive carbon mixed with 600 mg celite. This column is known to strongly adsorb planar, halogenated aromatics. The sample was washed with 40 ml of 20% benzene/acetone. Flow through the column was reversed, and the fraction containing PCDDs and PCDFs was eluted with 30 ml toluene.

The toluene was removed in boiling water bath using a stream of N_2 and the solvent was changed to dodecane. The sample was then applied to a 1 cm id x 8 cm long column containing 2% deactivated silica gel (Bio Rad) and eluted with hexane. The 0-10 ml fraction containing the PCDDs and PCDFs was collected. This chromatographic step is effective at eliminating relatively polar, highly colored components.

The sample was then directly applied to a 1 cm x 8 cm long column containing activated Florisil (180^{o} overnight). The column was washed with 20 ml benzene to remove PCBs and PCDD/PCDF fraction was eluted using 20 ml of 3% CH_3CN, 47% CH_2Cl_2, 50% Hexane. The sample was then concentrated to 80 1 in

benzene prior to capillary Gas Chromatography/high resolution Mass Spectrometry (GC/HRMS).

Instrumental Conditions

Gas Chromatography - A Carlo-Erba Model 4160 Capillary Gas Chromatograph with on-column injection was used with a 30m x 0.32 mm i.d. fused silica "Durabond" DB-5 column (J & W Scientific) and helium carrier gas. The effluent from the GC column was coupled via an open-split interface to the mass spectrometer source re-entrant using a length of 0.16 mm i.d. fused silica tubing de-activated with 2% Carbowax 20M in Methylene Chloride and coated with a 15% OV-17 in Methylene Chloride then conditioned at 50°C to 300°C at 1°C/min. The GC oven was programmed during analysis as follows: 70°C to 180° at 10°C/min then 3°C/min to 270°C and hold isothermal for 20 minutes. The GC/MS interface region was held isothermally at 275°C and typical on-column injection volumes were 2 1.

Mass Spectrometer - A Kratos MS-50 mass spectrometer operated in full scanning mode was used to acquire GC/MS data. The spectrometer was tuned to 10,000 (10% valley) dynamic resolution and scans were taken at 3 seconds per decade over the mass range $^{M}/_{Z}$ = 600-150 resulting in a 4 second cycle time scan. Perfluorokerosene (PFK) was used as a mass standard for high resolution scanning and was introduced concurrently with the GC effluent into the source of the mass spectrometer which was operated in electron impact (EI) mode at 70 eV, 8kV accelerating voltage and a source temperature of 250°C. Data was acquired during GC/MS runs using the DS-55 data acquisition system via the

preprocessor interface at a sampling rate of 100kH$_z$ and stored on disk as sample-time data for subsequent mass conversion. Each run comprised ca 800 scans each of which contained data from the mass standard reference peaks and mass peaks generated by compounds eluting from the gas chromatograph. The file of sample-time data was mass converted after acquisition producing a file of mass measured peaks and associated intensities, the exact mass being computed for the unknown peak by reference to the mass standard peak with a precision of ca 10ppm. The data system then allowed each GC run to be displayed in various graphical forms or as exact mass listings.

Post-Run Data Processing

To generate useful quantitative and qualitative information from the acquired GC/MS run, several data processing routines were employed. Firstly, sample-time data was mass converted to generate high resolution scanned data files. A total ion current (TIC) and exact mass chromatogram was then generated for mass ions of each compound group of interet along with a computer-generated worksheet. This worksheet then allowed the operator to select scan windows which encompassed GC peaks of interest. The scan windows were then fed into a software program which generated scan by scan exact mass-intensity reports ever specified mass ranges. These reports were then used to allow quantitation of the exact mass ions over the GC peak of interest by summing the intensity for each scan over the deviation of the GC peak. The facility to do this with software had not yet been developed for exact mass data. The resulting areas of the exact

mass chromatogram were used for quantitation and verification of ion abundance ratios. This process for a standard run is shown in Figures 1-27.

Quantitation of PCDFs

The only available labelled reference standard for PCDF was a [U-$^{37}Cl_4$]-TCDF sample obtained from KOR Isotopes Inc. As can be seen from Figure 3 the material was not pure and contained several Tetra isomers. Previous work had indicated that it contained less than 2% unlabelled material for all PCDF and PCDD congeners. This material was therefore used as internal standard and provided a method for calculating recovery of the clean-up procedure. Native PCDFs were available for Tetra CDF, Hexa CDF and Octa CDF and these compounds were used for external standardization. A mixture of native and labelled PCDF was run each day and data was collected on samples to verify response factors and instrument performance.

Quantitation of the samples was therefore performed using response factors obtained from the calibration run for Tetra, Hexa and Octa CDF. Response factors for Penta and Hepta CDF were linearly interpolated from adjacent congener responses. The recovery of each sample through the clean-up procedure was also calculated from the [U-$^{34}Cl_4$]-CDF response. Quantitation was therefore not isomer specific but represents a total quantity of each congener group and assumes equivalent responses for isomers within each group and the validity of the response interpolation for Penta and Hepta CDF.

Quality Control and Detection Limits

The Mass Spectrometer was tuned to 10,000 (10% valley) Resolving Power daily using PFK as mass standard and High Resolution Calibration was performed before the start of each run. A standard injection of PCDF and PCDD reference compounds was then run and calibration of response factors was performed. Table 2 lists the standard runs used for quantitation over the period of ca 2 months indicating an overall RSD of ca 25% for the various measured response factors. Detection limits were also calculated using the standard runs assumming a typical GC peak width of 6 scans and noise level of 250 counts per scan for a signal to noise level of $^{S}/N=2$. In addition for positive identification the 3 most intense ions in the parent cluster must show close agreement to the theoretical ion ratios and exact masses, retention time must agree with standard runs and mass spectrum obtained must match that of the standard reference spectrum.

Results and Discussion

Table I lists the quantitation for PCDFs in the 16 samples collected from each floor of the Binghamton State Office Building and the soot homogenate used for animal toxicology experiments. Recoveries based on [U-$^{37}Cl_4$]-Tetra CDF are reported and values for the samples are not corrected for recovery. The significance of these data and correlation with other testing is presented elsewhere (9).

Data from three GC/MS runs are presented in more detail to indicate the information that is available for each sample run. The extract of the soot obtained from the 5th floor and the soot homogenate is presented to be representative of the sample data and a standard used to quantitate the soot homogenate is also shown.

Figures 1-27 correspond to the standard run and show the Total Ion Current (Figure 1) and exact mass chromatograms for each compound and congener present. The tabular reports (Figures 2 and 10) are worksheets which allow the analyst to enter the scan ranges visually determined to contain the components for quantitation. Quantitative reports are then generated for these scans which give the exact mass and intensities of peaks within the specified mass range. These have been included for the Hexachlorodibenzofuran (Hexa CDF) standard Scans 437 to 446 over the time period when this compound was eluting from the GC (Figures 17-27). Summing the intensities for the three most intense ions in the parent group, i.e., 371.8236, 373.8207, 375.8177, give the total intensities of 113,735, 211,168, 158,794, respectively which agree well with the theoretical ion

ratios. The intensity of the 100% ion (373.8207) is used for computing the response factor and quantitation of samples. This method of peak integration was used for the quantitation of GC peak area for all standard and sample runs. Scan were only included in the summation if the exact mass of the 3 ions was in good agreement (better than 100ppm) and ion ratios were consistent with theoretical ratios (Table 4).

Figures 28-37 correspond to the GC/MS data from the 5th floor sample processed in a similar way to the standard. Figure 28 is a total ion current chromatograph the most intense GC peak being due to diisooctylphthalate is a contaminant found in all sample runs and to a lesser extent the standard runs. Figure 29 shows the exact mass chromatograms for each of the Cl_3-Cl_8 congeners of PCDF simplified by plotting only the most intense ion in the parent group. Figures 30-34 are mass spectra obtain from each congener group identified in the exact mass chromatograms. Figure 30 indicates the presence of Pentachloronaphthalene (exact mass 299.8648) as well as Trichlorodibenzofuran (exact mass 271.9376), in the spectrum of scan #242. Polychloronaphthalenes appears to be major components in the soot samples and are also found in the soot homogenate sample.

Figures 38-51 correspond to the data obtained for the soot homogenate sample. The total ion current chromatogram again indicates as a major GC peak the diisooctylphthalate (Figure 38). Figure 39 shows exact mass chromatograms for PCDFs and Figures 40-45 give typical full scan spectra for each congener. Figure 46 indicates the exact mass chromatograms for

Polychloronaphthalenes with intense peaks for Cl_5, Cl_6 and Cl_7 Chloro congeners.

Figure 47-51 again shows full can spectra for each of the Chloronaphthalene congeners. Quantitation has only been made for the PCDFs but as indicated qualitative evidence for Polychlorinated naphtahlenes has been shown and trace levels of Pentachlorobiphenylenes (see Figure 36A) at exact mass 323.8647 we indicated in the 5th floor sample. Carbon and solvent blanks showed no evidence of contaimination at the detection limits indicated. The vast amount of high resolution data is continuing to be reviewed to complete the data analysis and software development continues to aid in the process of data reduction and quantitation.

References

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2. C. Rappe, H.R. Buser and H.P. Bosshardt, CIPAC Symposium, Baltimore, June 5-6, 1979.
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TABLE I

Concentration (ppm) of Polychlorinated Dibenzofurans in Soot Samples Taken from the Bing

Floor #	1	2	3	4	5	6	7	8	9	10
PCDFs[4] Tetra CDF	0.06		1.8	2.5	44	5.1	77	35	160	47
^{37}Cl Tetra CDF (ng)[1]	10.7ng		3.8ng	7.2ng	4.4ng	8.6ng	14.2ng	9.6ng	1.9ng	6.5ng
Penta CDF	<0.06		<2.0	3.2	55	8.0	75	40	220	55
Hexa CDF	<0.07		<2.4	2.0	36	3.0	14	16	140	28
Hepta CDF	<0.08		<2.8	0.2	12	0.6	1.5	7.1	51	2.1
Octa CDF	<0.09		<3.1	<.2	4.2	<.2	--	1.4	17	0.56
[5]Recovery %	29		22	16	20	22	41	50	49	33

[1]Internal standard amount recovered (ng)

[2]Quantitated as Arochlor 1254

[3]Data lost due to instrument malfunction

[4]Data uncorrected for recovery

[5]Recovery based on ^{37}Cl Tetra CDF internal standard

TABLE 2

Reproducibility of Standard Runs

Response Factors [Counts/ng]

Date of Run	Tetra CDF	$^{37}Cl_4$ Tetra CDF	Hexa CDF	Octa CDF
1/26/82	1380	4938	771	630
1/29/82	1193	3987	834	913
1/30/82	1475	4904	815	660
2/10/82	1803	5358	953	793
2/15/82	1306	4777	919	754
2/15/82	1802	6568	---	874
2/16/82	1354	5926	1225	837
3/3/82	1880	7017	1339	1075
3/4/82	1961	6690	1492	1178
3/5/82	1978	6794	1271	1193
3/10/82	2616	9606	1700	1336
3/11/82	2293	8622	1472	1147
Average Response	1753	6265	1163	978
Standard Deviation	430	1645	320	217
%RSD	25	26	27	22

TABLE 3

Average Detection Limits For PCDFs[1]

	Average Response Factor counts/ng	DL(ng)S/N=2	[2]PPM in Soot
Tetra-CDF	1753	1.7	0.17
$^{37}Cl_4$-TCDF	6265	0.5	--
Penta-CDF	1458	2.0	0.2
Hexa-CDF	1163	2.6	0.26
Hepta-CDF	1070	2.8	0.28
Octa-CDF	978	3.0	0.30

[1]Based on GC peak width 6 scans noise level 250 counts/scan

[2]Assuming an injection volume equivalent to 10ng of extracted sample

Table 4

Exact Masses and Intensities of Molecular Ions of Chlorinated Dibenzofurans

Chlorination Number (Molecular Formula)	Exact Mass (3)	Intensities
Monochloro	202.018539	100.0
$C_{12}H_7OCl$	204.015590	32.5
Dichloro	235.979568	100.0
$C_{12}H_6OCl_2$	237.976618	65.0
Trichloro	269.940596	100.0
$C_{12}H_5OCl_3$	271.937647	97.5
	373.934697	31.7
Tetrachloro	303.901625	76.9
$C_{12}H_4OCl_4$	305.898675	100.0
	307.895726	48.7
Pentachloro	337.862653	61.5
$C_{12}H_3OCl_5$	339.859704	100.0
	341.856754	65.0
Hexachloro	371.823682	51.2
$C_{12}H_2OCl_6$	373.820732	100.0
	375.817783	81.2
Heptachloro	407.781761	100.0
$C_{12}HOCl_7$	409.778811	97.5
	411.775861	52.8
Octachloro	441.742789	87.9
$C_{12}OCl_8$	443.739840	100.0

DS-55 CROSS SCAN REPORT, RUN: TD1S1 Fig. 1 - TIC Chromatogram of typical standard injection

+ TIC

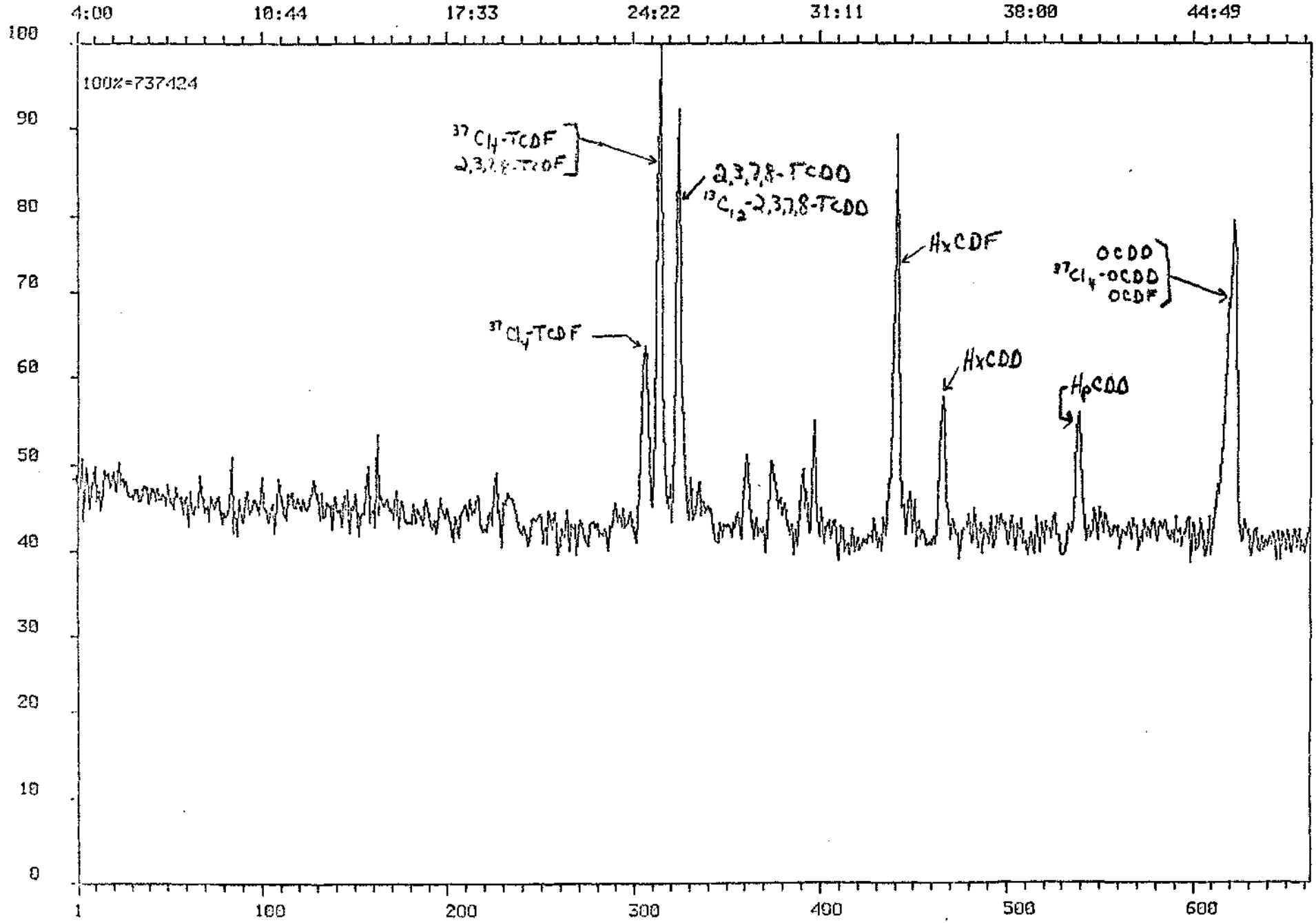


Fig 2 - Worksheet used to list scans containing intensity of compounds of interest

TABLE OF POLYCHLORODIBENZOFURAN MASSES, INTENSITIES AND SCAN NUMBERS (FROM XSCAN DATA)

TRICHLORO- 270(100.0), 272(97.5)
SCANS: * REFINED SCANS:

DICHLORO- 236(100.0), 238(65.0)
SCANS: * REFINED SCANS:

MONOCHLORO-202(100.0), 204(32.5)
SCANS: * REFINED SCANS:

TETRACHLORO- 304, (76.9), 306(100.0), 312(INTERNAL STANDARD)
SCANS: 285-345 (m/z 303-334) REFINED SCANS:

PENTACHLORO- 338(61.5), 340(100.0)
SCANS: * REFINED SCANS:

HEXACHLORO- 374(100.0), 376(81.2)
SCANS: 335-355 (m/z 371-384) REFINED SCANS:

HEPTACHLORO- 408(100.0), 410(97.5)
SCANS: * REFINED SCANS:

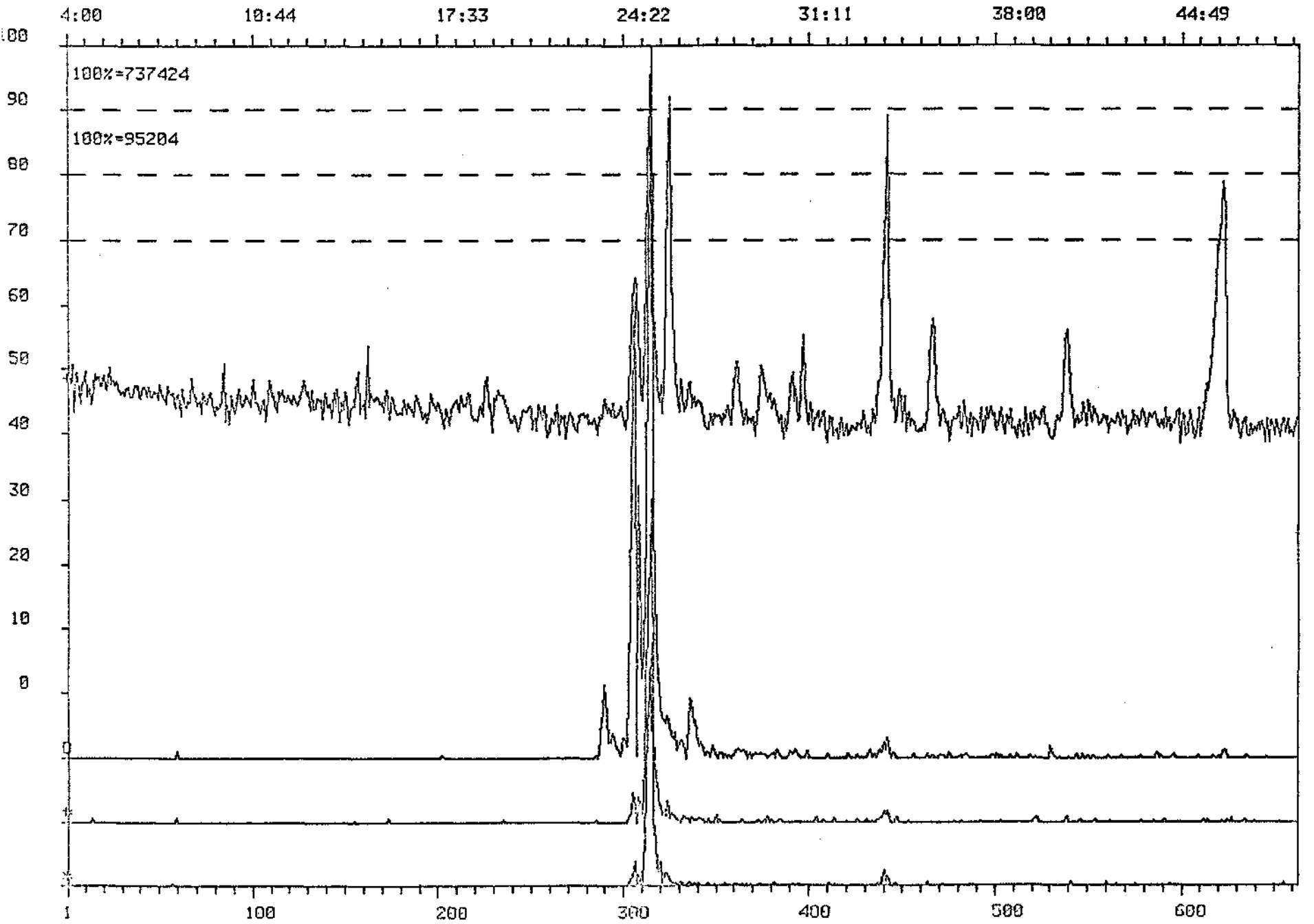
OCTACHLORO- 444(100.0), 442(87.9)
SCANS: 610-630 (m/z 455-442) REFINED SCANS:

* None of these compounds were included in the standard mixture

Fig. 3 - Exact mass chromatograms of ions due to native TCDF and $^{37}Cl_4$-TCDF. The data system plots only those ions acquired which are within 200 ppm of the theoretical mass of TCDF and labelled TCDF ions. The mass values appear in upper left-hand corner in nominal form

)S-55 CROSS SCAN REPORT, RUN: TD1S1

× 304 ⌖ 306 O 312 + TIC



085

Fig. 4- Mass spectrum of scan 306 of data set displayed in Fig. 3

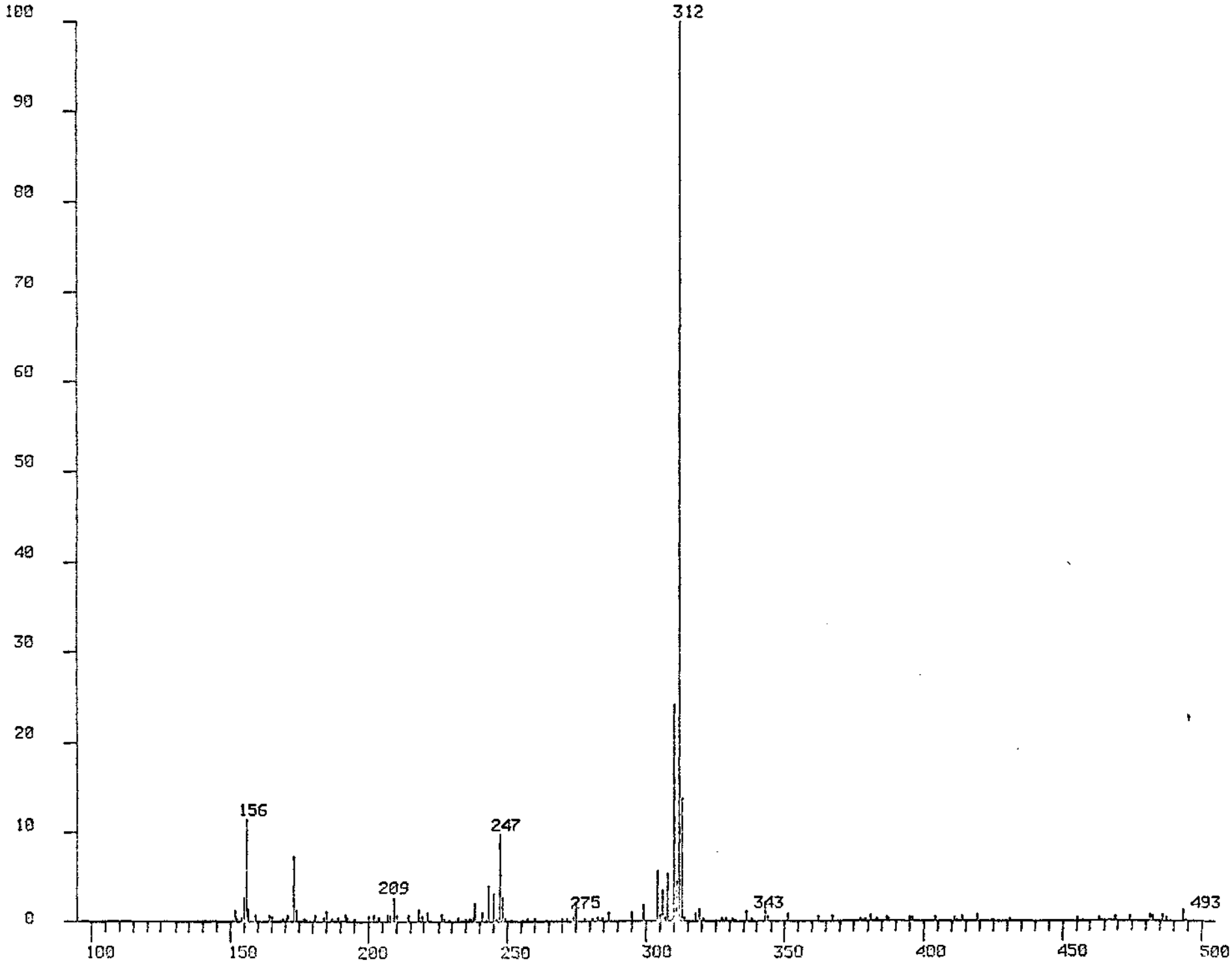


Fig.5- Mass spectrum of scan 314 of data set shown in Fig 3.

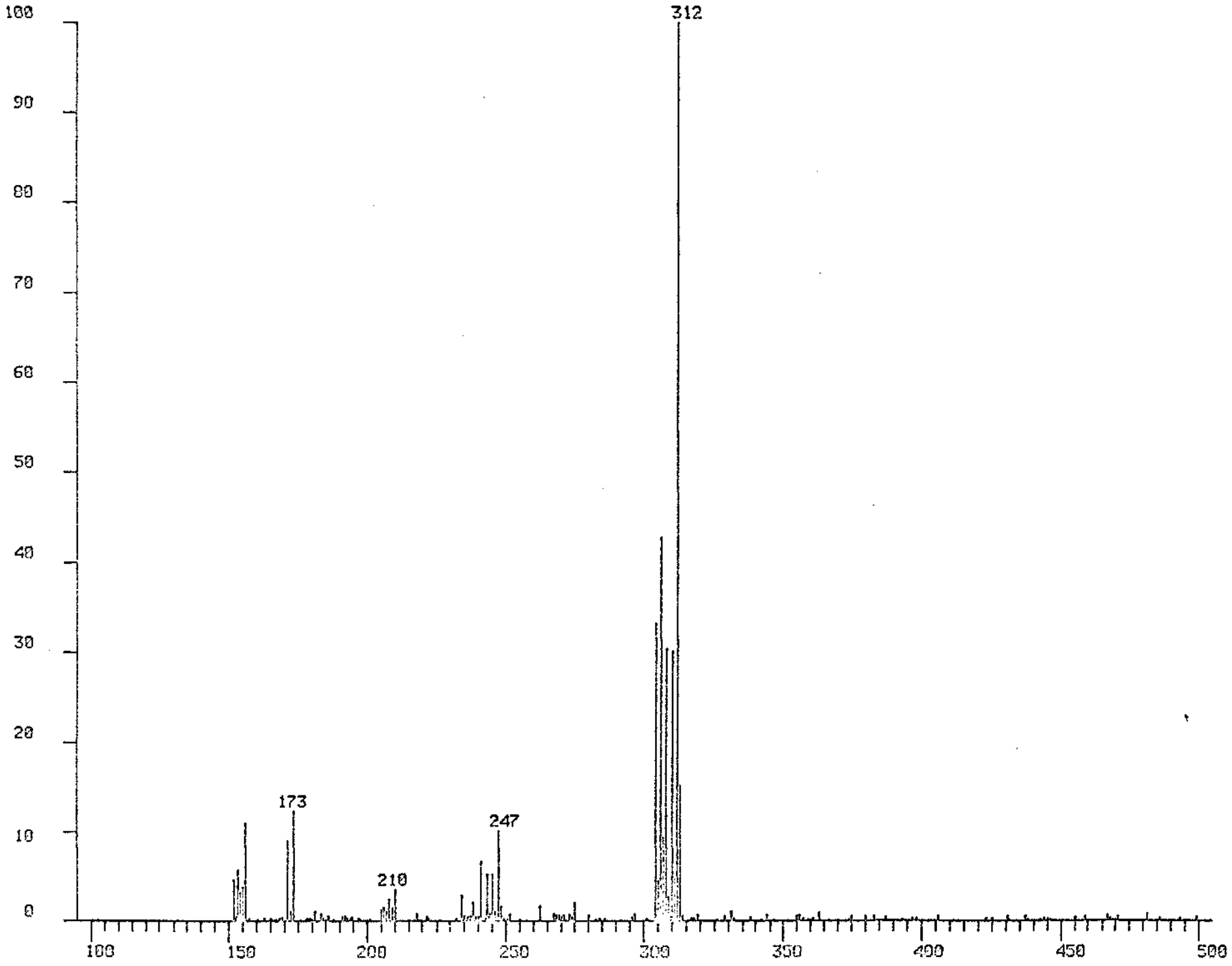


Fig: Exact mass chromatogram of HexaCDF.

DS-55 CROSS SCAN REPORT, RUN: TD1S1

* 338 # 340 O 374 & 376 $ 408 % 410 + TIC

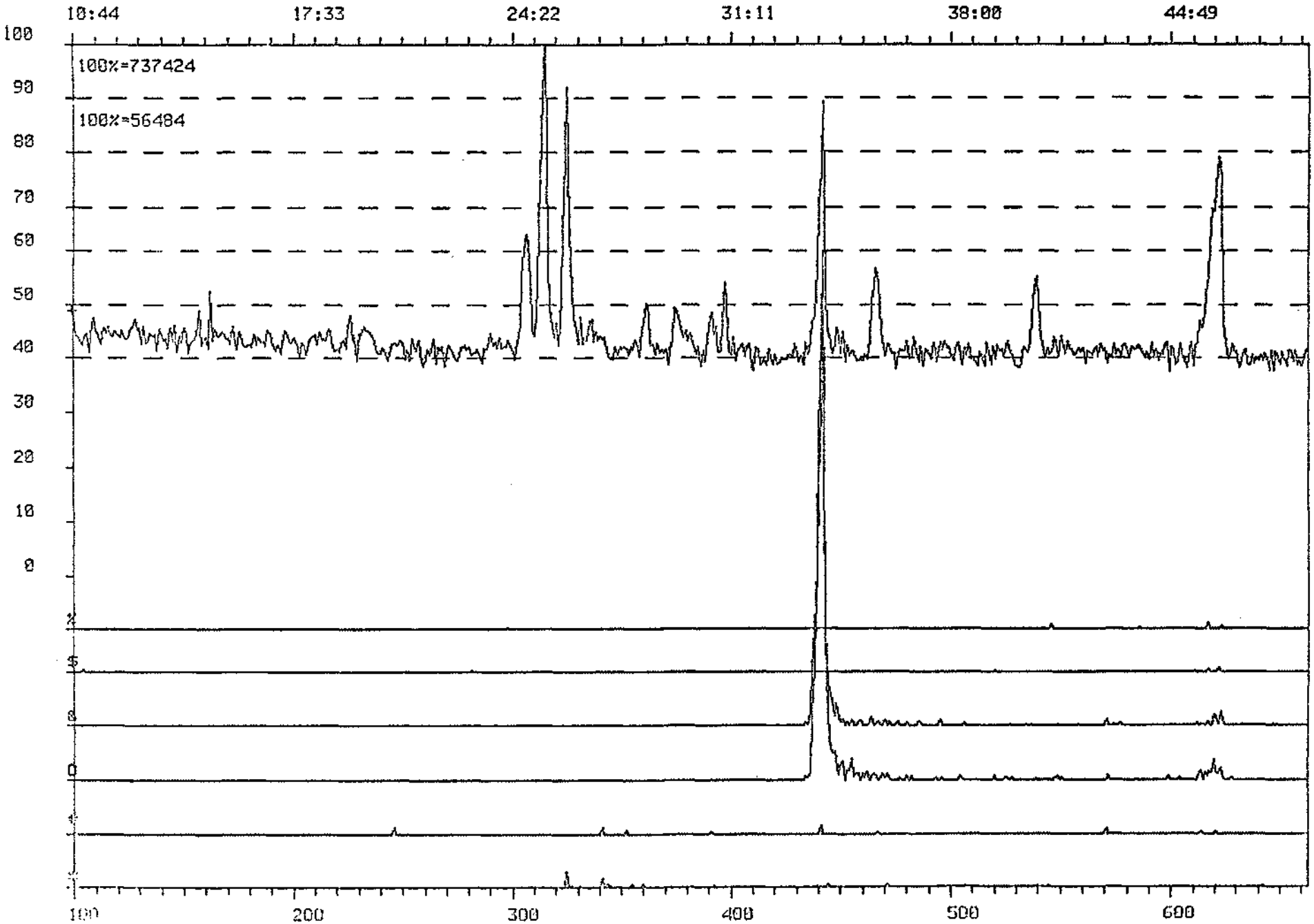


Fig.7 - Mass Spectrum of Scan 442 of data set TDIS1, a typical standard

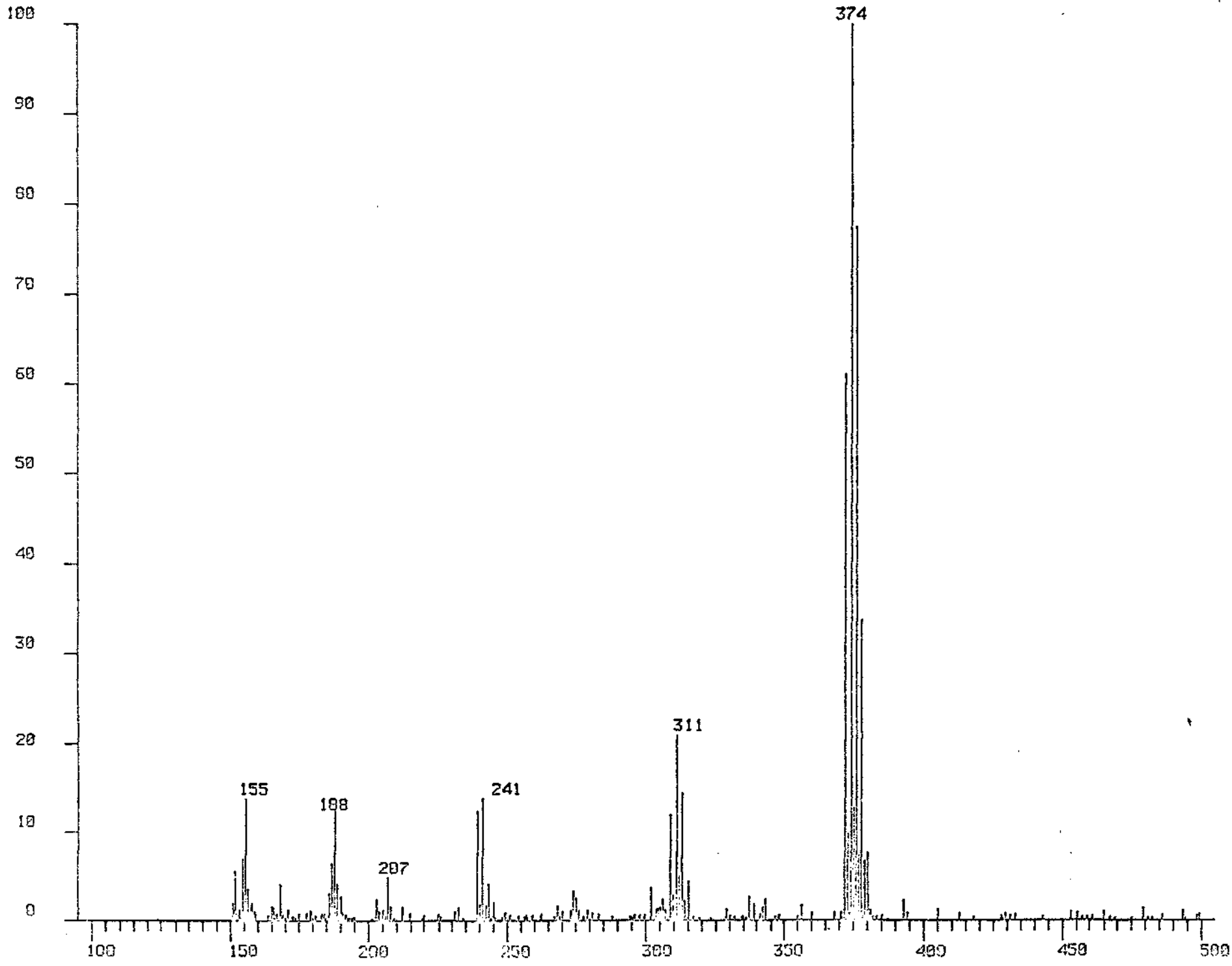


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Fig 8 - Extracted mass chromatogram of ~~isomer~~ to octachlorofuran

DS-55 CROSS SCAN REPORT, RUN: TD1S1

* 444 # 442 + TIC

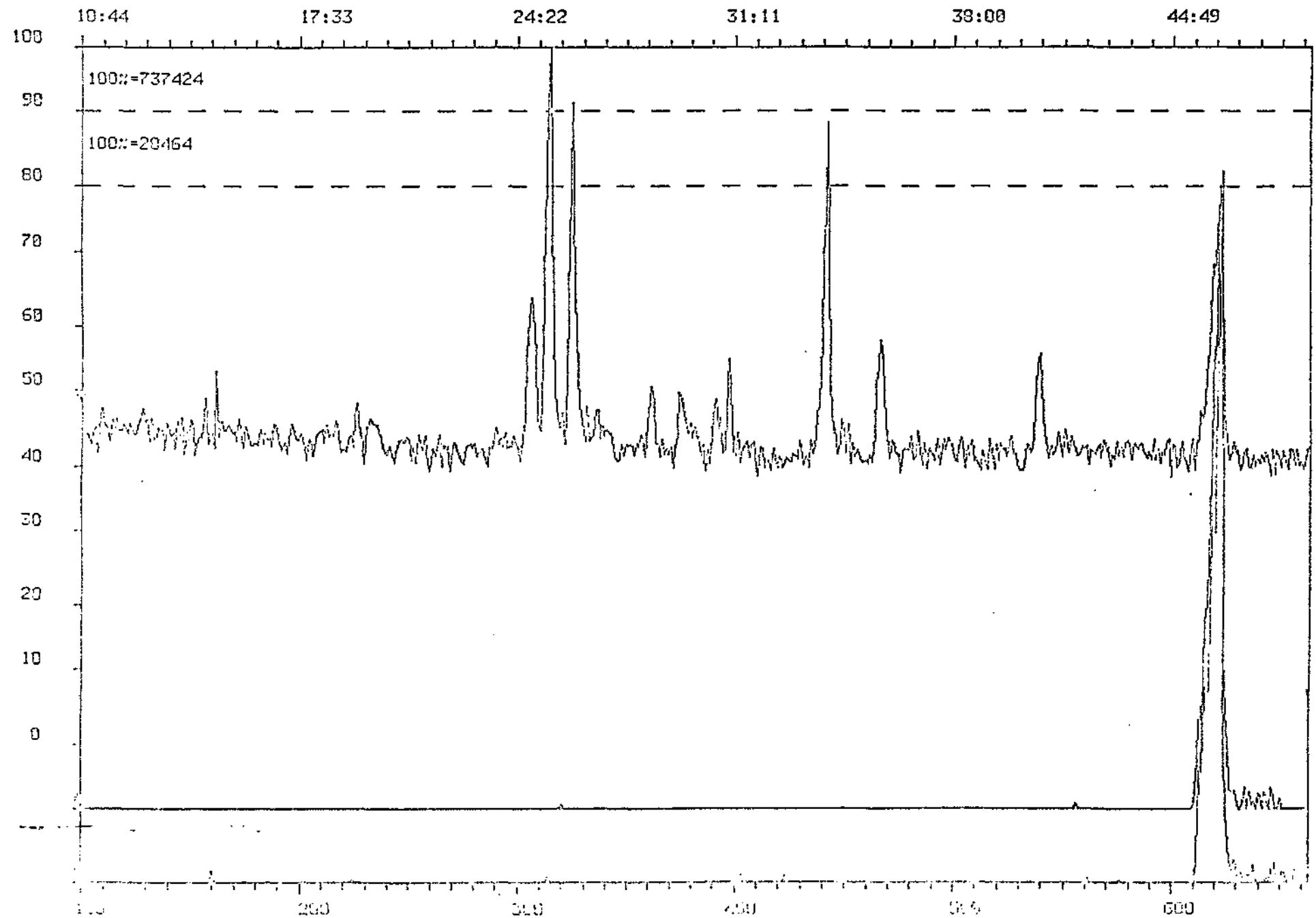

Fig. 9 - Mass Spectrum of scan 622, OCDD. OCDF and $^{37}Cl_6$-OCDD.

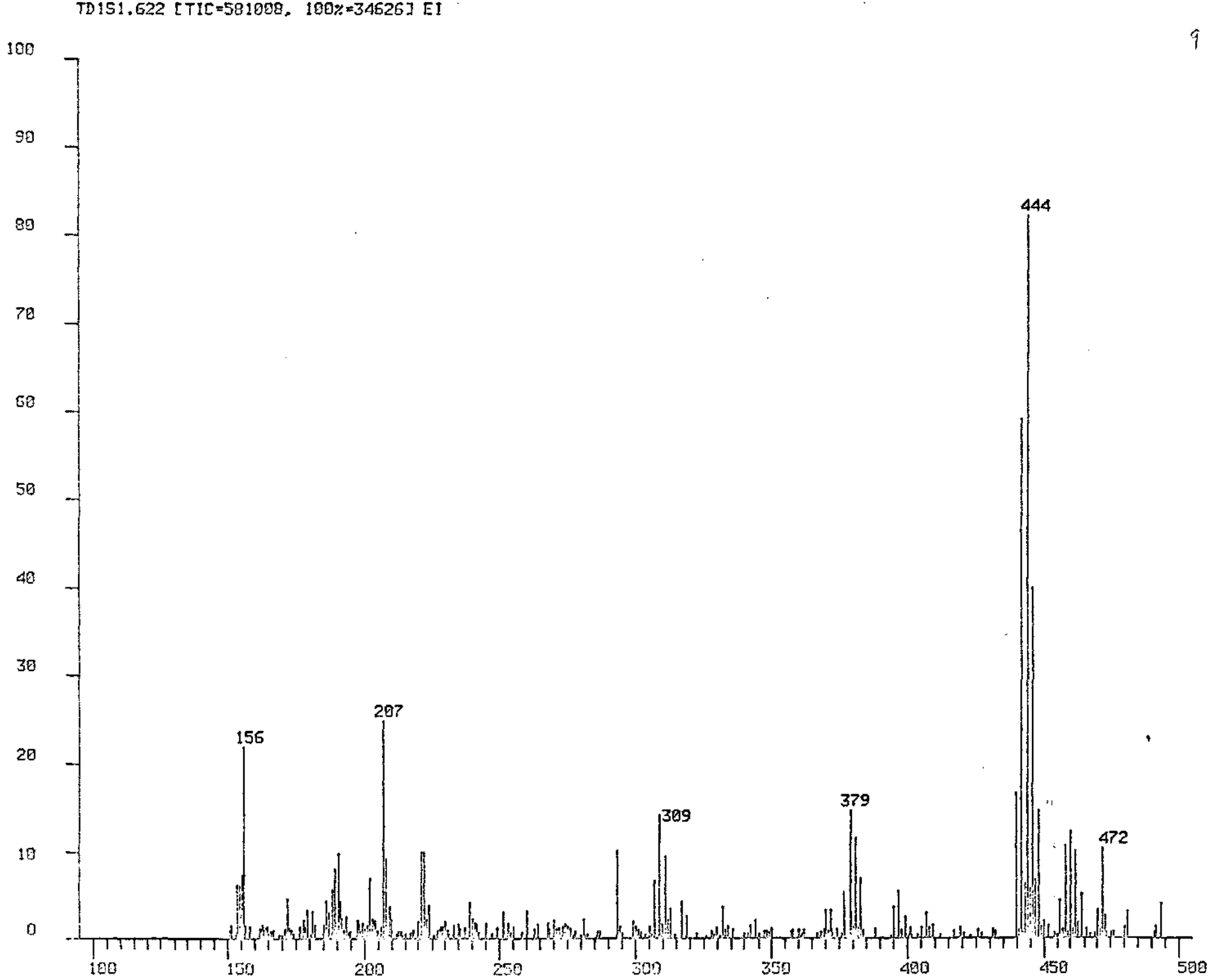

Fig 10 - Worksheet for PCDD's.

TABLE OF POLYCHLORODIBENZO-P-DIOXIN MASSES, INTENSITIES AND SCAN NUMBERS (FROM XSCAN DATA)

TRICHLORO-283(100.0),285(97.5)
SCANS: * REFINED SCANS:

DICHLORO-252(100.0),254(65.0)
SCANS: * REFINED SCANS:

MONOCHLORO-218(100.0),220(32.5)
SCANS: * REFINED SCANS:

TETRACHLORO-320(76.9),322(100.0),334(INT.STD.)
SCANS: REFINED SCANS:
(see data output for TCDF)

PENTACHLORO-356(100.0),358(65.0)
SCANS: * REFINED SCANS:

HEXACHLORO-390(100.0),392(81.2)
SCANS: 460-475 (m/z 387-400) REFINED SCANS:

HEPTACHLORO-424(100.0),426(97.5)
SCANS: 535-550 (m/z 415-437) REFINED SCANS:

OCTACHLORO-458(87.9),460(100.0),462(65.0),472(INT.STD.)
SCANS: REFINED SCANS:
(see data output for OCDF)

* None of these compounds were included in the standard mixture

Fig. 11- Exact Mass chromatogram of TCDD in the Standard Mixture

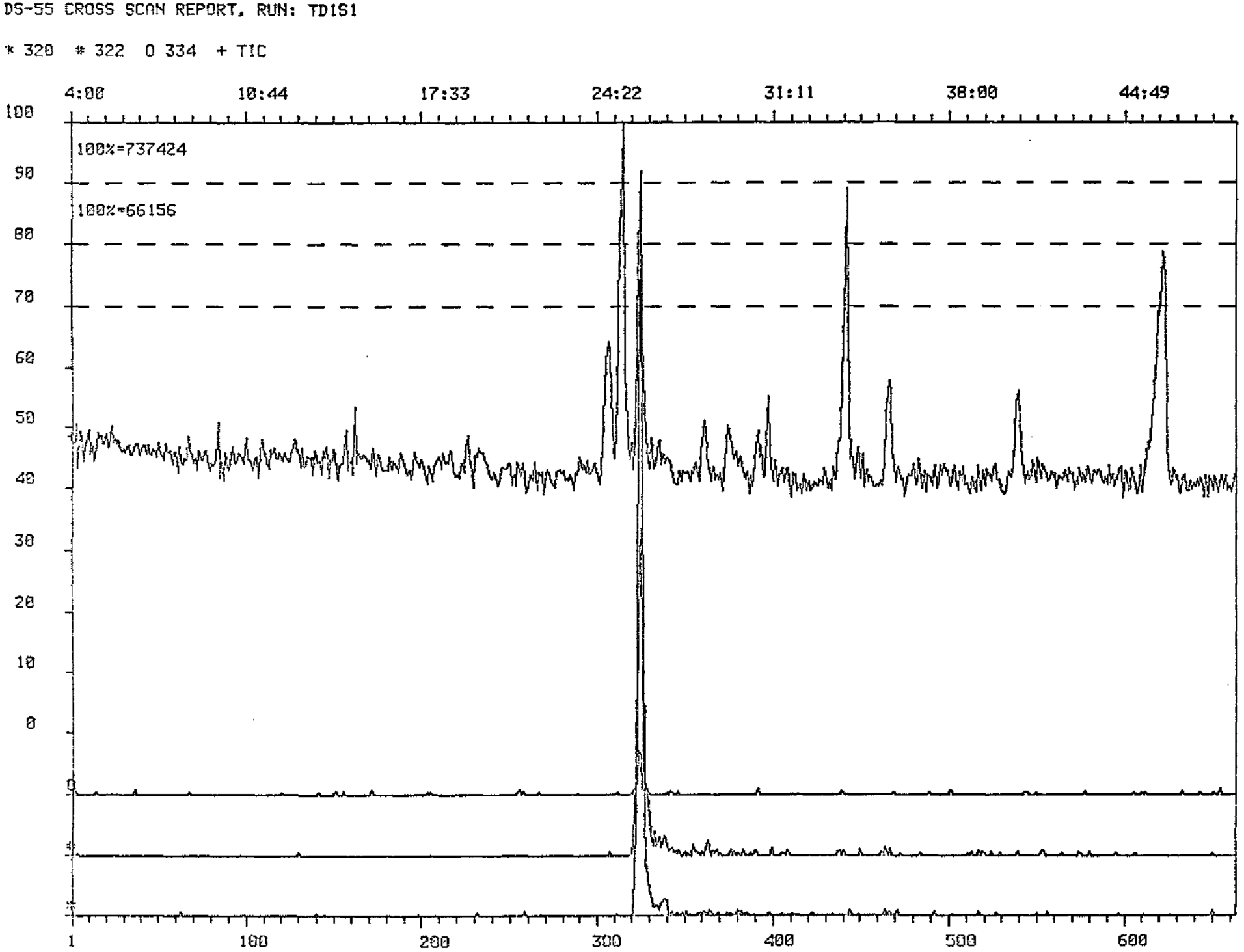

Fig. 12 - Mass Spectrum from chromatographic peak due to TCDD in the Standard Mixture.

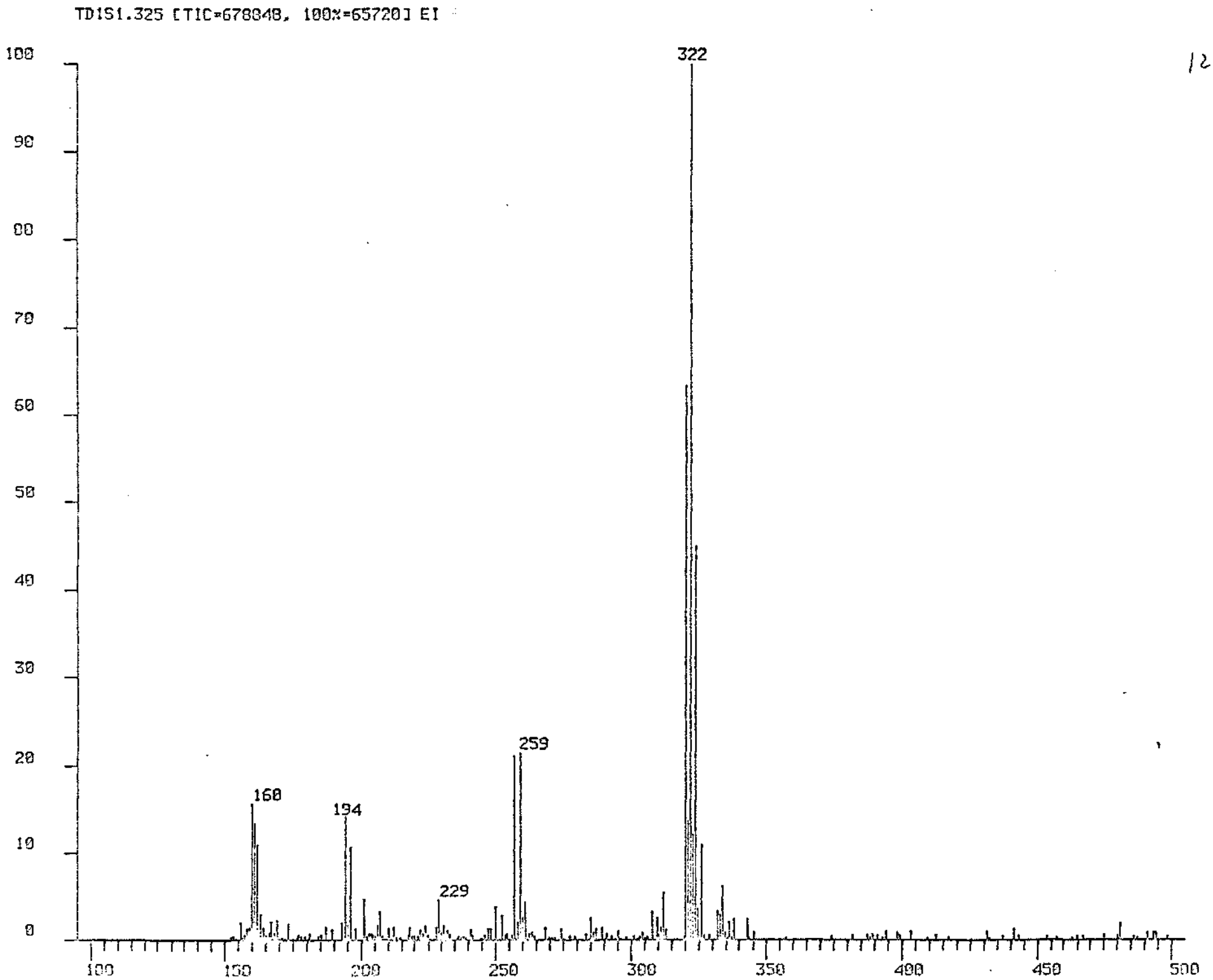

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Fig. 13 - Exact mass chromatograms of Hex_6CDD and Hp_7CDD in Standard Mixture

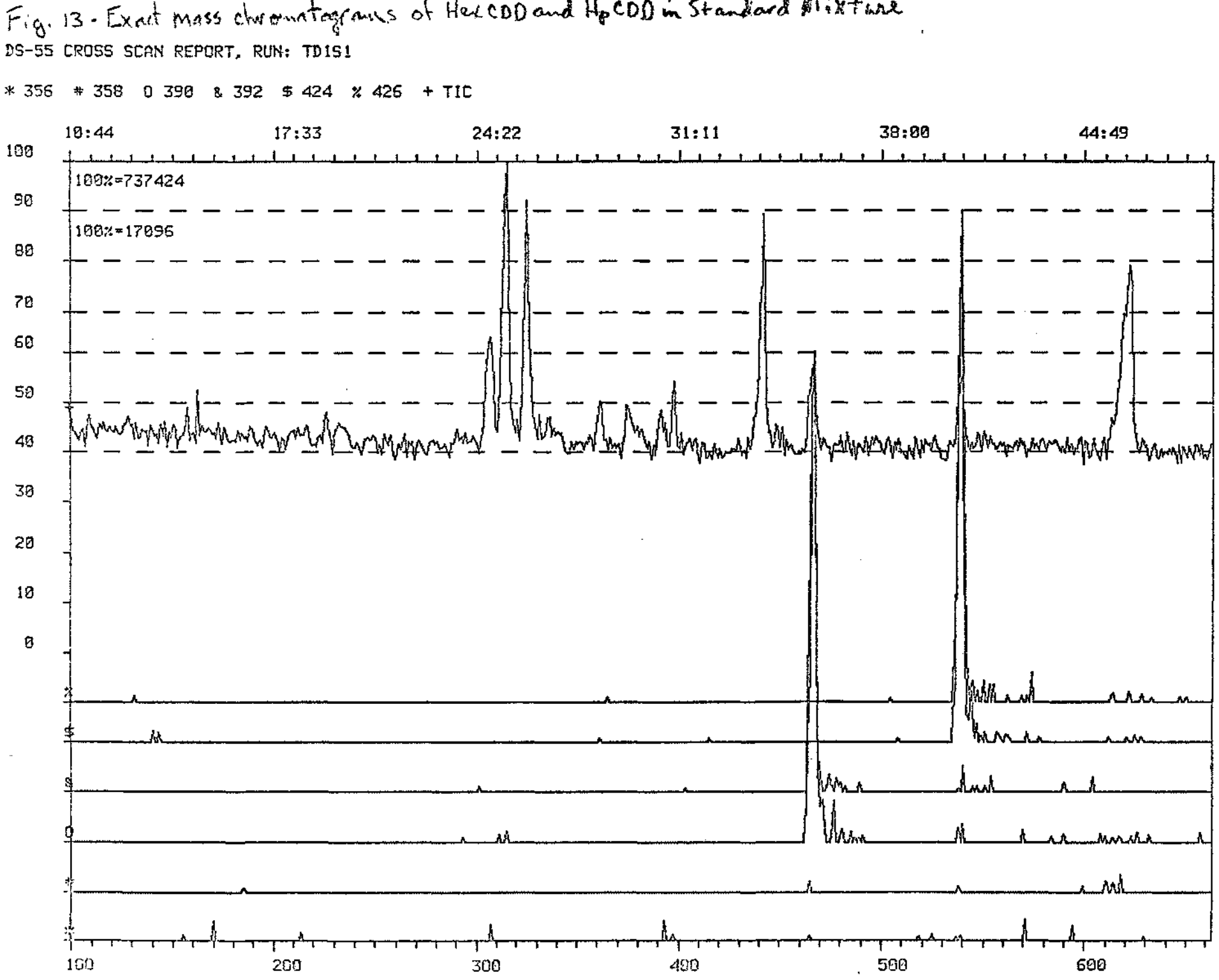


Fig. 14 - Mass Spectrum from chromatographic peak due to HexCOD.

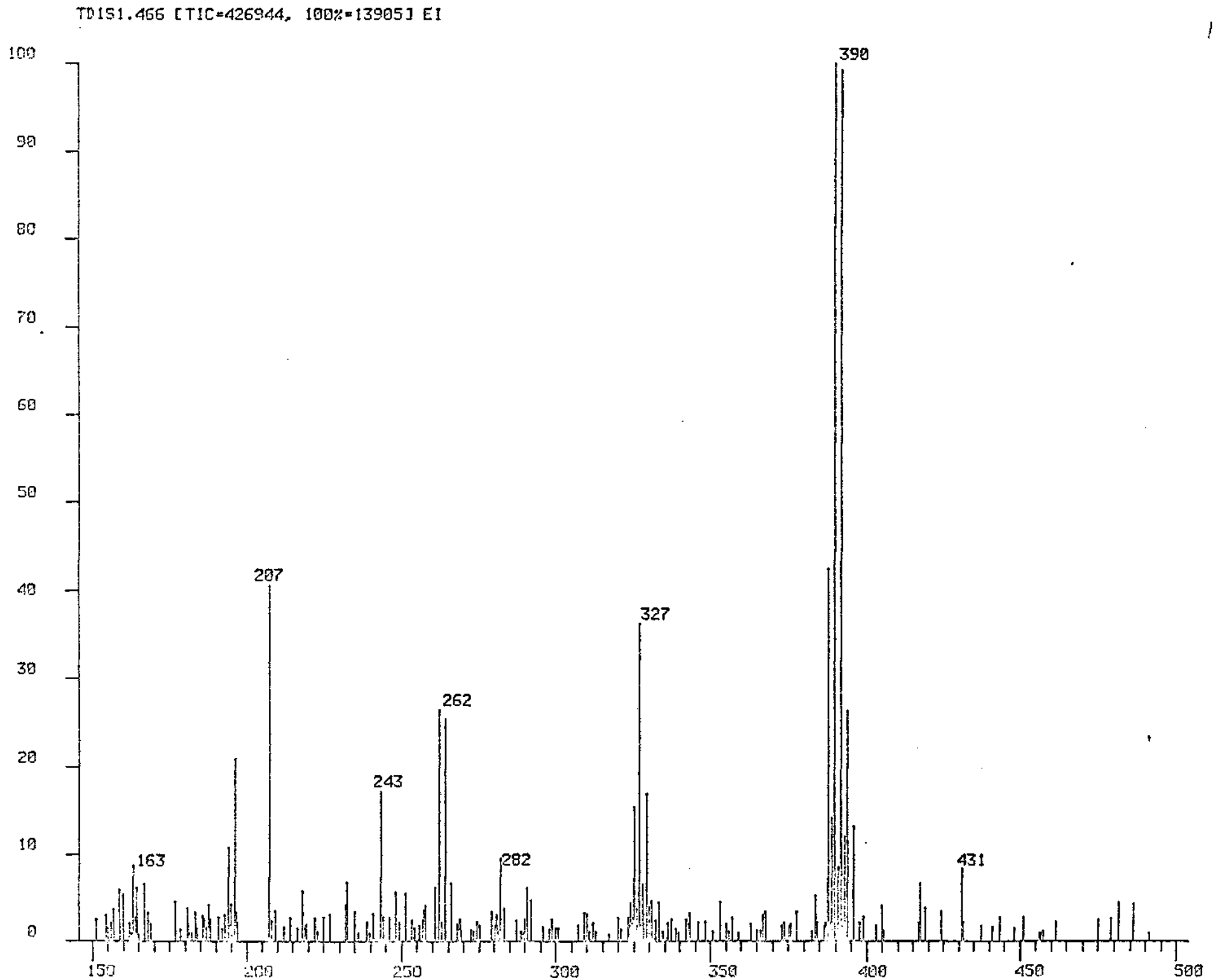


Fig. 15. Mass spectrum from chromatographic peak due to H_pCDD.

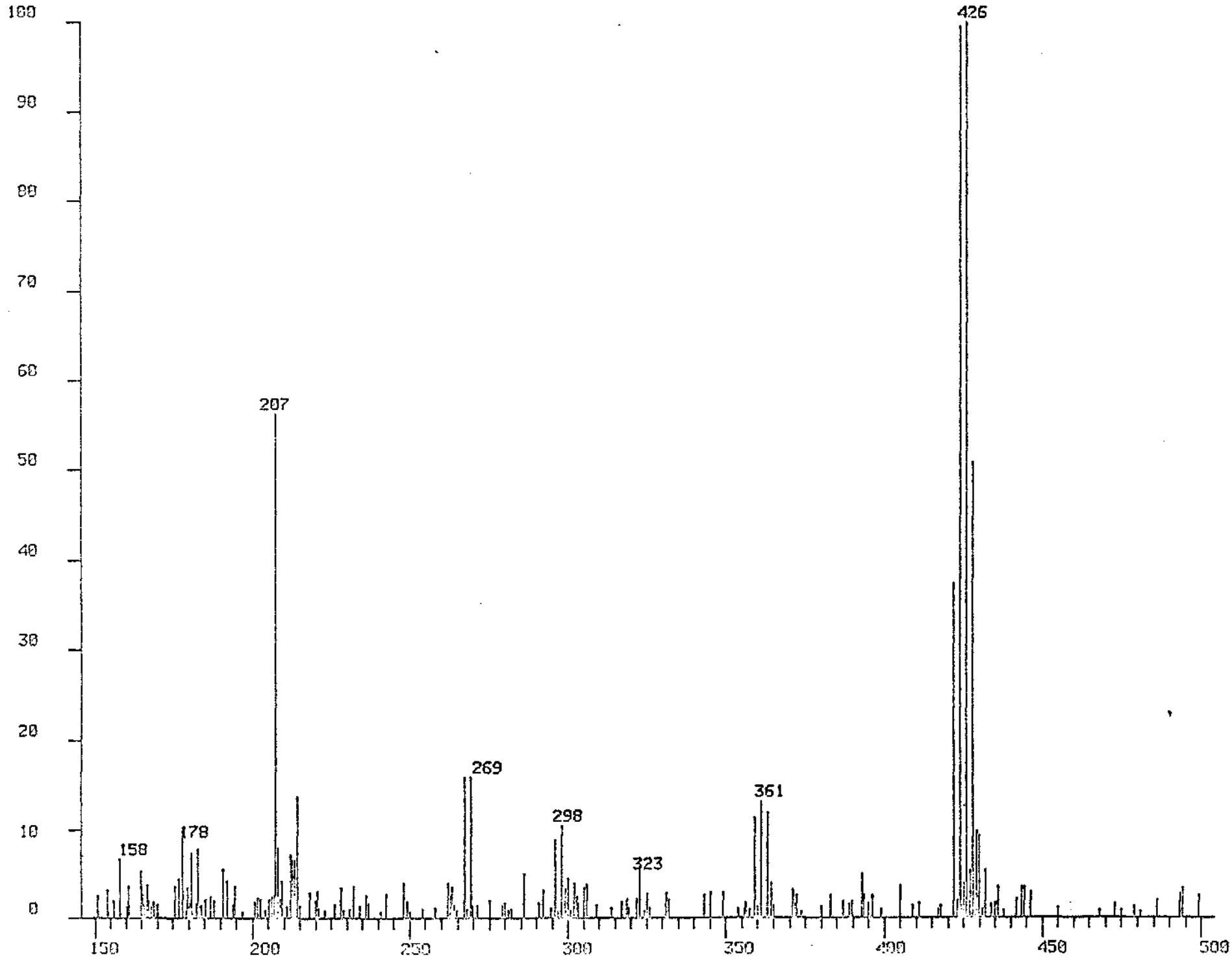


Fig. 1. Exact mass chromatograms of ions of OCDD and $^{37}Cl_8$-OCDD.

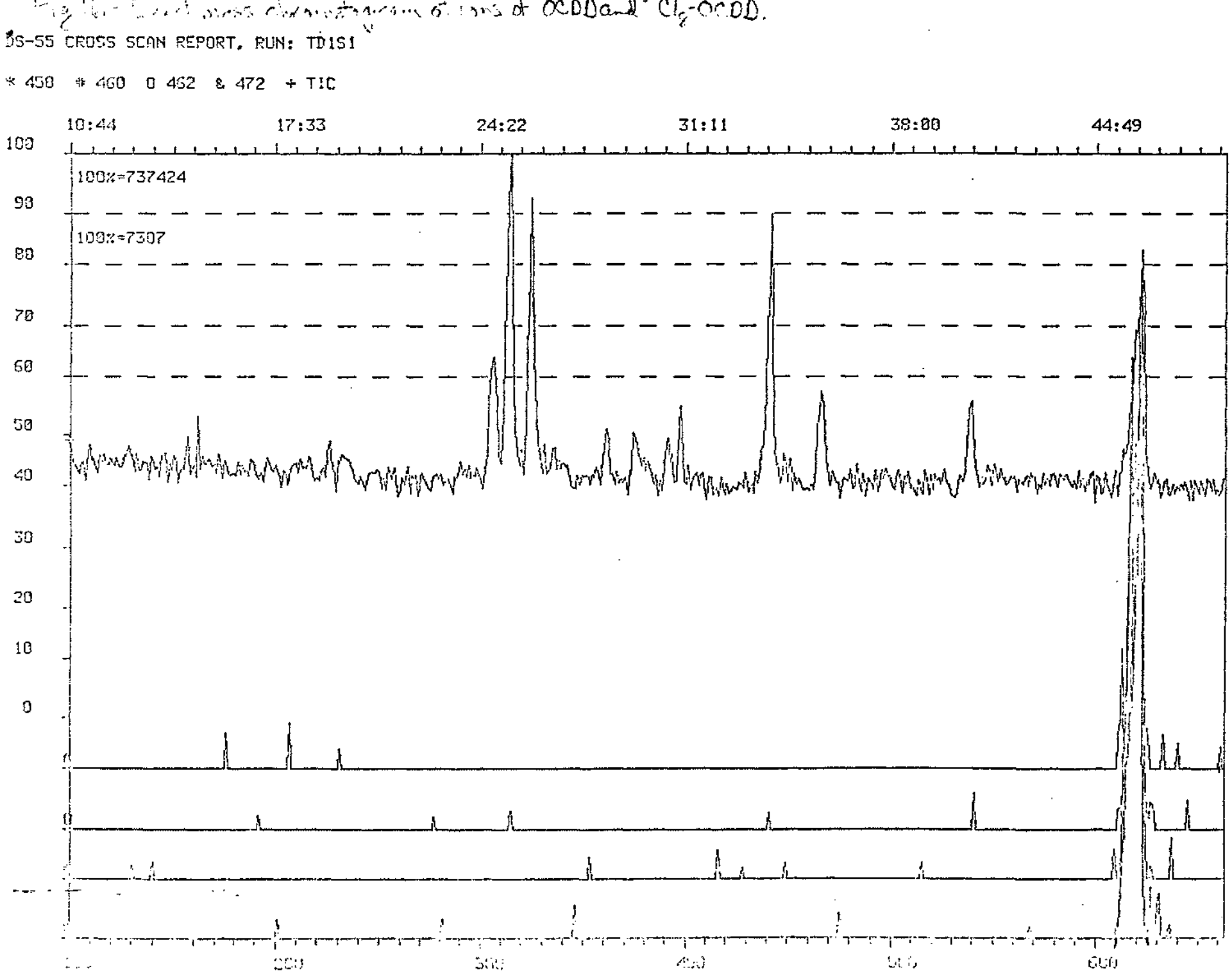
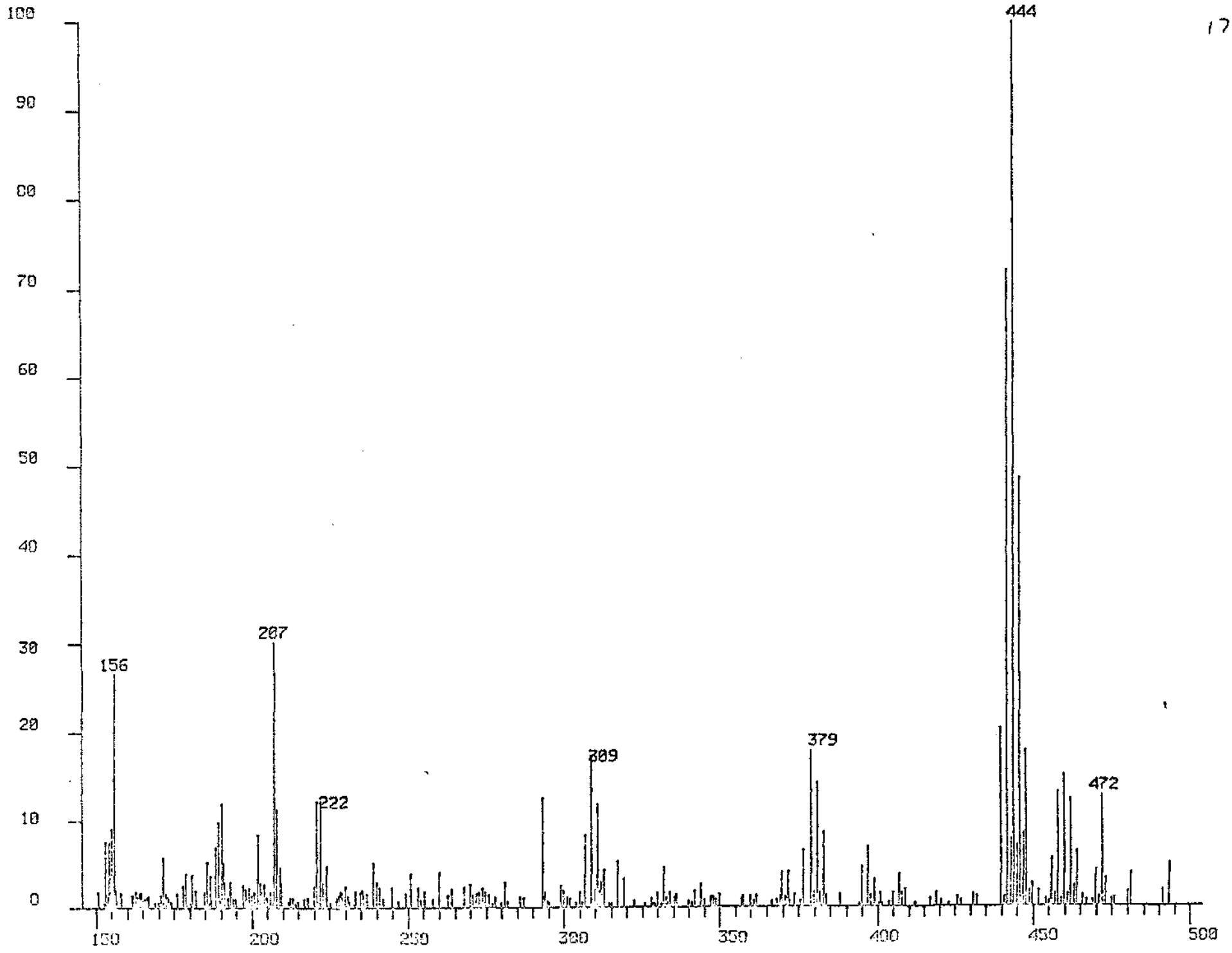


Fig 17 - Mass Spectrum from exact mass chromatographic peak of OCDD and $^{37}Cl_8$-OCDD



17

Figs 18-27 - Lists of exact masses and intensities of mass scans acquired during the elution of HexCDF. The range of exact masses shown is over molecular ion region only. Intensity ratios of ions in this region were used for verification of the presence of the compound. The most intense ion in the molecular cluster was used for quantitation. The average exact mass of the ions of the compound (averaged over the chromatographic peak) is an additional verification of the presence of the compound as well as an indication of the possible presence of other compounds with similar mass ions under the same chromatographic peak. Similar lists covering the appropriate mass range were generated for all compounds of interest in the data collected on the samples and standards.

DPB:TD151.MS
SCAN: 437, 3/11/82 14:51

IONISATION: EI
NO. PEAKS: 300/ 11
BASE/NREF INT: 30699./ 6315.
TIC: 350512./ 30193.
MASS RANGE: 149.9904 - 654.9601
RETN TIME/MISC: 33:43/ 53/ 0/ 50

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	31376.	6	460.	1.2	-	0.1
2	33588.	6	310.	0.8	-	0.0
3	33888.	5	292.	0.8	-	0.0
4	147089.	6	344.	0.9	-	0.1
5	183809.	5	243.	0.6	-	0.0
6	194559.	5	255.	0.7	-	0.0
101	302.0065	8	713.	1.8	11.3	0.2
102	301.9692	8	543.	1.4	-	0.2
103	301.9130	8	365.	0.9	5.8	0.1
104	300.9761	17	2558.	6.6	-	0.7
105	300.9102	6	376.	1.0	6.0	0.1
106	379.8908	5	205.	0.5	3.2	0.1
107	378.9742	8	745.	1.9	-	0.2
108	377.8208	21	1617.	4.2	25.6	0.5
109	376.9874	5	278.	0.7	4.4	0.0
110	376.8486	4	142.	0.4	2.2	0.0
111	376.8304	4	154.	0.4	2.4	0.0
112	375.8275	25	6101.	15.8	96.6	1.7!
113	375.8317	0	729.	1.9	11.5	0.2
114	374.8165	4	151.	0.4	2.4	0.0
115	373.9791	5	228.	0.6	3.6	0.0
116	373.8452	8	1909.	5.1	31.5	0.6*!
117	373.8194	12	4043.	12.8	78.3	1.4*
118	371.8334	21	6315.	16.3	100.0	1.8!

- Fig. 19

DP0:TD1S1.MS
SCAN: 430, 3/11/02 14:51

IONISATION: EI
NO. PEAKS: 301/ 7
BASE/NREF INT: 40098./ 12060.
TIC: 358656./ 13809.
MASS RANGE: 149.9904 - 654.9601
RETN TIME/MISC: 33:47/ 69/ 4/ 47

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	32446.	5	226.	0.6	-	0.0*
2	33041.	4	150.	0.4	-	0.0
3	34067.	4	148.	0.4	-	0.0
4	76687.	4	151.	0.4	-	0.0
5	102810.	5	274.	0.7	-	0.0
6	105698.	5	243.	0.6	-	0.0
7	195216.	4	171.	0.4	-	0.0
8	195809.	8	472.	1.2	-	0.1
101	381.9746	6	379.	0.9	-	0.1
102	381.7098	4	149.	0.4	1.2	0.0
103	381.0170	6	202.	0.7	2.3	0.0
104	380.9761	17	4050.✓	10.1	-	1.1
105	380.9263	6	365.	0.9	3.0	0.1*
106	379.8357	6	442.	1.1	3.7	0.1
107	378.8160	6	228.	0.6	1.9	0.0
108	377.8127	17	2898.	7.2	24.0	0.8
109	377.6045	5	231.	0.6	1.9	0.0
110	376.8139	12	1904.	4.7	15.8	0.5!
111	375.8533	6	228.	0.6	1.9	0.0
112	375.8025	21	10424.	26.0	86.4	2.9
113	375.7232	5	206.	0.5	1.7	0.1
114	374.9751	5	289.	0.7	-	0.0
115	374.8892	10	1489.	3.7	12.3	0.4
116	373.9669	8	670.	1.7	5.6	0.2
117	373.8108	25	12068.	30.1	100.0	3.4
118	372.8093	14	1603.	4.0	13.3	0.4!
119	371.8095	21	4568.	11.4	37.9	1.3

Fig. 20

DP0:TD1S1.MS
SCAN: 439, 3/11/82 14:51

IONISATION: EI
NO. PEAKS: 323/ 6
BASE/NREF INT: 41123./ 24258.
TIC: 426064./ 8360.
MASS RANGE: 149.9904 - 642.9601
RETN TIME/MISC: 33:51/ 81/ 3/ 50

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	30824.	6	432.	1.1	-	0.1#
2	31417.	5	240.	0.6	-	0.1
3	33002.	6	412.	1.0	-	0.1
4	185920.	5	195.	0.5	-	0.0
5	192959.	8	598.	1.5	-	0.1
6	193934.	4	163.	0.4	-	0.0
7	194529.	17	791.	1.9	-	0.2
8	196917.	4	185.	0.4	-	0.0
94	383.5946	4	164.	0.4	0.7	0.0
95	381.9915	5	219.	0.5	-	0.1
96	380.9761	21	5635.	13.7	-	1.3
97	379.8131	14	1161.	2.8	4.8	0.3
98	379.0176	6	414.	1.0	1.7	0.1
99	378.9858	8	783.	1.9	-	0.2
100	378.8289	10	858.	2.1	3.5	0.2
101	377.8439	10	1804.	4.4	7.4	0.4*!
102	377.8070	17	7120.	17.3	29.4	1.7*
103	376.8476	8	574.	1.4	2.4	0.1
104	376.8182	10	1345.	3.3	5.5	0.3
105	375.8139	25	16648.	40.5	68.6	3.9
106	374.8230	21	2347.	5.7	9.7	0.6
107	373.8167	25	24258.	59.0	100.0	5.7
108	372.8806	4	170.	0.4	0.7	0.0
109	372.8896	12	993.	2.4	4.1	0.2
110	371.8170	25	14002.	34.0	57.7	3.3

Fig 21

DP0:TD1S1.MS
SCAN: 440, 3/11/02 14:51

IONISATION: EI
NO. PEAKS: 354/ 18
BASE/NREF INT: 41399./ 36504.
TIC: 513632./ 47000.
MASS RANGE: 149.9904 - 654.9601
RETN TIME/MISC: 33:55/ 71/ 4/ 47

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	32345.	8	674.	1.6	-	0.1*
2	33642.	6	504.	1.2	-	0.1
3	34077.	5	210.	0.5	-	0.0
4	35791.	4	186.	0.4	-	0.0
5	120190.	5	203.	0.5	-	0.0
6	159326.	5	230.	0.6	-	0.0
7	185341.	5	215.	0.5	-	0.0
8	185585.	4	146.	0.4	-	0.0
111	383.5280	5	204.	0.5	0.6	0.0
112	381.9626	8	1078.	2.6	3.0	0.2!
113	381.0270	5	204.	0.5	0.6	0.0
114	380.9761	25	3092.	7.5	-	0.6
115	380.7949	8	511.	1.2	1.4	0.1
116	380.2530	5	261.	0.6	0.7	0.1
117	379.8304	10	1147.	2.8	3.1	0.2*
118	379.7974	12	1167.	2.8	3.2	0.2*
119	378.0180	17	1658.	4.0	4.5	0.3
120	377.0097	35	14440.	34.9	39.6	2.8
121	376.0511	10	730.	1.8	2.0	0.1*
122	376.0105	17	3853.	9.3	10.6	0.8*!
123	376.0178	4	157.	0.4	0.4	0.0
124	375.0159	29	26838.	64.8	73.5	5.2
125	375.6740	8	358.	0.9	1.0	0.0
126	374.0268	25	7277.	17.6	19.9	1.4!
127	373.0171	35	36504.	88.2	100.0	7.1
128	373.7535	6	327.	0.8	0.9	0.0
129	372.0286	17	3205.	7.7	8.8	0.6!
130	371.0229	25	19186.	46.2	52.3	3.7

Fig. 22.

DP0:TD1S1.MS
SCAN: 441, 3/11/02 14:51

IONISATION: EI
NO. PEAKS: 362/ 10
BASE/NREF INT: 52611./ 46696.
TIC: 561264./ 25996.
MASS RANGE: 140.9904 - 654.9601
RETN TIME/MISC: 33:59/ 81/ 2/ 41

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	13683.	6	394.	0.7	-	0.0
2	15030.	8	616.	1.2	-	0.1
3	30657.	5	234.	0.4	-	0.0
4	31128.	4	147.	0.3	-	0.0
5	32998.	8	404.	0.8	-	0.0
6	117616.	6	358.	0.7	-	0.0
7	194130.	10	509.	1.0	-	0.0
8	195695.	4	143.	0.3	-	0.0
104	383.8062	8	552.	1.0	1.2	0.1
105	382.9759	6	323.	0.6	0.7	0.1
106	381.9815	17	1169.	2.2	-	0.2
107	380.9761	17	3095.	5.9	-	0.6
108	380.8286	8	645.	1.2	1.4	0.1
109	379.8101	21	2519.	4.8	5.4	0.4
110	378.8106	17	2157.	4.1	4.6	0.4
111	377.8165	29	16821.	32.0	36.0	3.0
112	377.1945	4	183.	0.3	0.4	0.0
113	376.8190	14	2692.	5.1	5.8	0.5
114	375.8207	29	34202.	65.0	73.2	6.1
115	375.7563	4	158.	0.3	0.3	0.0
116	374.9701	8	471.	0.9	1.0	0.0
117	374.8225	21	3507.	6.7	7.5	0.6
118	374.2968	6	294.	0.6	0.6	0.1
119	373.9615	4	158.	0.3	0.3	0.0
120	373.8194	35	46696.	88.8	100.0	8.3
121	373.7541	6	441.	0.8	0.9	0.0
122	373.4754	4	180.	0.3	0.4	0.0
123	373.2477	6	375.	0.7	0.8	0.0
124	372.8484	17	2157.	4.1	4.6	0.4
125	371.9722	5	272.	0.5	0.6	0.0
126	371.8194	25	21210.	40.3	45.4	3.8
127	371.5324	5	243.	0.5	0.5	0.0

Fig. 23

DPO:TD1S1.MS
SCAN: 442, 3/11/82 14:51

IONISATION: EI
NO. PEAKS: 397/ 42
BASE/NREF INT: 56045./ 56045.
TIC: 656890./ 276128.
MASS RANGE: 149.9304 - 654.9281
RETN TIME/MISC: 34: 3/ 80/ 2/ 51

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	132977.	4	174.	0.3	-	0.0*
2	140512.	5	257.	0.5	-	0.0
3	183174.	6	455.	0.8	-	0.0
4	195757.	6	296.	0.5	-	0.0
5	196046.	4	207.	0.4	-	0.0
6	196572.	5	218.	0.4	-	0.0
7	196843.	6	334.	0.6	-	0.1
110	383.0175	5	286.	0.5	0.5	0.0
111	381.9847	12	1219.	2.2	-	0.2
112	381.8945	5	247.	0.4	0.4	0.0
113	381.7877	6	290.	0.5	0.5	0.0
114	380.9761	25	7321. ✓	13.1	-	1.1
115	380.8361	8	637.	1.1	1.1	0.1
116	380.8097	4	172.	0.3	0.3	0.0
117	380.5241	6	423.	0.8	0.8	0.0
118	379.8458	8	989.	1.6	1.6	0.1*
119	379.8074	17	4237.	7.6	7.6	0.6*!
120	378.9777	10	473.	0.8	-	0.0
121	378.8209	21	3745.	6.7	6.7	0.6!
122	377.9242	5	263.	0.5	0.5	0.0
123	377.8139	25	18875.	33.7	33.7	2.9!
124	377.4177	4	158.	0.3	0.3	0.0
125	377.8211	6	298.	0.5	0.5	0.0
126	376.8448	10	1585.	2.8	2.8	0.2*
127	376.8128	12	4116.	7.3	7.3	0.6*!
128	376.6392	4	149.	0.3	0.3	0.0
129	375.8287	29	43409.	77.5	77.5	6.6!
130	375.7568	5	212.	0.4	0.4	0.0
131	374.9888	8	442.	0.8	-	0.0
132	374.8636	8	1596.	2.7	2.7	0.2*!
133	374.8249	17	7016.	12.5	12.5	1.1*!
134	374.5739	4	172.	0.3	0.3	0.0
135	373.8232	29	56045.	100.0	100.0	8.5!
136	373.7567	6	439.	0.8	0.8	0.0
137	373.3094	6	406.	0.7	0.7	0.1
138	372.8259	17	5474.	9.8	9.8	0.8!
139	371.8393	35	34259.	61.1	61.1	5.2!
140	370.9956	4	153.	0.3	0.3	0.0

Fig 24

DPO:TD1S1.MS
SCAN: 443. 3/11/02 14:51

IONISATION: EI
NO. PEAKS: 314/ 7
BASE/NREF INT: 41005./ 14530.
TIC: 379200./ 9496.
MASS RANGE: 149.9904 - 653.0029
RETN TIME/MISC: 34: 7/ 83/ 0/ 41

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	40654.	6	401.	1.0	-	0.1
2	120081.	4	200.	0.5	-	0.1
3	175586.	5	235.	0.6	-	0.1
4	194751.	5	347.	0.8	-	0.0
5	195163.	5	233.	0.6	-	0.1
6	195454.	5	282.	0.7	-	0.0
98	382.0541	6	396.	0.9	2.7	0.1
99	381.9696	8	439.	1.0	-	0.1
100	381.8062	6	341.	0.8	2.3	0.0
101	381.6939	8	575.	1.4	4.0	0.2
102	380.9761	17	3912.	9.4	-	1.0
103	379.8144	6	365.	0.9	2.5	0.1
104	379.5494	4	170.	0.4	1.2	0.0
105	378.9853	4	187.	0.4	-	0.0
106	378.8759	8	518.	1.2	3.6	0.1
107	378.8295	6	260.	0.6	1.8	0.0
108	377.8883	29	7077.	16.9	48.7	1.9
109	376.8900	12	1745.	4.2	12.0	0.5
110	375.8207	29	10579.	25.3	72.8	2.8
111	374.9054	5	200.	0.5	-	0.1
112	374.8201	14	1679.	4.0	11.6	0.4
113	373.8726	6	443.	1.1	3.0	0.1
114	373.8137	25	14530.	34.8	100.0	3.8
115	373.7593	4	204.	0.5	1.4	0.1
116	372.8550	6	317.	0.8	2.2	0.0
117	372.8148	14	874.	2.1	6.0	0.2
118	372.8166	6	381.	0.9	2.6	0.1
119	371.8790	5	233.	0.6	1.6	0.1
120	371.8158	21	7901.	18.9	54.4	2.1

Fig. 25-

DP0:TD1S1.MS
SCAN: 444. 3/11/82 14:51

IONISATION: EI
NO. PEAKS: 278/ 2
BASE/NREF INT: 40571./ 7635.
TIC: 330112./ 2555.
MASS RANGE: 149.9904 - 654.8452
RETN TIME/MISC: 34:11/ 65/ 1/ 56

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	13514.	6	437.	1.1	-	0.1*
2	31740.	5	276.	0.7	-	0.0
3	34225.	6	300.	0.7	-	0.0
4	92946.	6	314.	0.8	-	0.1
5	194961.	10	646.	1.6	-	0.2
6	195553.	5	258.	0.6	-	0.0
90	384.4293	5	256.	0.6	3.4	0.0
91	383.8317	5	213.	0.5	2.8	0.0
92	382.9715	5	258.	0.6	3.4	0.0
93	381.0250	12	715.	1.8	9.4	0.2
94	380.9761	17	5009.	12.5	-	1.5
95	379.8166	6	314.	0.8	4.1	0.1
96	378.9773	5	272.	0.7	-	0.0
97	378.8634	5	268.	0.7	3.5	0.0
98	377.8338	25	2387.	5.9	31.3	0.7
99	377.3150	4	191.	0.5	2.5	0.1
100	376.8212	5	305.	0.8	4.0	0.0
101	375.8262	25	4512.	11.1	59.1	1.4
102	374.9922	4	140.	0.3	-	0.0
103	374.8219	8	326.	0.8	4.3	0.1
104	373.9682	8	627.	1.5	8.2	0.2
105	373.8244	21	7635.	18.8	100.0	2.3
106	372.8490	0	363.	0.9	4.8	0.1
107	371.9204	5	277.	0.7	3.6	0.0
108	371.8335	17	3360.	8.3	44.0	1.0
109	371.6684	4	174.	0.4	2.3	0.1

Fig 2L

DP0:TD151.MS
SCAN: 445. 3/11/82 14:51

IONISATION: EI
NO. PEAKS: 273/ 6
BASE/NREF INT: 44198./ 3419.
TIC: 333664./ 10673.
MASS RANGE: 149.9904 - 654.9093
RETN TIME/MISC: 34:15/ 72/ 3/ 41

PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	14750.	8	462.	1.0	-	0.1*
2	31467.	6	397.	0.9	-	0.1
3	33745.	6	366.	0.8	-	0.1
4	93326.	4	151.	0.3	-	0.0
5	112296.	4	174.	0.4	-	0.1
6	182689.	8	464.	1.0	-	0.1
94	381.9603	4	194.	0.4	5.7	0.1
95	380.9761	14	3245.	7.3	-	1.0*
96	380.9513	8	1889.	4.3	55.3	0.6**!
97	380.8020	5	222.	0.5	6.5	0.0
98	378.9693	6	357.	0.8	-	0.1
99	377.7965	6	350.	0.8	10.2	0.1
100	376.8327	4	175.	0.4	5.1	0.1
101	375.8399	8	487.	1.1	14.2	0.1
102	375.8062	14	3419.	7.7	100.0	1.0!
103	374.8065	8	561.	1.3	16.4	0.2
104	373.8506	10	1000.	2.3	29.2	0.3
105	373.8129	14	3245.	7.3	94.9	1.0
106	372.8047	8	488.	0.9	11.9	0.1
107	371.9098	4	167.	0.4	4.9	0.1
108	371.8389	21	2344.	5.3	68.6	0.7
109	370.9711	5	204.	0.5	6.0	0.1

Fig 27

DPB:TDIS1.MS
SCAN: 445, 3/11/02 14:51

IONISATION: EI
NO. PEAKS: 259/ 8
BASE/NREF INT: 42934./ 3739.
TIC: 397584./ 14414.
MASS RANGE: 149.9904 - 654.9601
RETN TIME/MISC: 34:20/ 79/ 1/ 38

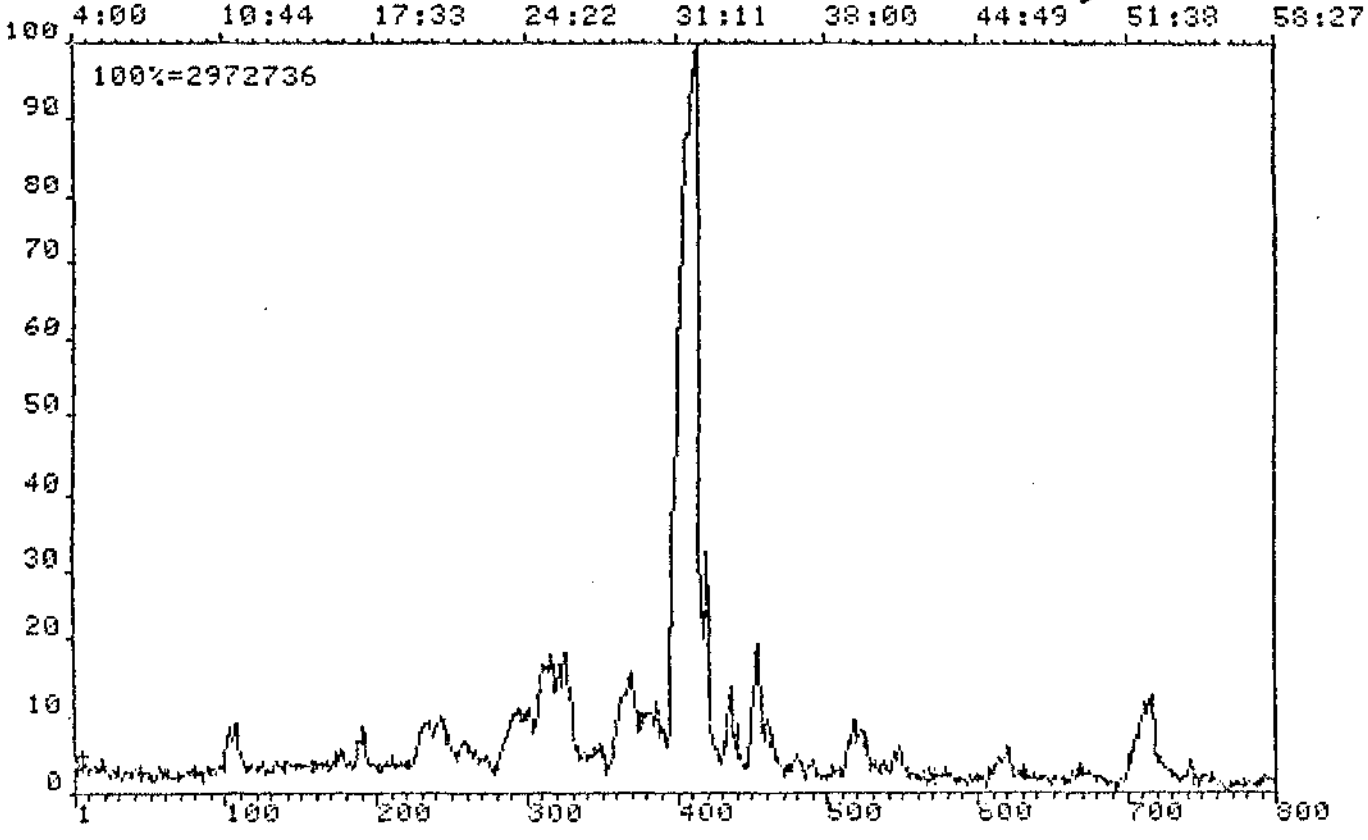
PEAK NO.	MEASURED MASS	NO. POINTS	ABSOLUTE INTENSITY	% INT. BASE	% INT. NREF	% TOT. ION
1	32271.	4	165.	0.4	-	0.1
2	33039.	6	504.	1.2	-	0.2
3	33022.	5	217.	0.5	-	0.0
4	120463.	6	209.	0.5	-	0.0
5	194800.	6	313.	0.7	-	0.1
6	195273.	6	352.	0.8	-	0.1
7	196530.	6	246.	0.6	-	0.0
90	383.7921	10	1392.	3.2	37.2	0.5
91	383.0029	4	195.	0.5	5.2	0.0
92	382.0130	6	491.	1.1	13.1	0.2
93	381.9813	10	451.	1.1	-	0.1
94	381.7953	4	160.	0.4	4.3	0.1
95	380.9761	25	3827.	7.1	-	1.0
96	379.9918	8	516.	1.2	13.8	0.2
97	379.8442	5	190.	0.4	5.1	0.1
98	379.8212	6	338.	0.8	9.0	0.1
99	378.9961	8	420.	1.0	11.2	0.1
100	377.9578	4	159.	0.4	4.3	0.1
101	377.8260	4	140.	0.3	3.7	0.0
102	375.8229	25	2678.	6.2	71.6	0.9
103	373.9838	5	219.	0.5	5.9	0.0
104	373.8683	10	580.	1.4	15.5	0.2
105	373.8196	17	2255.	5.3	60.3	0.7
106	373.5948	6	394.	0.9	10.5	0.1
107	372.8154	6	490.	1.1	13.1	0.2
108	371.8227	8	670.	1.6	17.9	0.2

Fig 28

3-5-82

DS-55 CROSS SCAN REPORT, RUN: FDS3
+ TIC

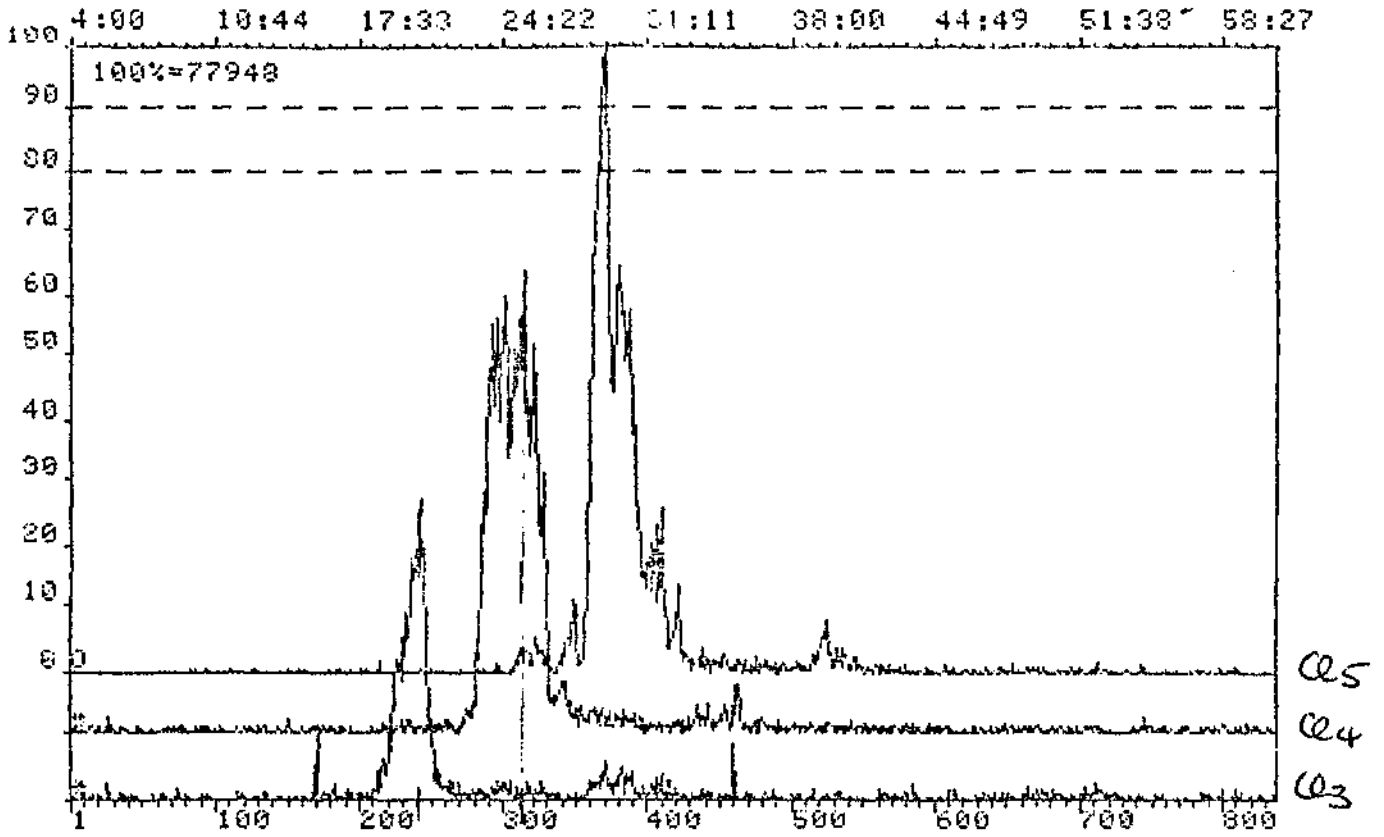
TIC chromatogram of 3.3 ml Bing sample # 706 (5th Floor) [30 ml]



R

Fig 29 - Exact Mass chromatograms of most intense ion of the tri - penta chloro furans.

DS-55 CROSS SCAN REPORT, RUN: FDS3
* 270 # 306 O 340

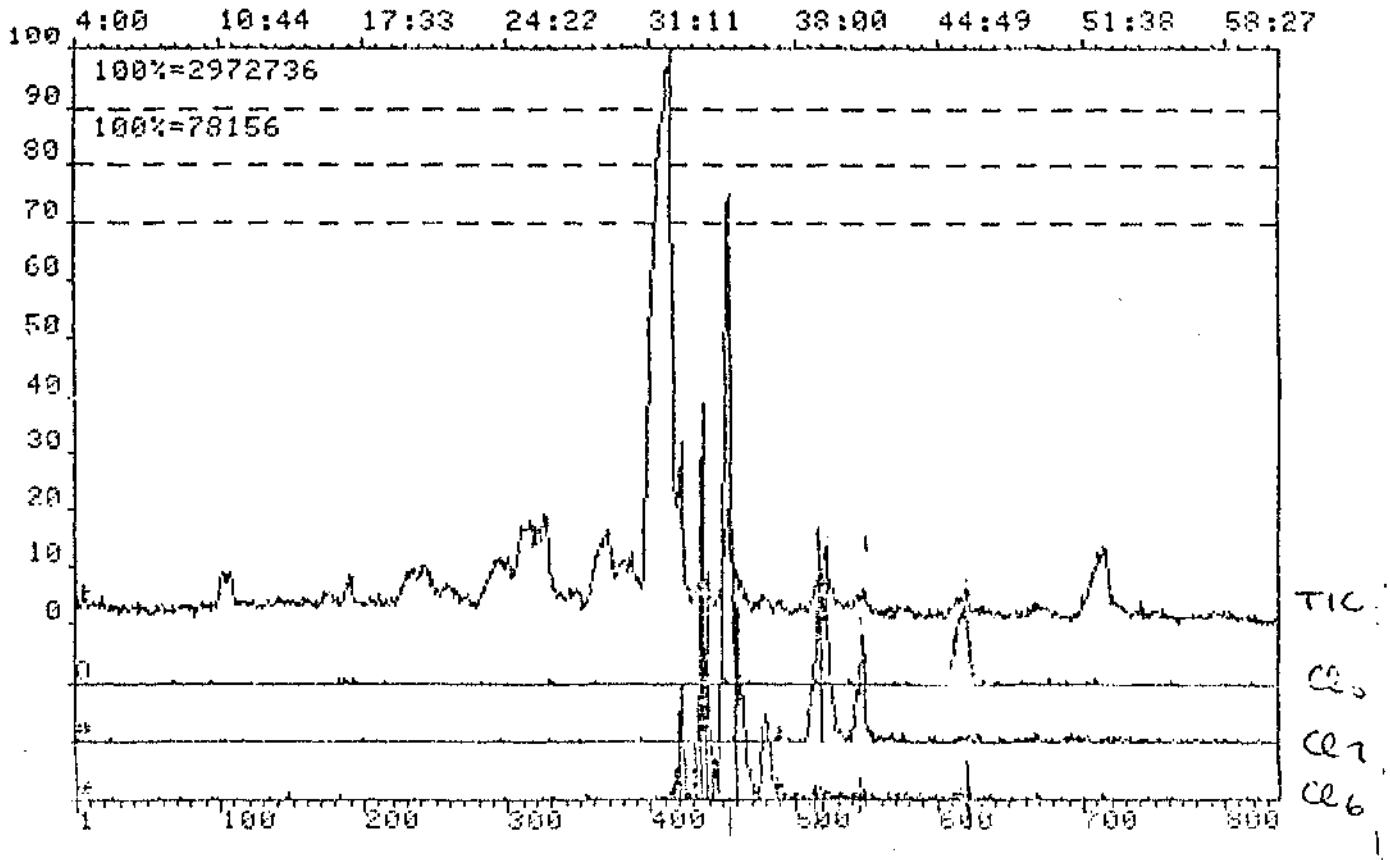


R

3-5-88

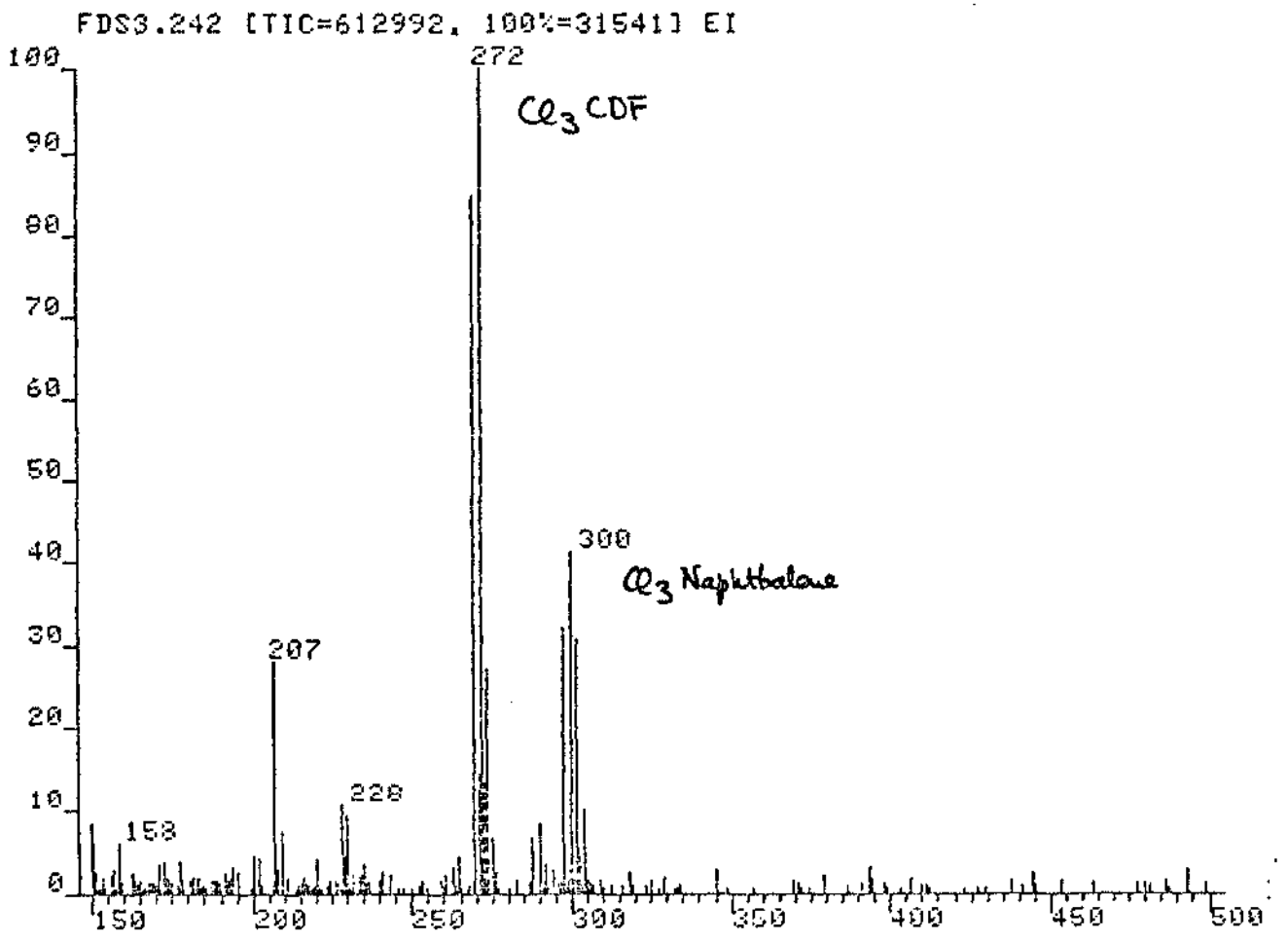
DS-55 CROSS SCAN REPORT, RUN: FDS3
* 374 # 408 O 444 + TIC

- furan

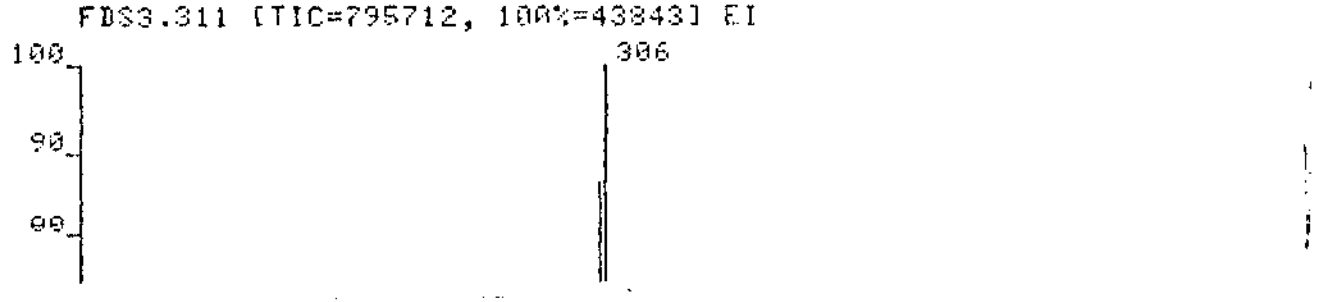


R

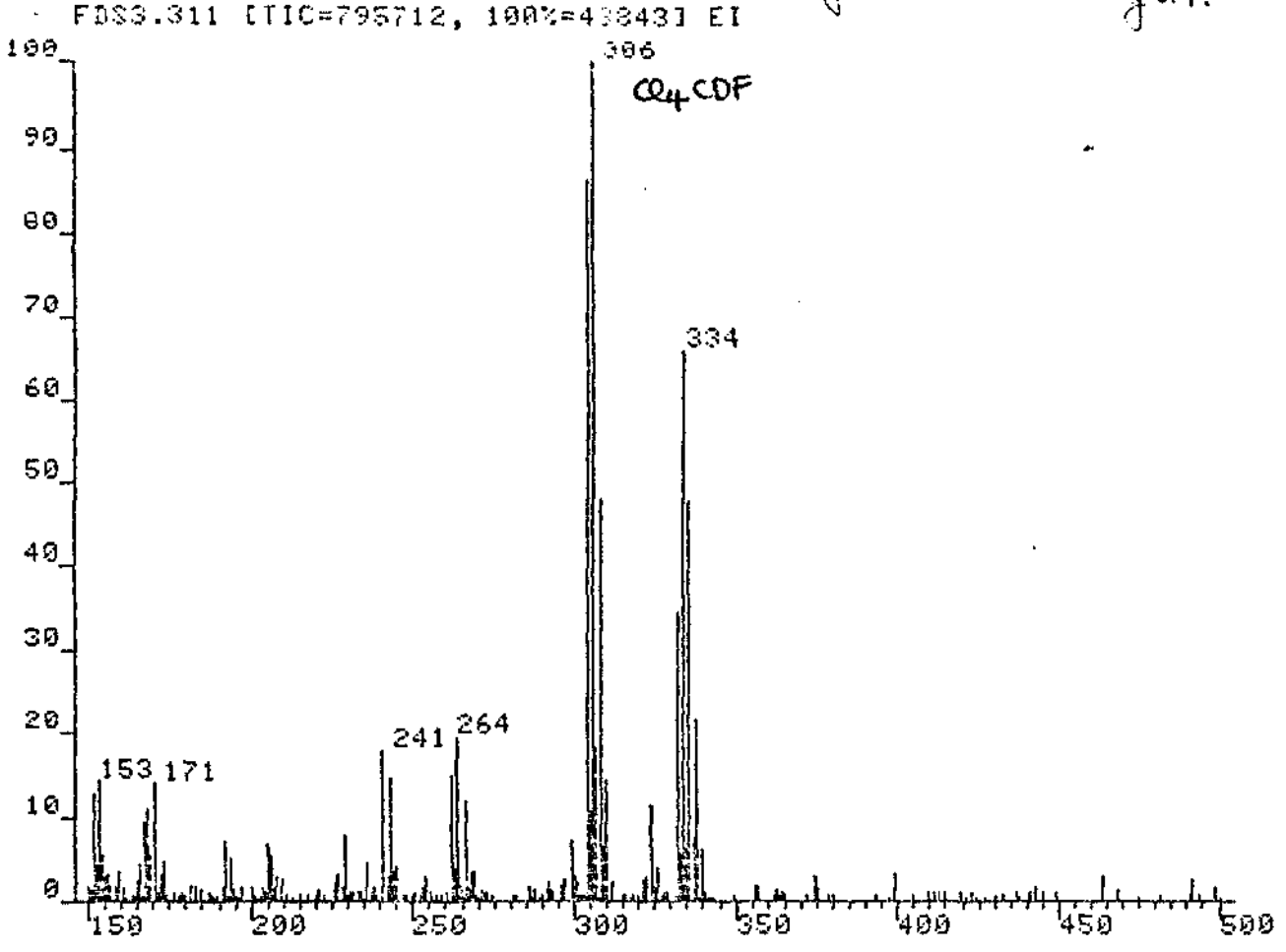
Fig 30 - Mass Spectrum of Scan 242 from chromatogram given in Fig 29



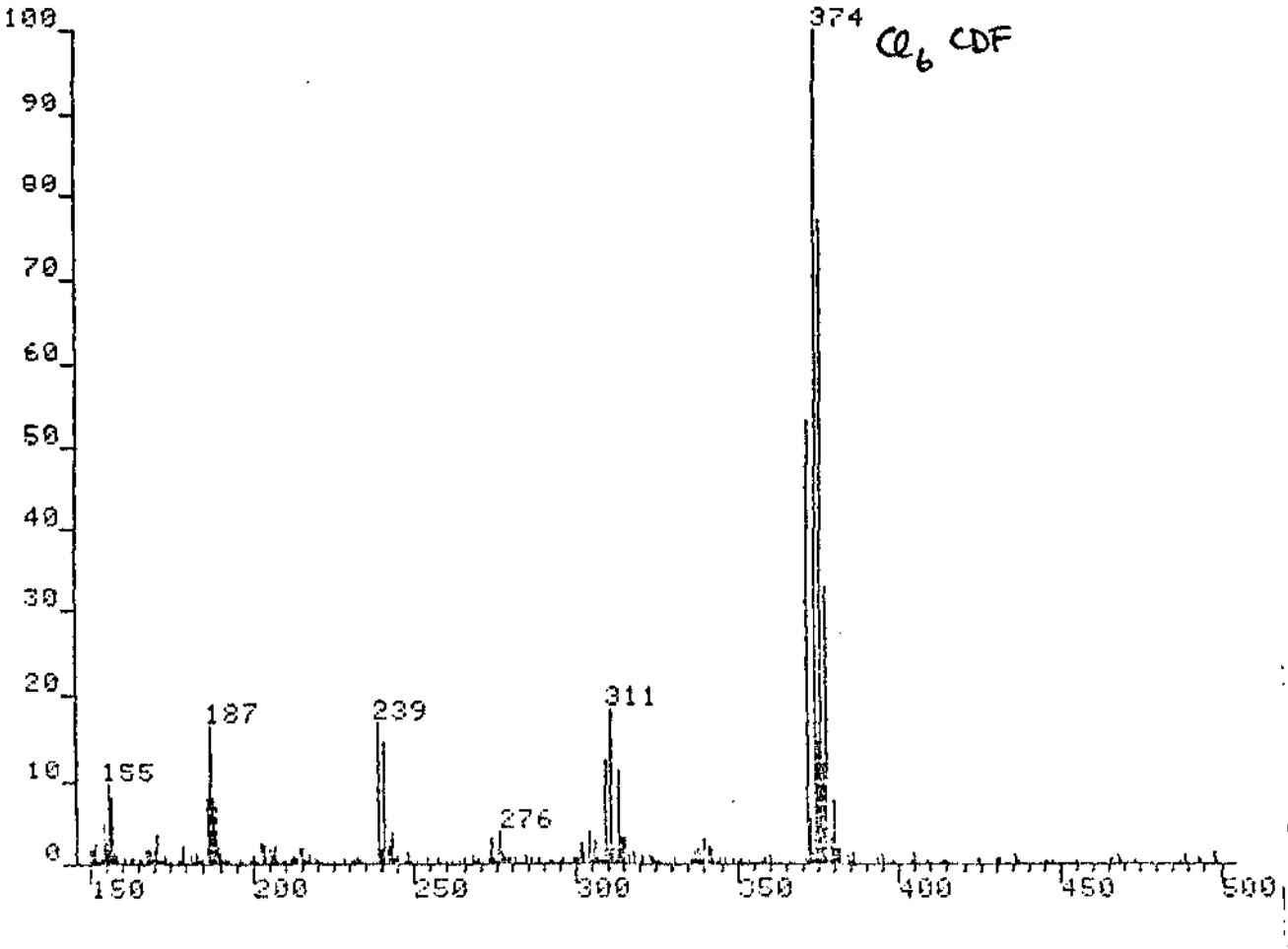
R



Mass spectra of scans from the chromatogram shown in Fig 29.

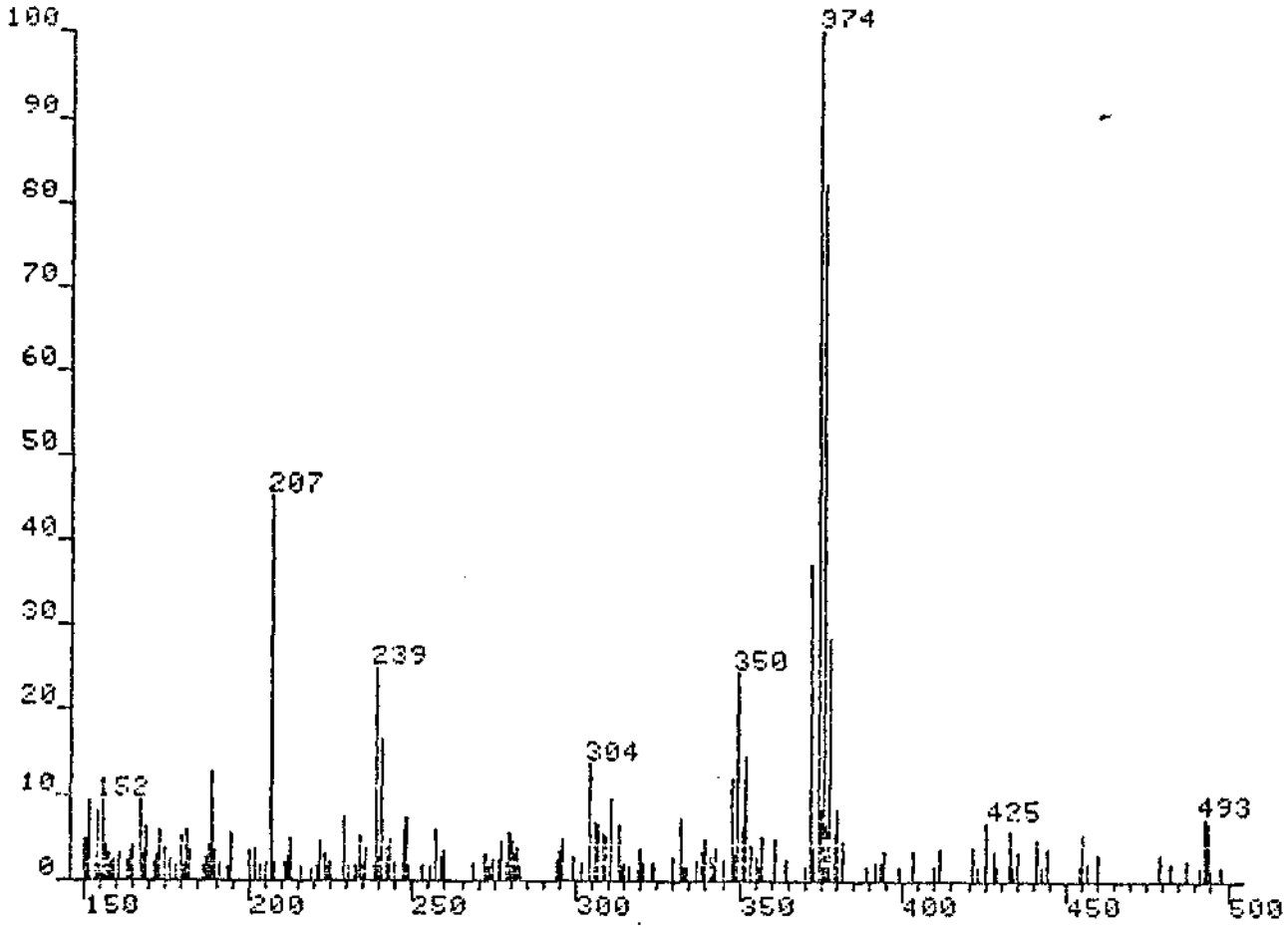


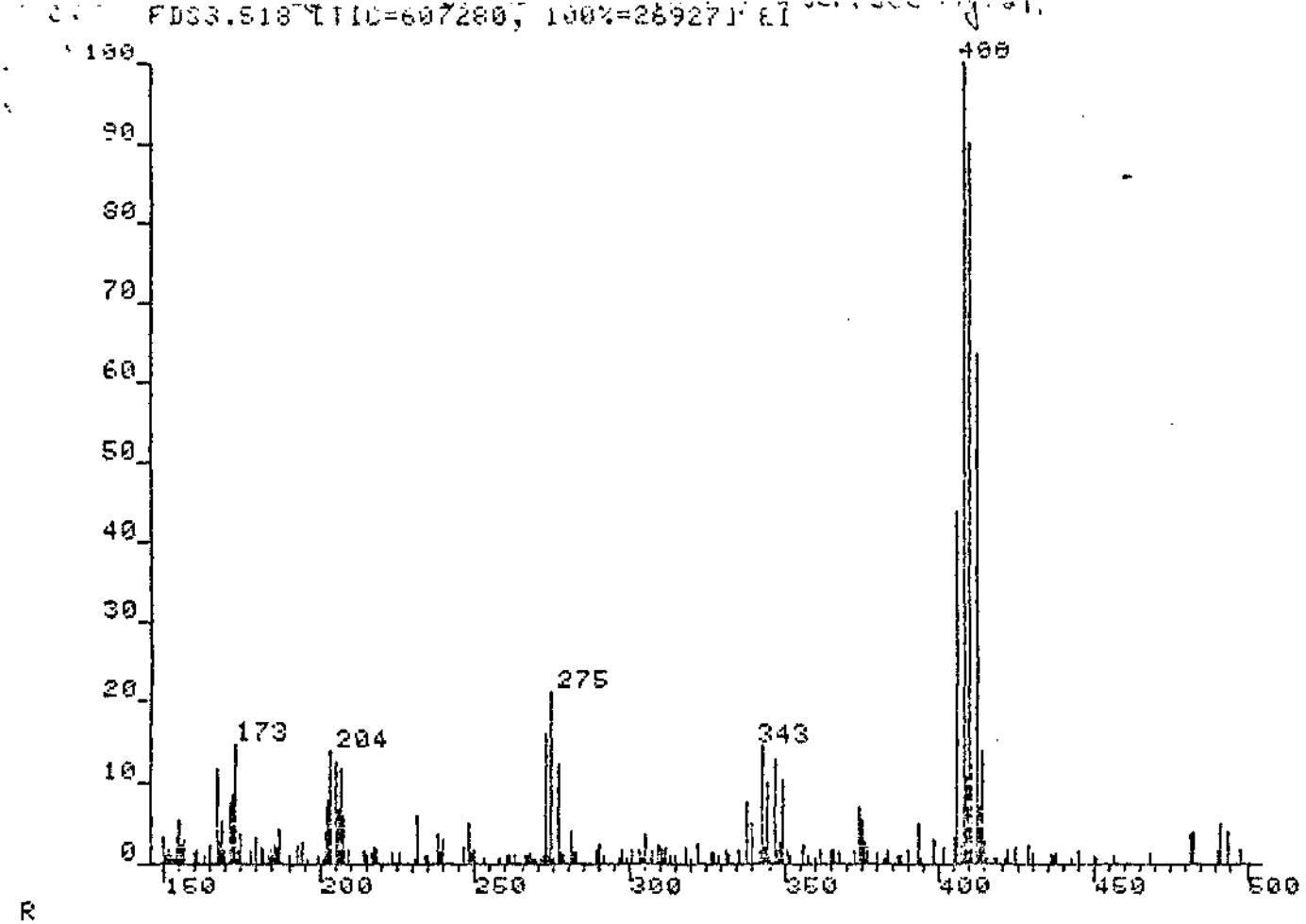
R



R

Fig 32 - Mass spectrum of scan 480 from data set shown in Chromatograms at Fig. 27.




FDS3.518 [TIC=607280, 100%=26927] EI
100
90
80
70
60
50
40
30
20
10
0
150
200
250
300
350
400
450
500
400
275
173
204
343
R

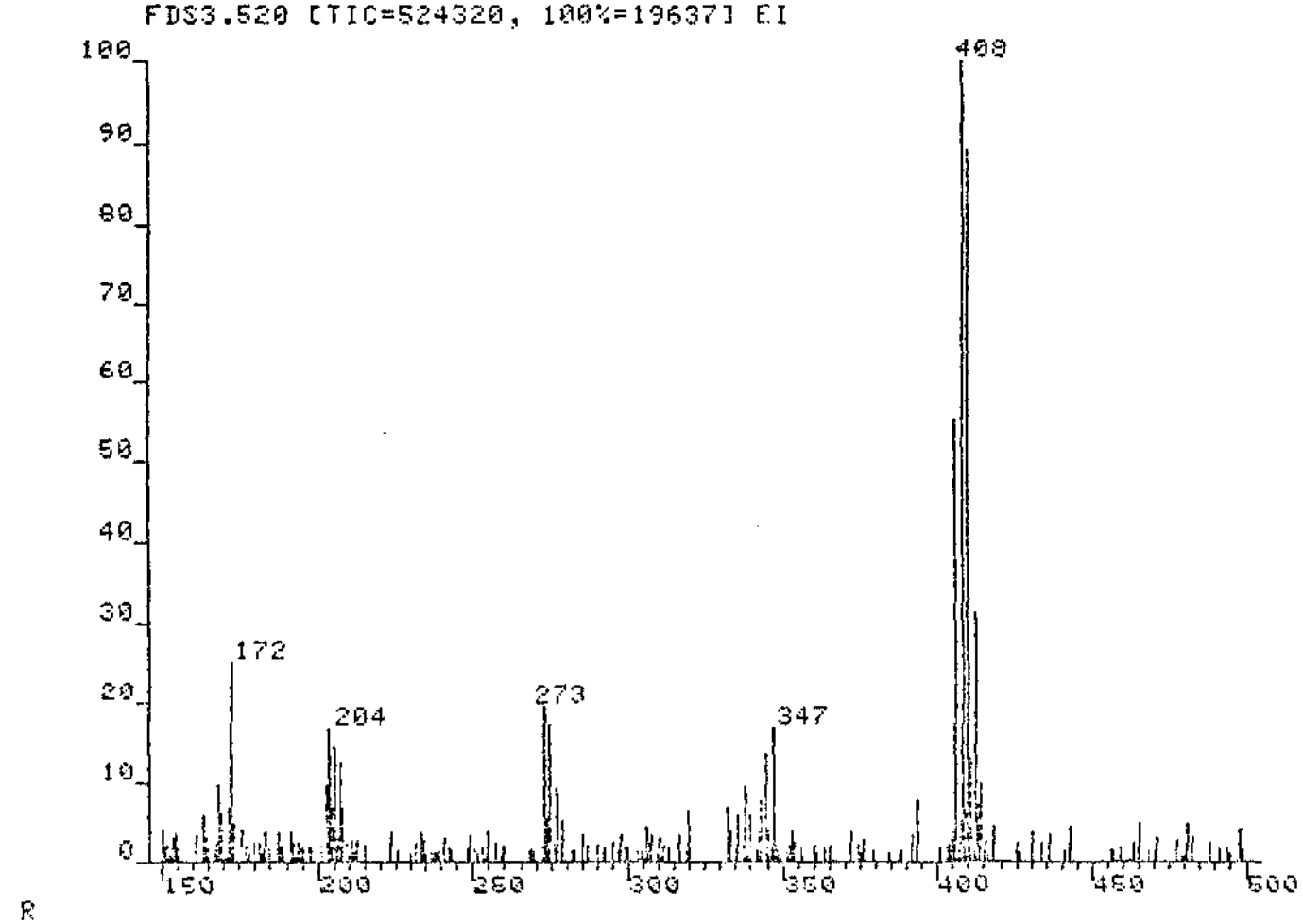
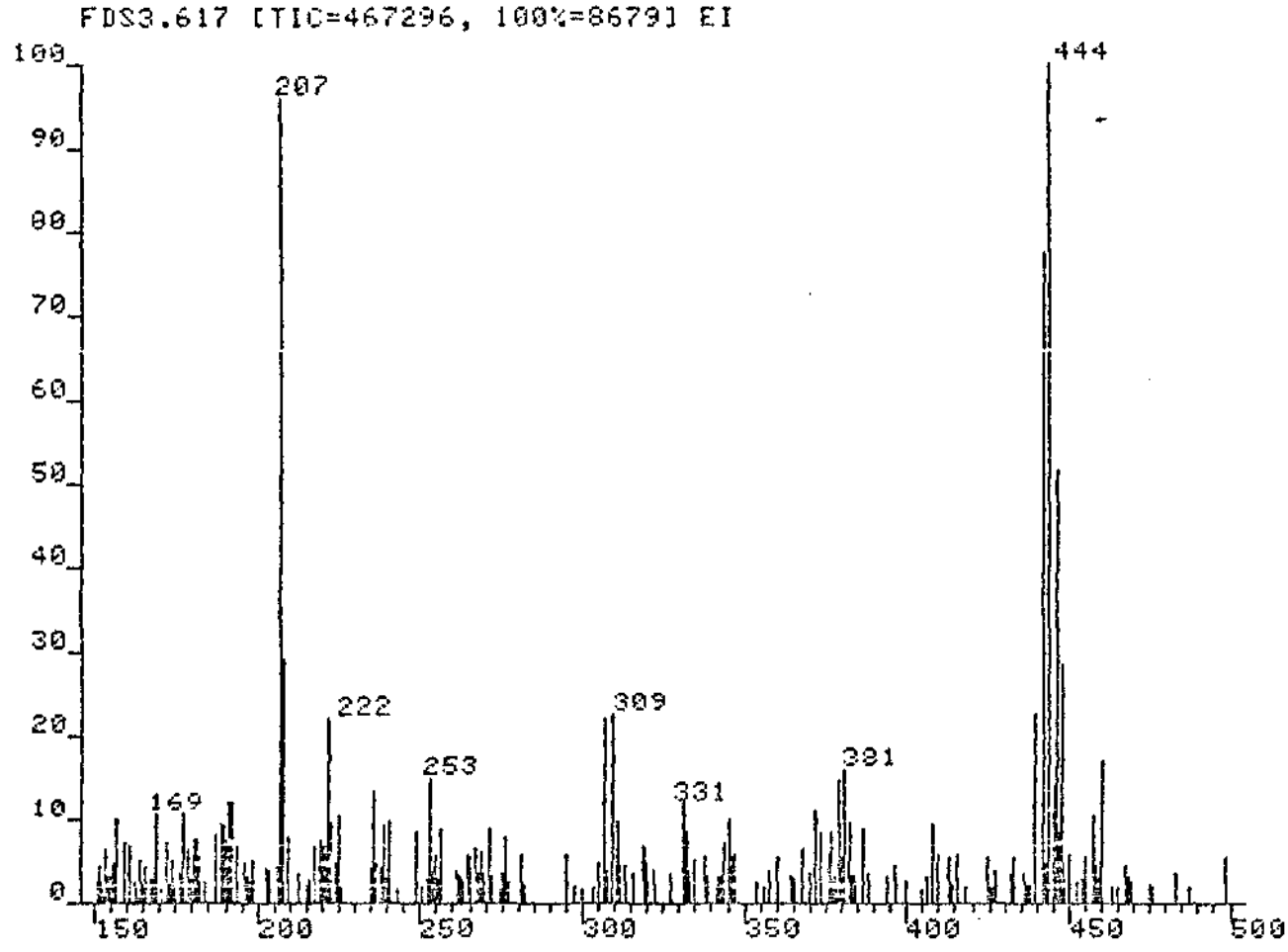

FDS3.520 [TIC=524320, 100%=196371 EI
100
90
80
70
60
50
40
30
20
10
0
150
200
250
300
350
400
450
500
409
172
204
273
347
R

Fig. 34


FDS3.617 [TIC=467296, 100%=86791] EI
100
90
80
70
60
50
40
30
20
10
0
207
444
222
309
253
331
381
169
150
200
250
300
350
400
450
500
R

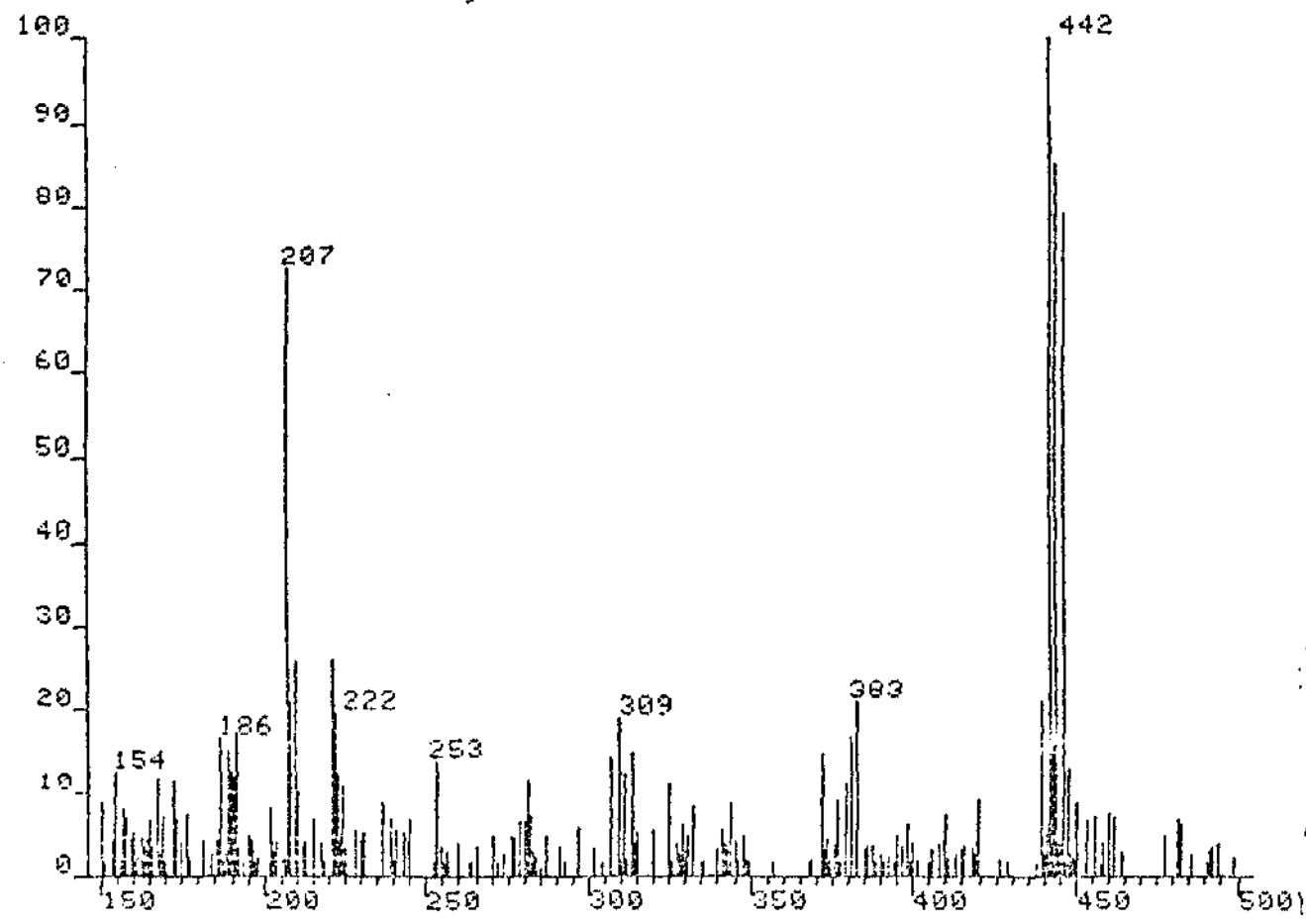
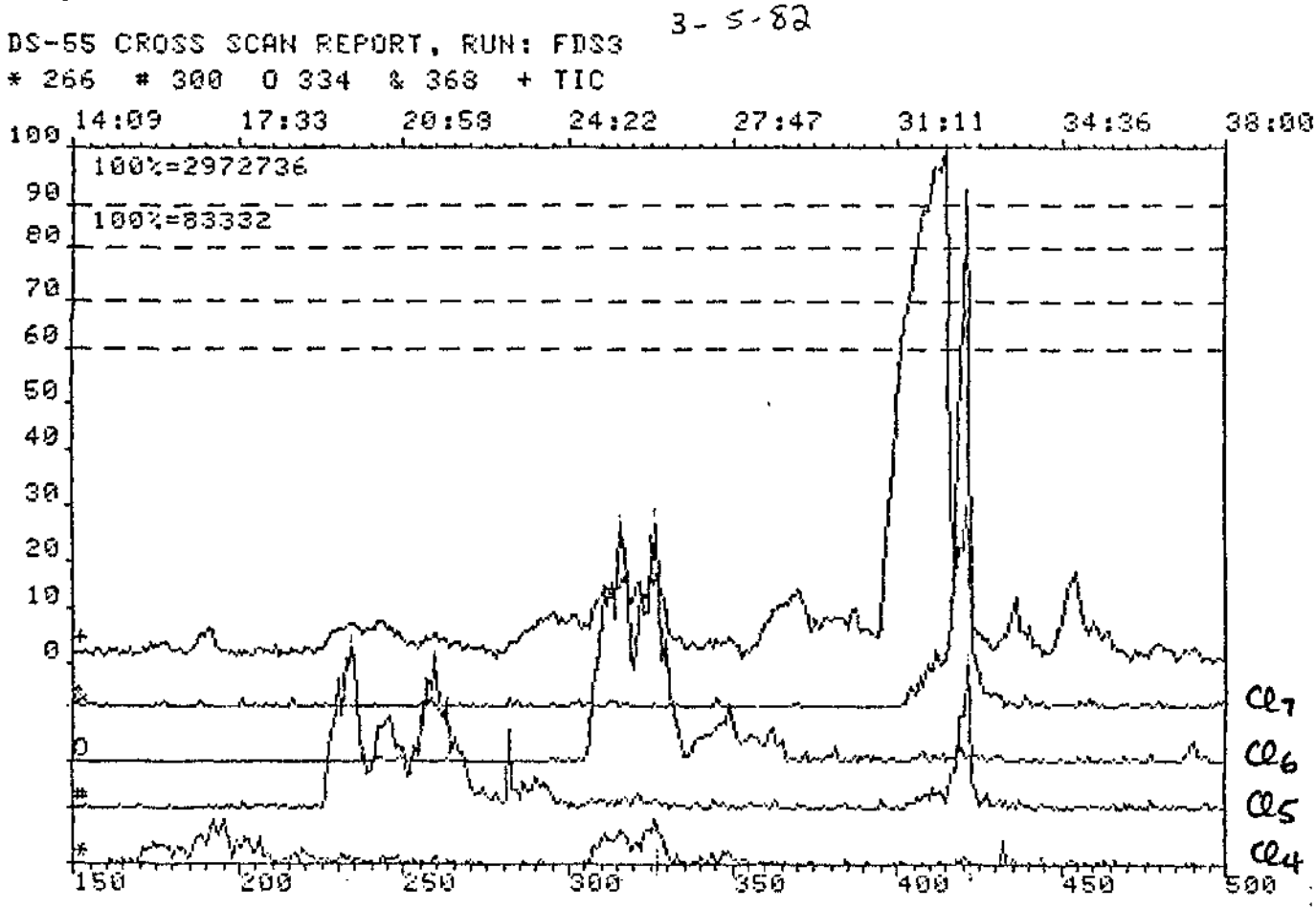

FDS3.620 [TIC=512112, 100%=105521] EI
100
90
80
70
60
50
40
30
20
10
0
207
442
222
309
303
253
186
154
150
200
250
300
350
400
450
500
R

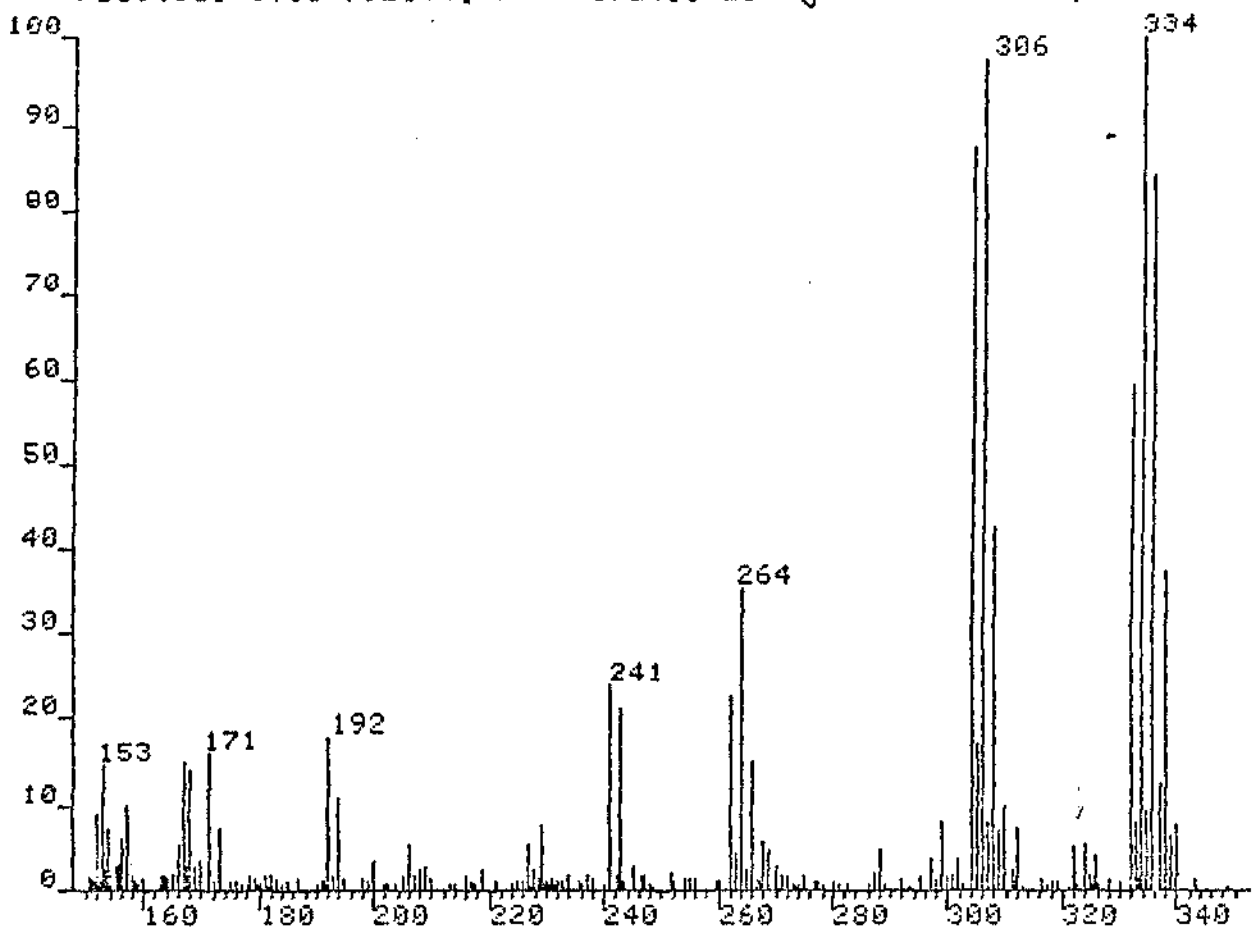
Fig. 35

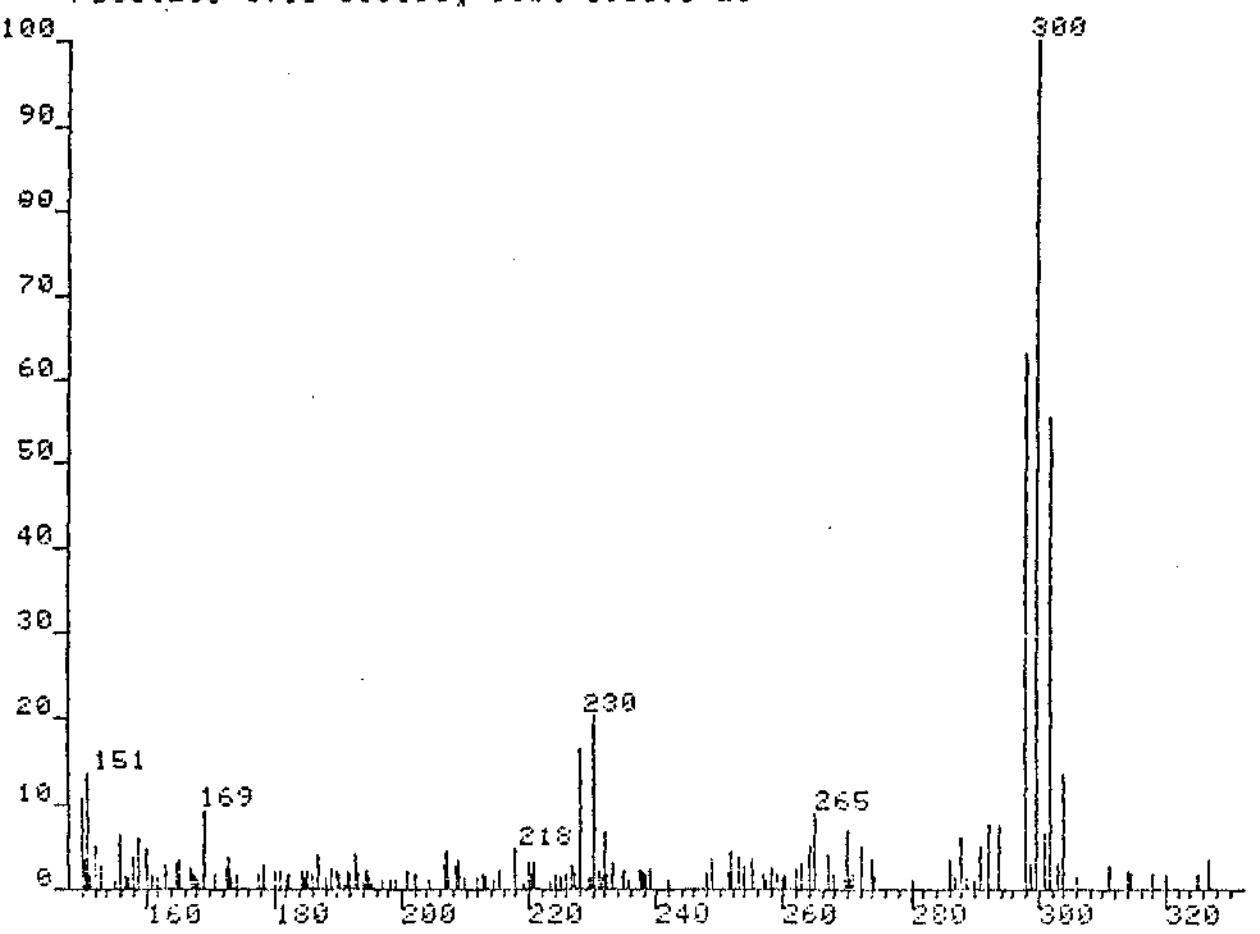
3-5-82



R Exact Mass Chromats of Chloronaphthalenes in 5th floor sample.
Top trace is TIC chromatogram.

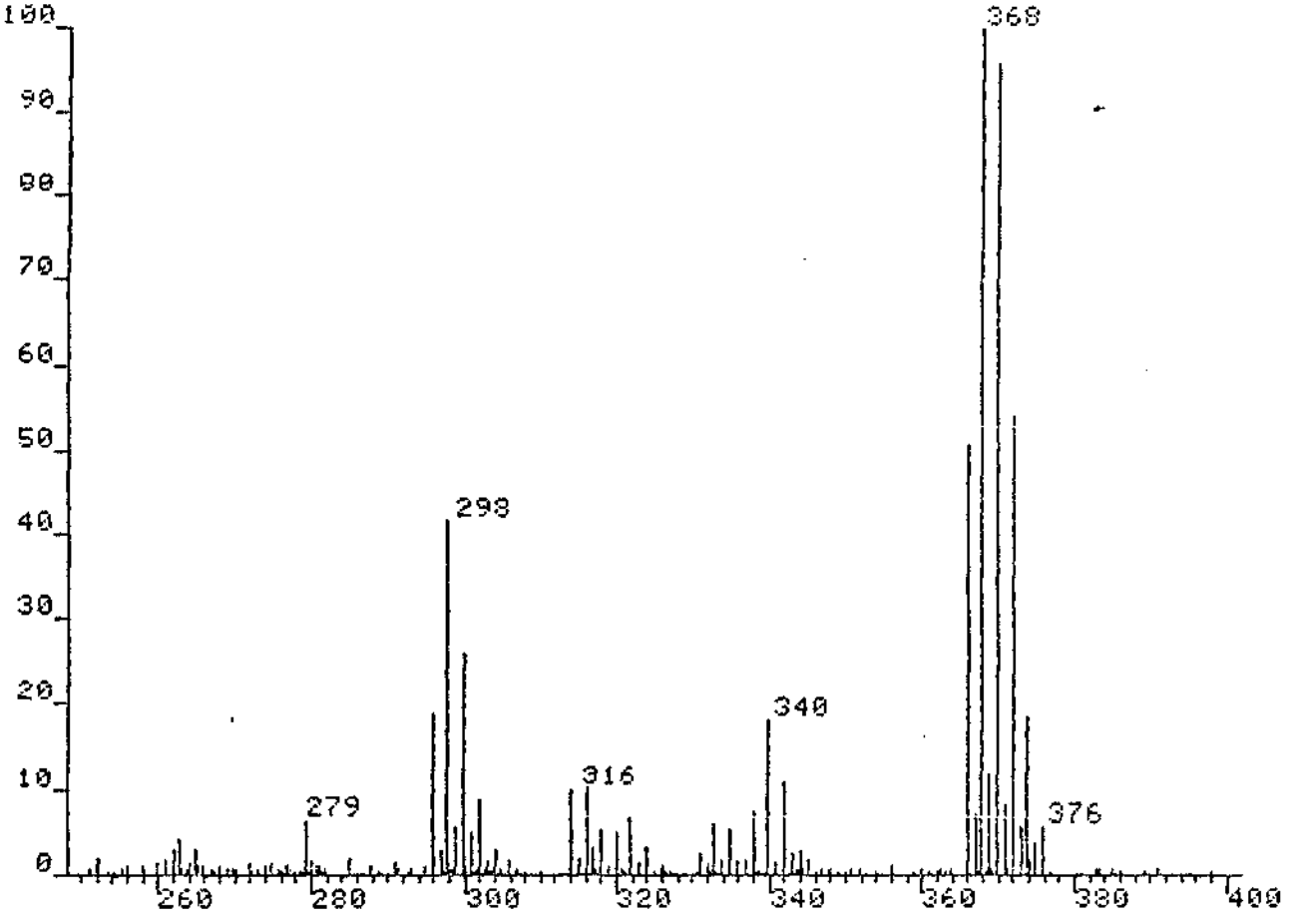
Fig 3.6 & 3.7 - Mass Spectra from 5th floor injection showing various chloronaphalenes.





37.

FDS3.422 [TIC=957216, 100%=60380] EI



R

Fig 38 - TIC chromatogram of soil homogenate.

Fig 38

GC-MS CROSS SCAN REPORT, RUN: WD1S4

+ TIC

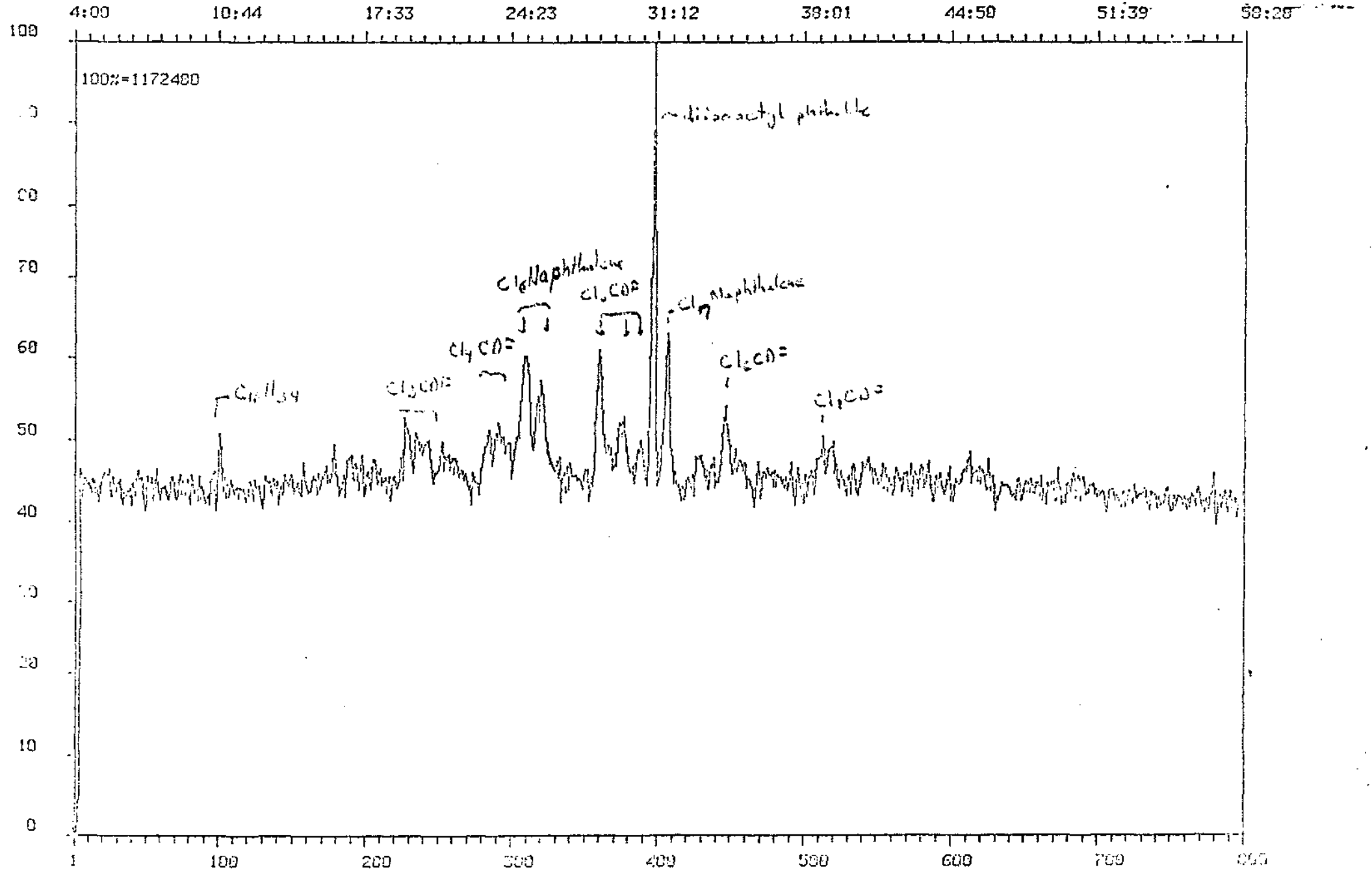


Fig. 39. Exact mass chromatograms of ions due to the PCDF's.

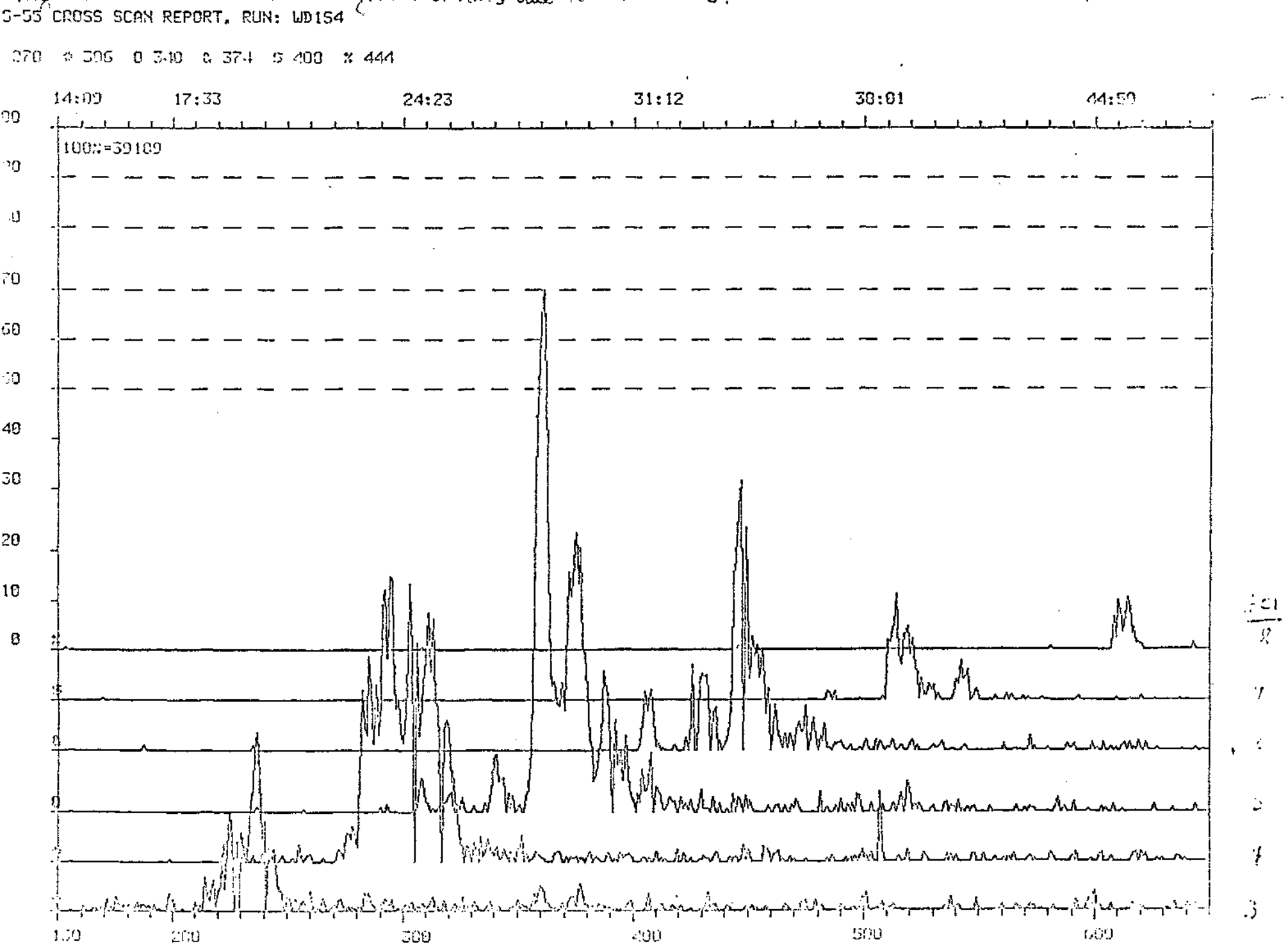


Fig. 40-#5 - Mass spectra from soot homogenate data verifying the presence of the PCDF's.

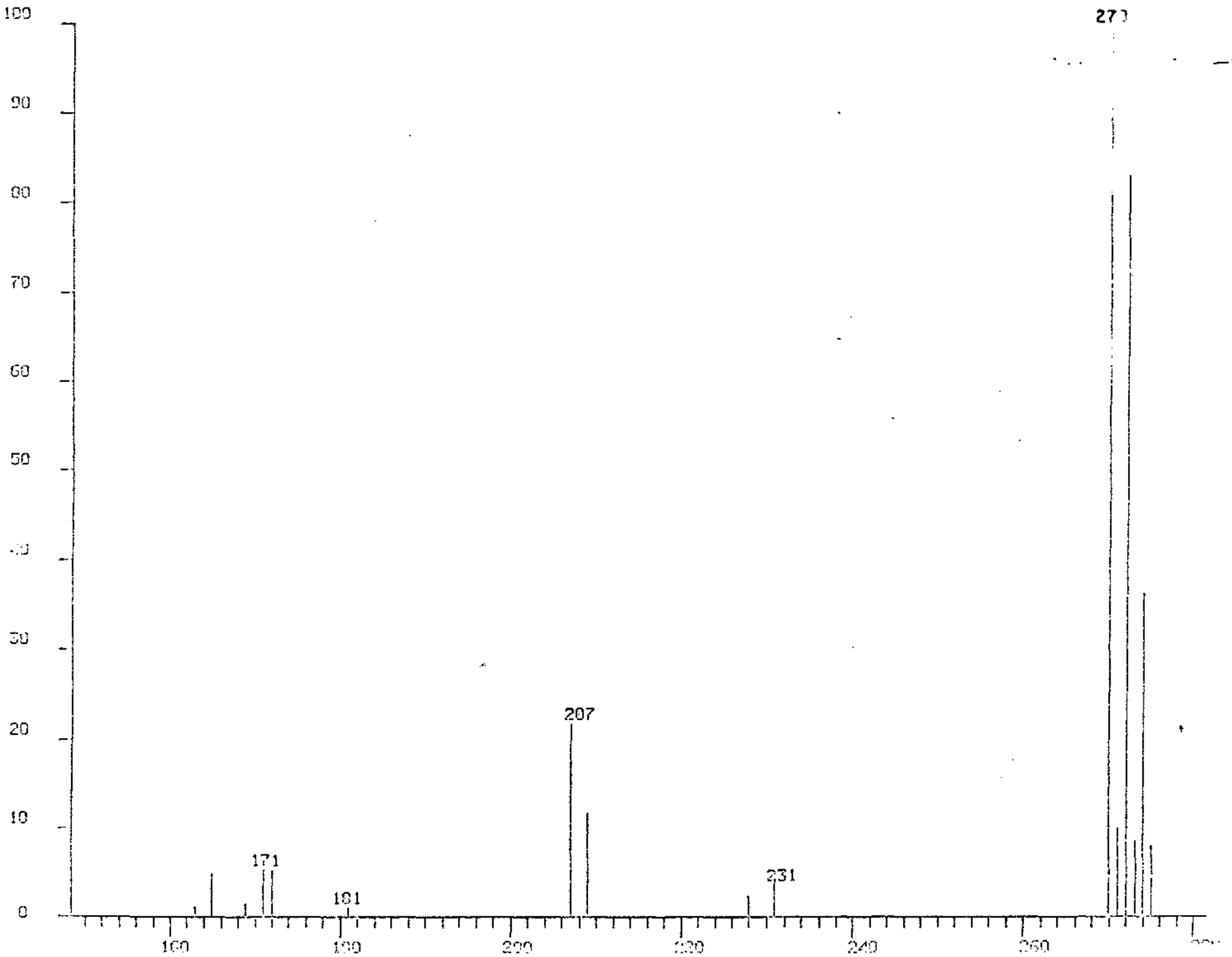


Fig. 41.

TEF292.1 [TIC=512112, 100%=16480] EI

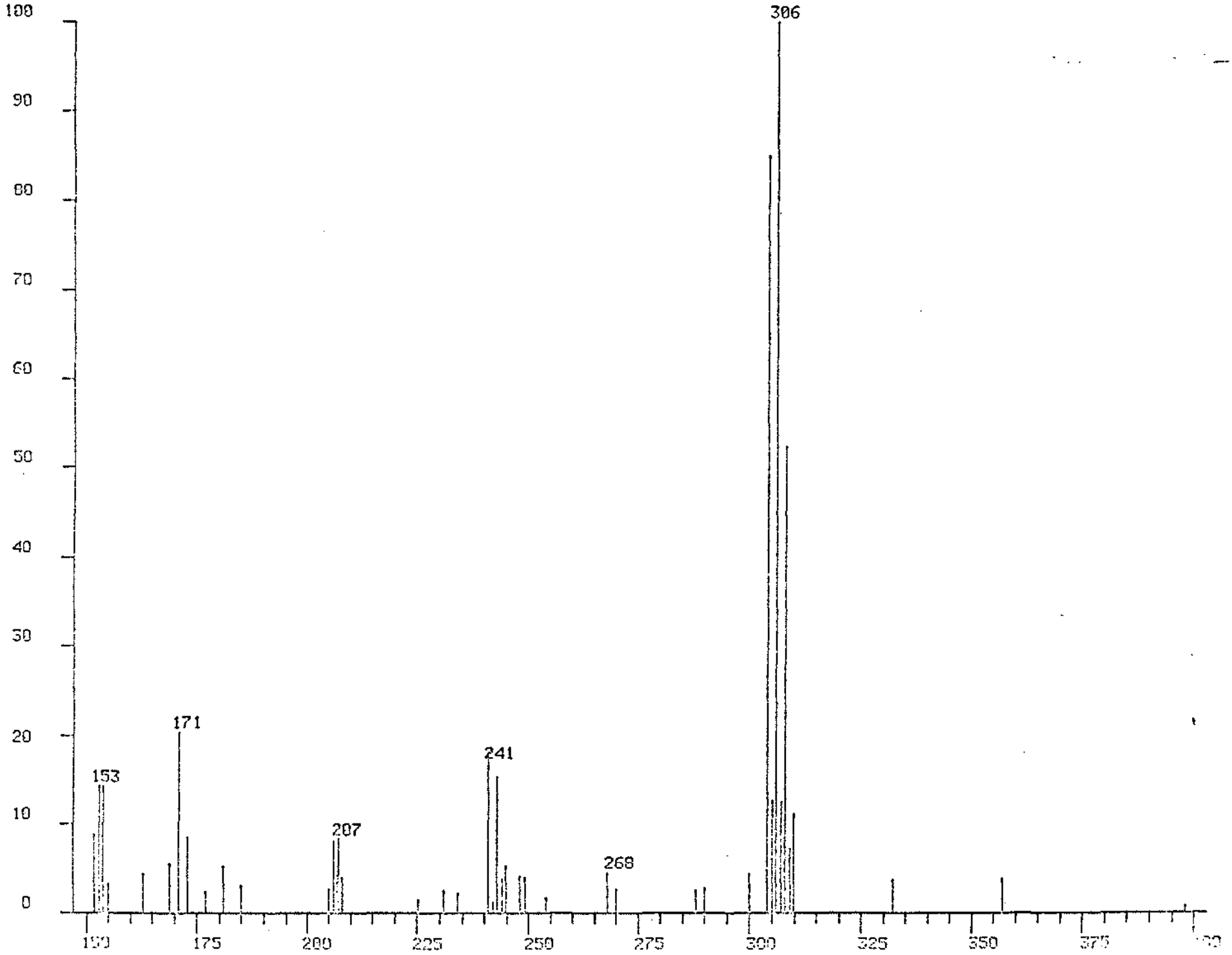


Fig. 42.

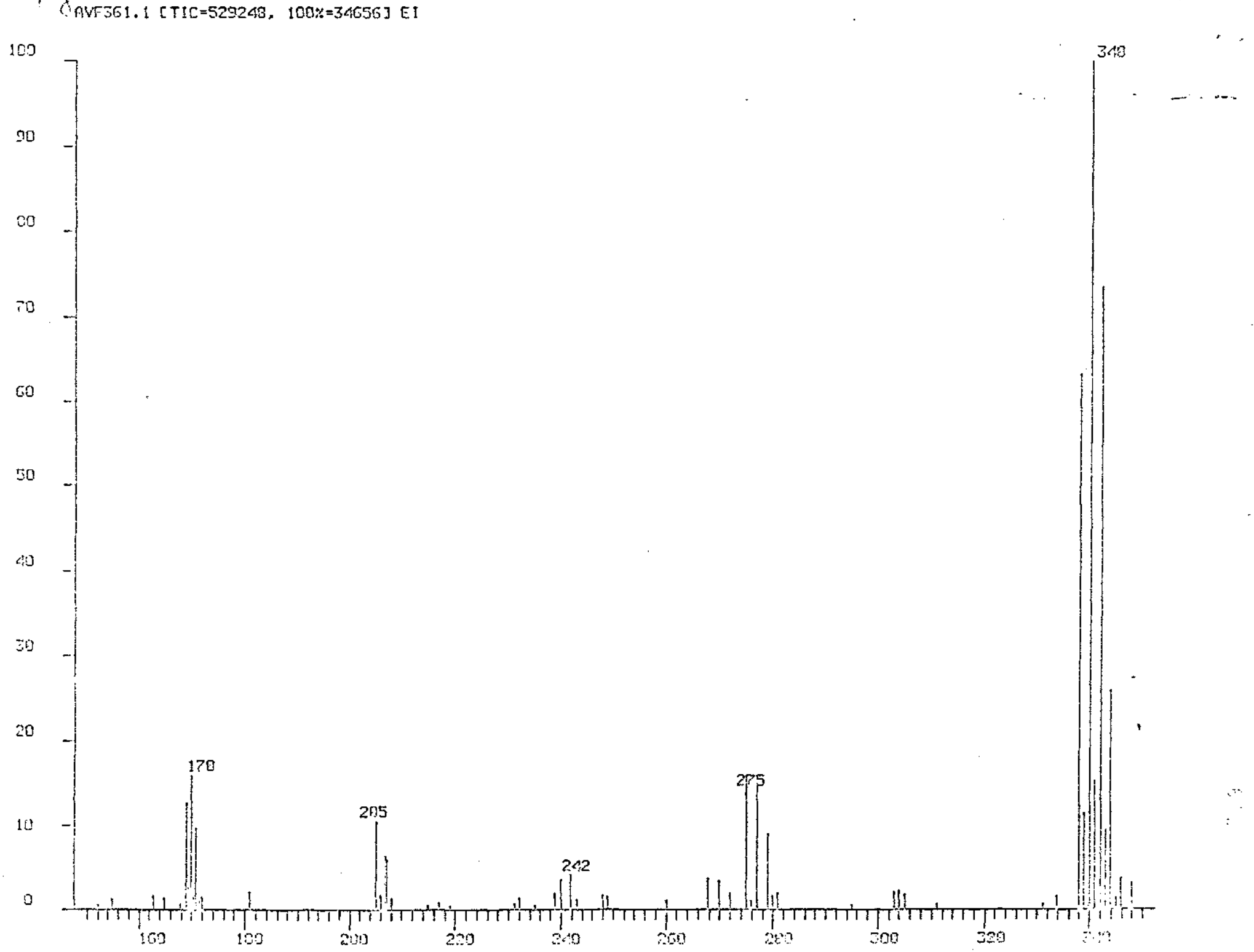

AVF361.1 [TIC=529248, 100%=34656] EI
100
90
80
70
60
50
40
30
20
10
0
348
275
205
242
178
160
180
200
220
240
260
280
300
320

Fig. 43

HX5447.1 [TIC=460520, 100%=175191] EI

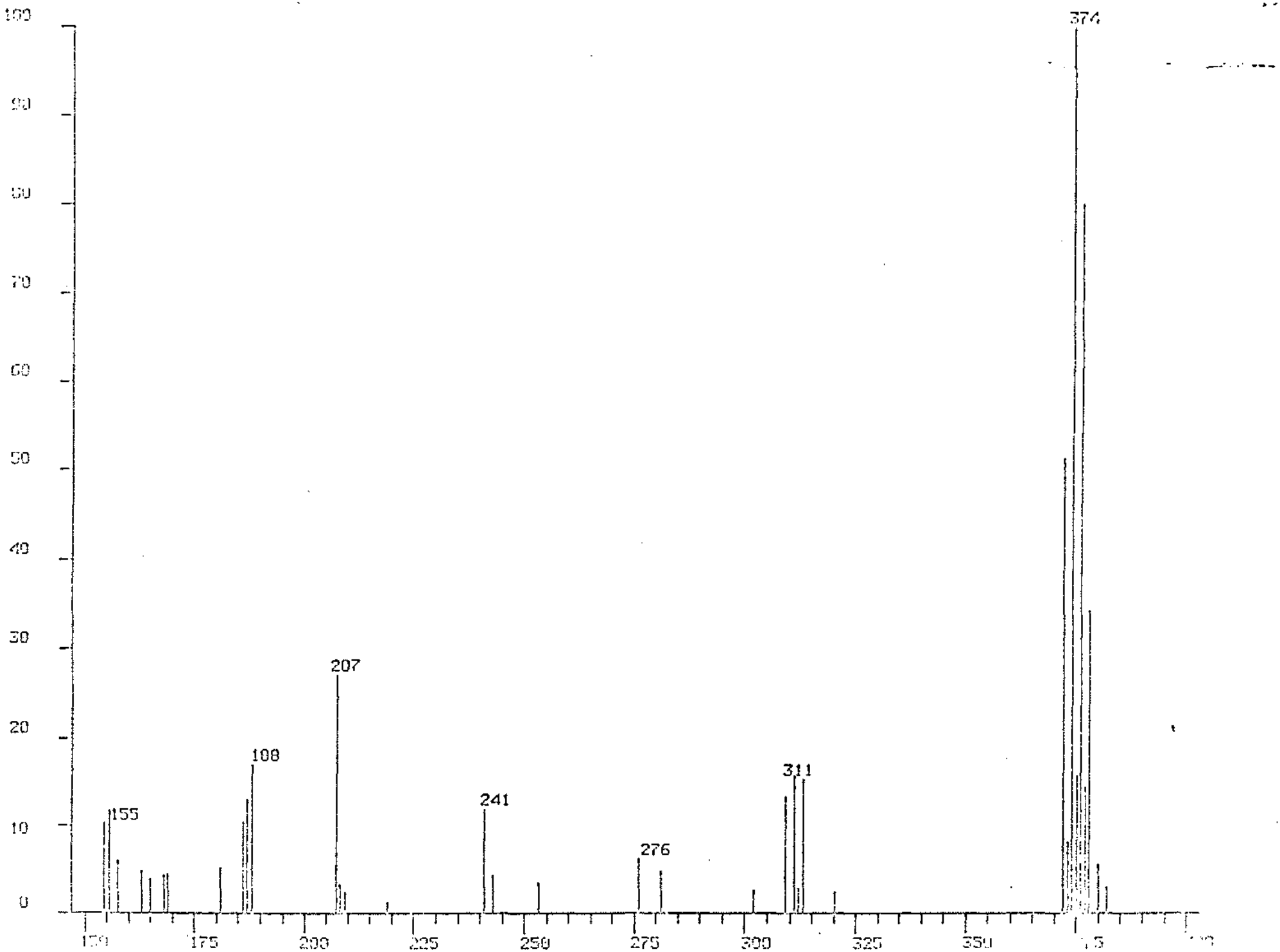


Fig 44

HPF514.1 [TIC=95480, 100%=4097] EI

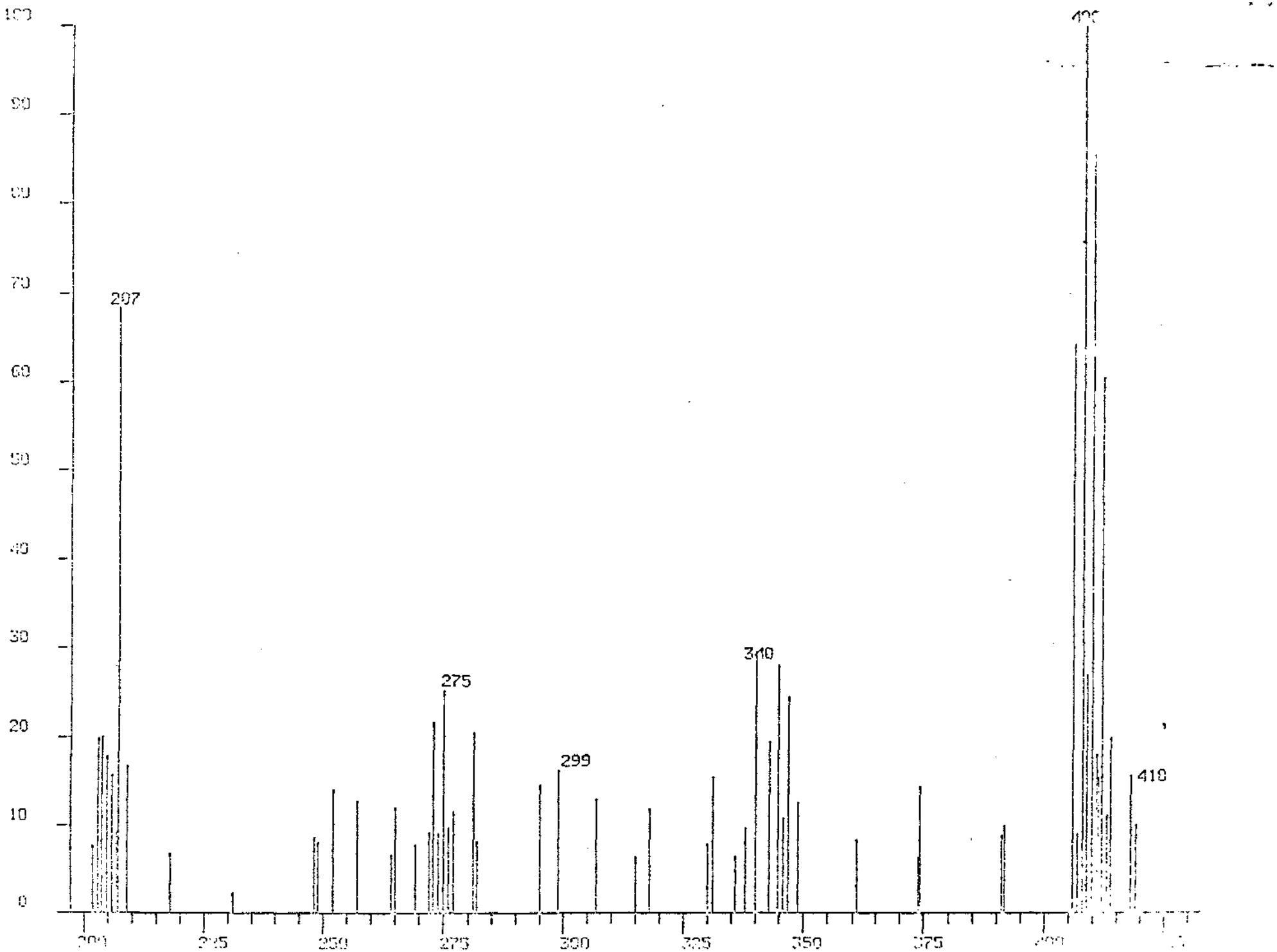


Fig. 45.

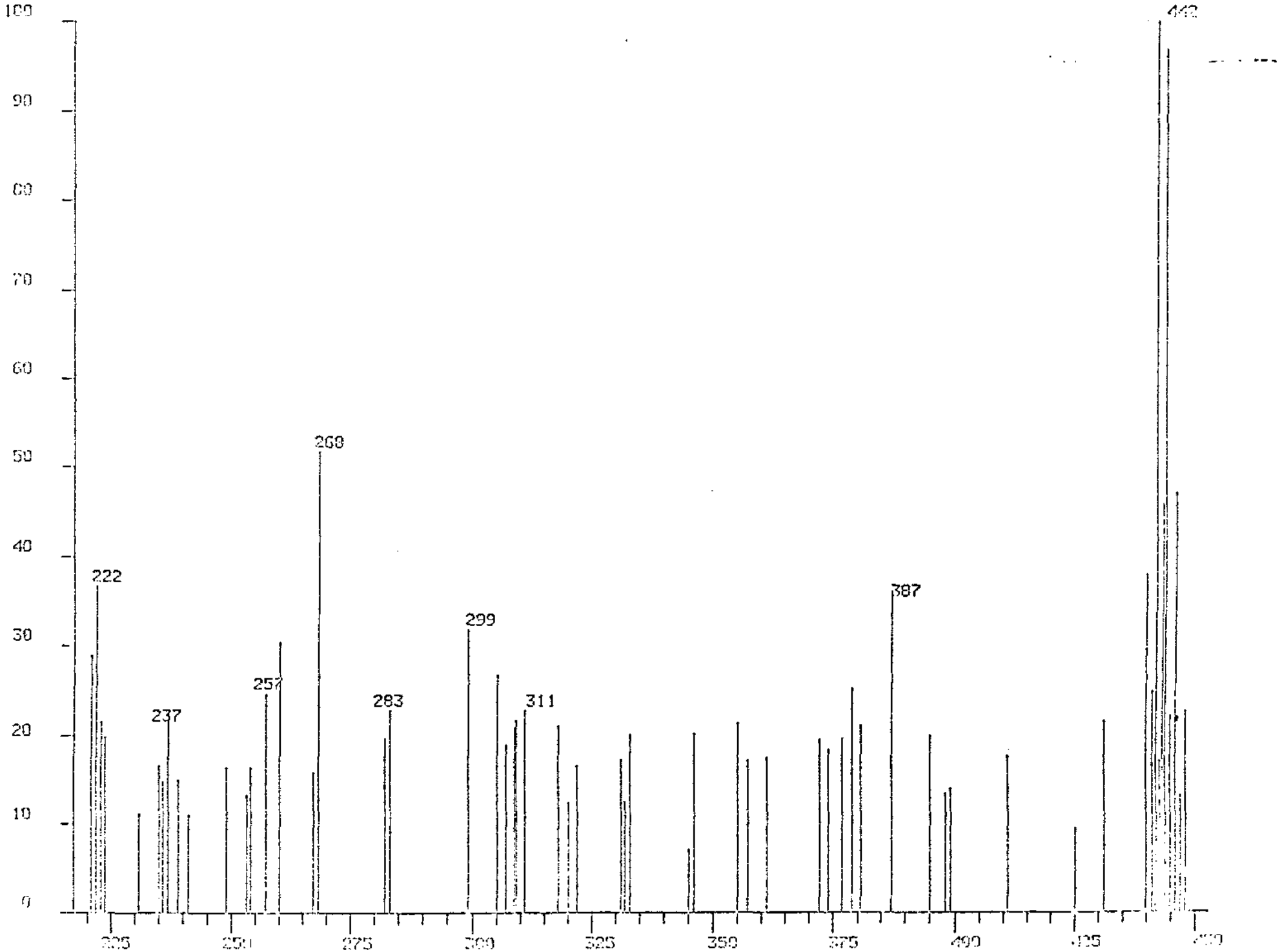

DMFG10.1 [TIC=73152, 100%=2067] EI
100
90
80
70
60
50
40
30
20
10
0
222
237
257
268
283
299
311
387
442
225
250
275
300
325
350
375
400
425

Fig 4b - Exact Mass Chromatograms of ions due to polychloronaphthalenes: Soot homogenate data.

Fig 4b

DS-55 CROSS SCAN REPORT, RUN: WD154

× 266 * 300 0 334 & 368 $ 404 + TIC

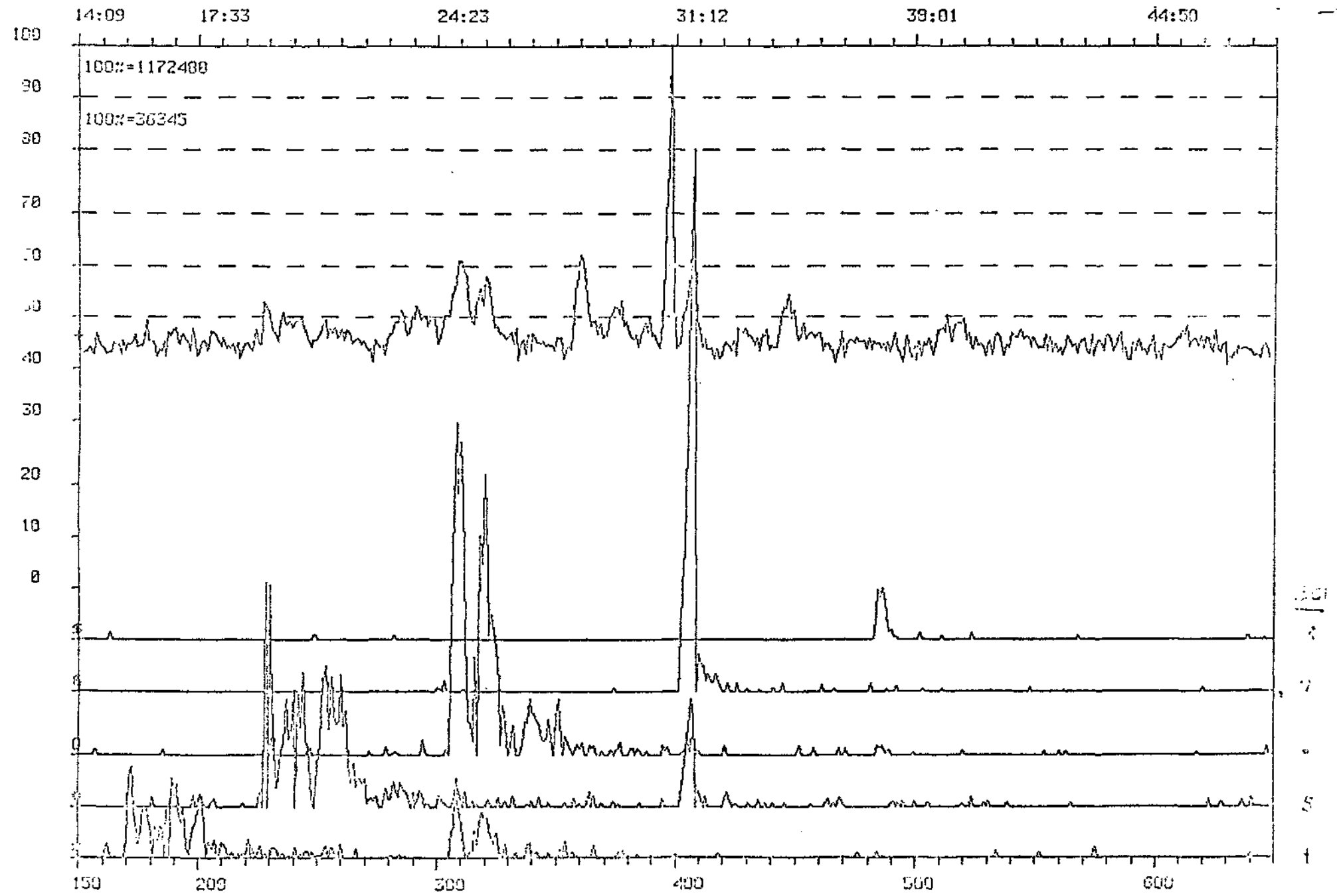


Fig. 47-51. Mass Spectra verifying the presence of various polychloronaphthalenes in soot [illegible] sample.

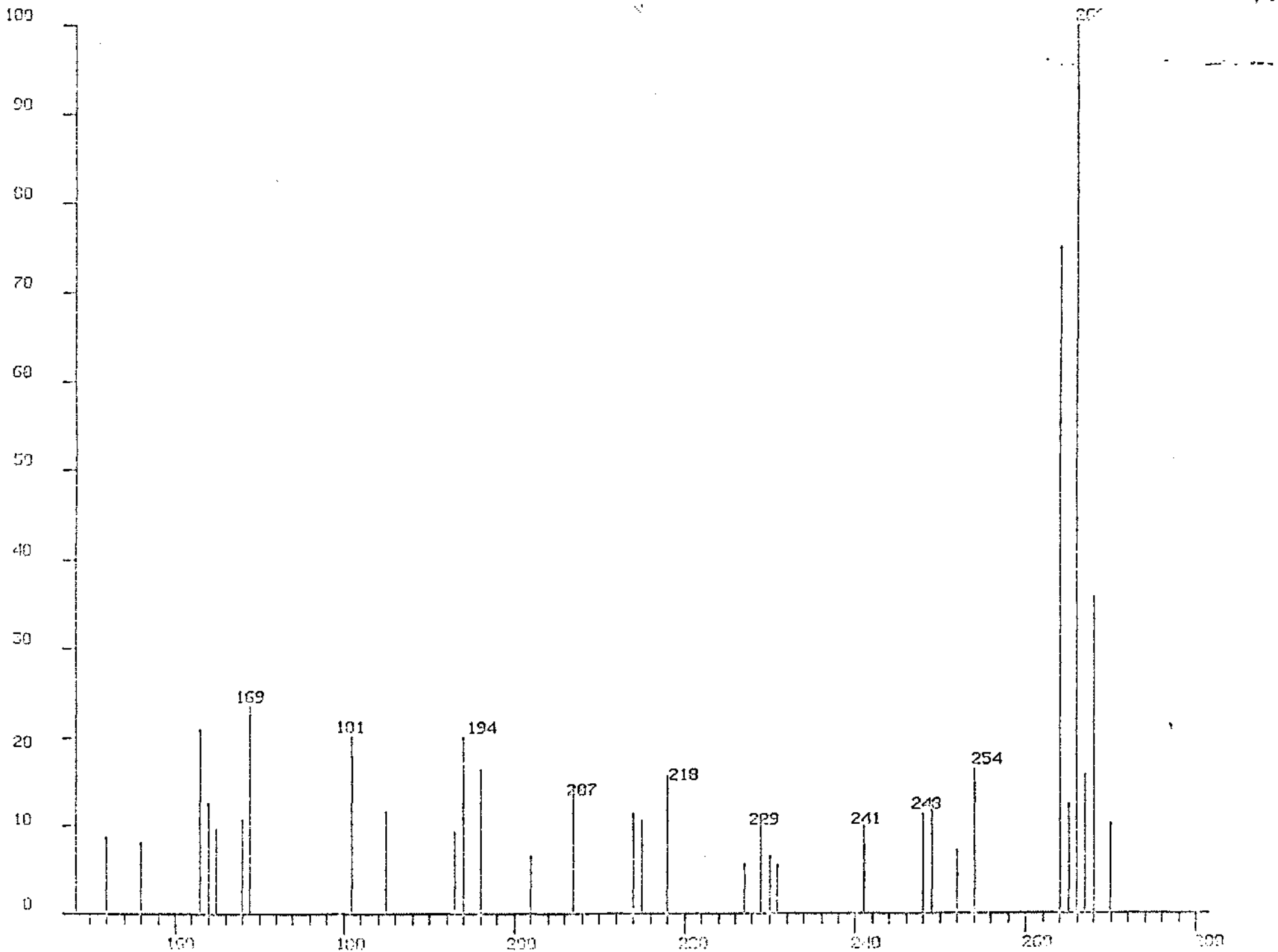


Fig. 4A

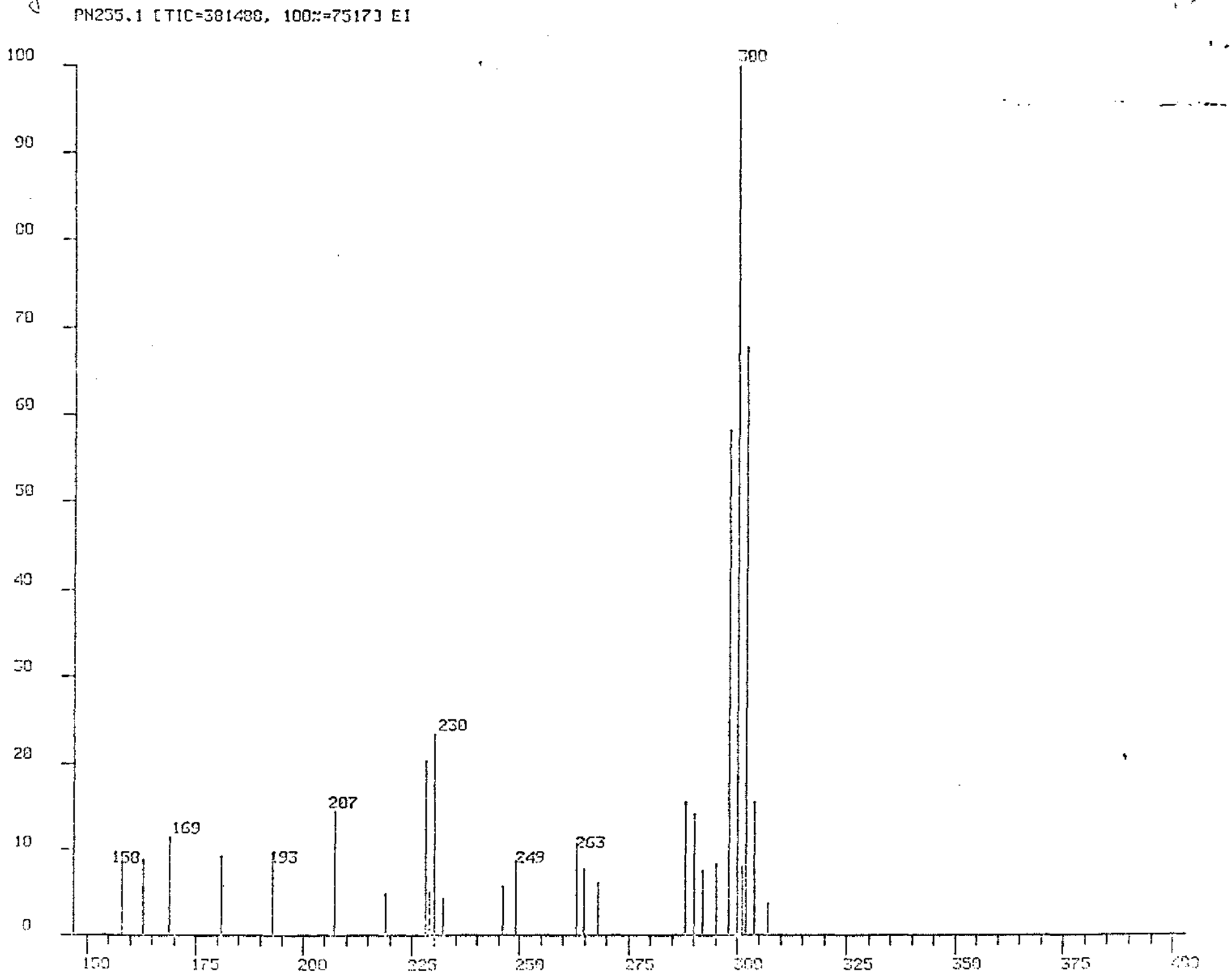

PN255.1 [TIC=381488, 100%=75173] EI
100
90
80
70
60
50
40
30
20
10
0
150
169
193
207
230
249
263
300
150
175
200
225
250
275
300
325
350
375
400

Fig 49

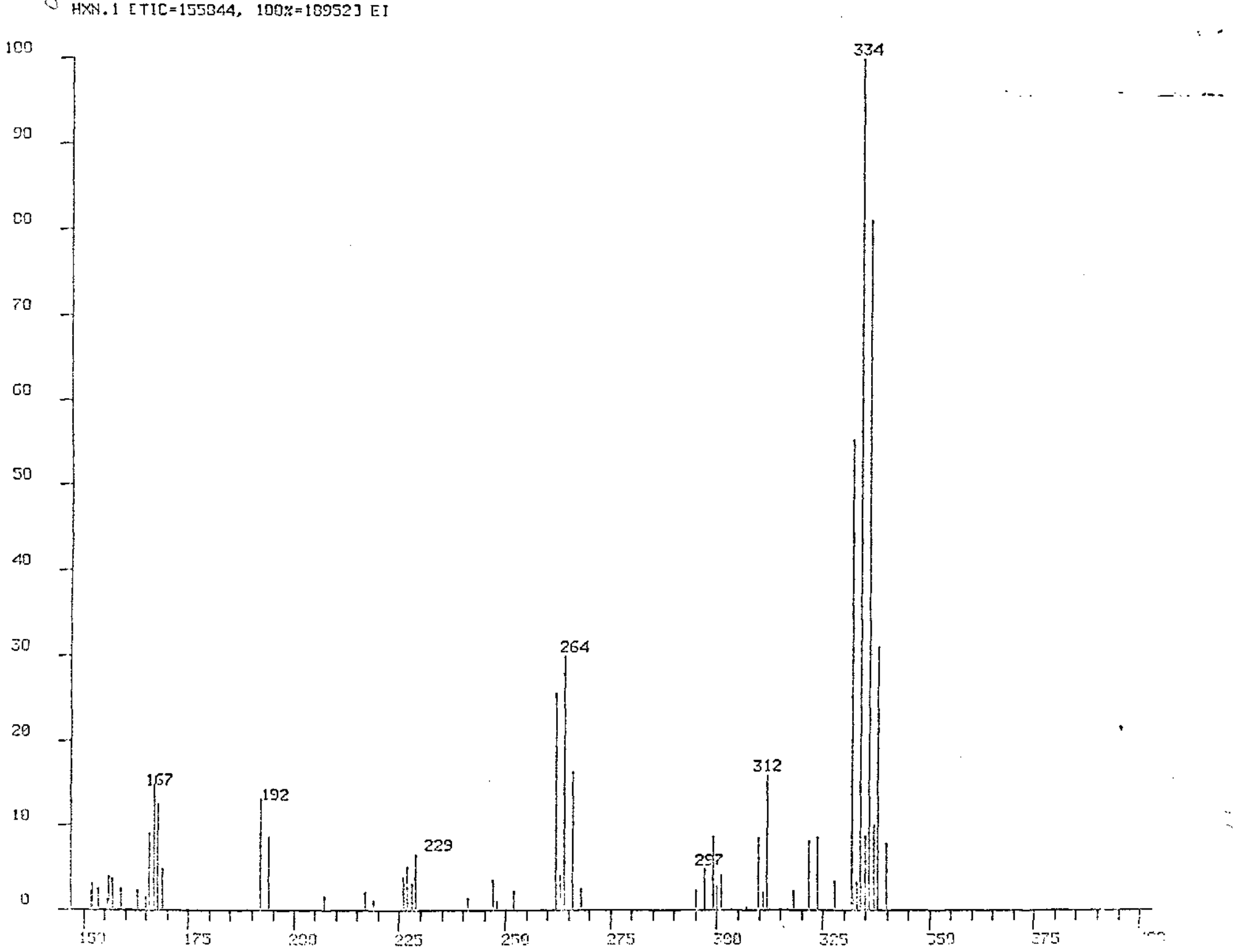

HXN.1 ETIC=155844, 100%=18952] EI
100
90
80
70
60
50
40
30
20
10
0
334
264
312
167
192
229
297
150
175
200
225
250
275
300
325
350
375

Fig. 5D

M2N433.1 [TIC=824936, 100%=313083] EI

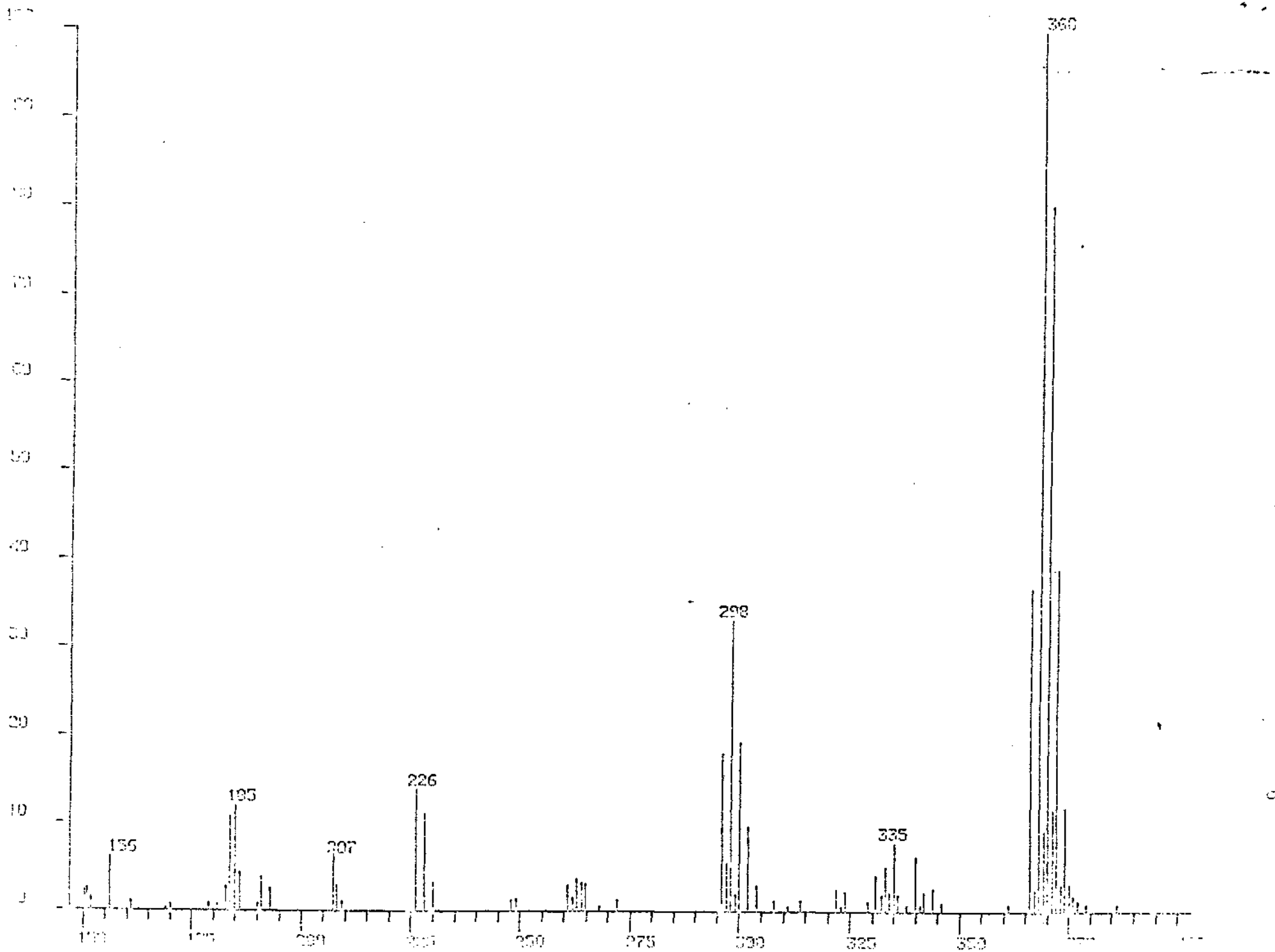


Fig. 51.