

HYDROCARBON ANALYSIS OF SEDIMENT CORES FROM BLIGH WATER, FIJI

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SUMMARY OF FINDINGS

1. Sediment cores from the 1993 Bligh Water Survey , offshore Fiji, contain petroleum hydrocarbons.
2. Analysis of headspace gas in the cores showed the presence of hexane (up to 3ppb) and heptane (up to 8 ppb). The most prominent hydrocarbon in the headspace gas was toluene (up to 300 ppb). C1-C5 hydrocarbons, if present, were below our detection limit of 0.1 ppb.
3. Analysis of extractable organic matter obtained from the sediments showed the presence of hydrocarbons with the aspect of a water-washed and partly biodegraded petroleum. The pattern was the same in all cores studied, including a "core of a core". This suggested that the petroleum was indeed indigenous to the sediment and not the result of contamination.
4. Gas chromatograms of the extractable organic matter were dominated by peaks with a marked carbon number predominance, initially suggesting material of biogenic origin. However, separation of the saturated, aromatic and polar compounds showed that this signature was confined to the polar fraction and was probably due to waxy alcohols of distinct biological origin. The saturated hydrocarbons themselves showed a pattern of n-alkanes with no carbon number preference over the range C21-C31,consistent with an origin from mature petroleum. As well as the n-alkanes, we identified pristane, phytane, steranes and hopanes, all of which had similar distributions in all the cores.
5. The extracts were low in aromatic compounds and the saturated hydrocarbons showed relative abundances which generally increased with increasing carbon number. This pattern is seen in weathered petroleum where the aromatics are depleted, partly as a result of their partial solubility in water and the lighter n-alkanes are removed as a function of increasing volatility (lower MW). The oil has also probably been affected by microbial attack but this has not been so severe as to cause complete destruction of n-alkanes as often happens to biodegraded oil in reservoirs.
6. Although the low molecular weight end of the oil was mostly removed we estimate that the pristane/phytane ratio was about 1.5-2 suggesting an oil of marine origin. This was confirmed by the presence of two steranes, 24-n-propylcholestane

and dinosterane which are commonly found in abundance in oils containing marine organic matter. 28,30-Bisnorhopane was also abundant and this is also a feature normally associated with marine oils. Oleanane was detected (tentatively) in trace amounts signifying some contribution from terrestrial sources. This need not have been a major contribution since this compound is present in many marine oils, apparently as a result of the ubiquitous and recalcitrant nature of its precursor lipids during transport and sedimentation. Based on our present knowledge, oleanane probably only occurs in oils of late Cretaceous age and younger and is generally indicative of those from the Cainozoic. Caution still has to be exercised in the validity and interpretation of peaks at the retention time of oleanane. Bicadinanes, which can also be indicators of Tertiary age, were not present.

7. We compared the patterns of hydrocarbons over ten samples and found only small differences in the ratios of key biomarkers. All of these differences could be attributed to differences in degrees of biodegradation and weathering. For example, #13 top showed a number of indicators such as the S/S+R ratio of C29 steranes, diasterane/regular sterane ratio and sterane/hopane ratio which differed slightly from the rest of the samples. This sample had lower abundances of n-alkanes, compared to the peak of the internal standard, consistent with its greater degree of weathering.

8. We compared the pattern of biomarkers with that for the Pili Oil seep from Tonga (AGSO # 741 and #742). The patterns were similar with respect to the markers indicative of maturity and source. The Bligh Water hydrocarbons were distinguished by a lower abundance of indicators, such as 30-norhopanes, for carbonate in the source rocks and a higher abundance of compounds normally associated with oils from clastic sediments. The higher diasterane/sterane ratio of the Bligh Water hydrocarbons is particularly noticeable.

9. In summary, the Bligh water sediment cores contain petroleum hydrocarbons. In core 13, the oil appears to be increasingly weathered toward the sediment/water interface. The source of the oil appears to be a predominantly clastic sediment of marine origin and probably of Cainozoic age.

EXPERIMENTAL SECTION

Samples arrived 1700 Thursday September 9, 1993. They were chilled and were kept at 40'C prior to and between analyses. PVC tubes came with and without septa. Those without were fitted with septa prior to drilling a sampling hole through the PVC.

HEADSPACE GAS ANALYSIS

Tubes were allowed to warm to room temperature and sampling was then carried out using a gas-tight syringe in the manner we routinely use for head-space analysis of soil and water.

Initially all the samples were screened using a Carle gas analyser fitted with a Thermal Conductivity Detector (TCD). This method is normally used for coal and petroleum-derived gases. It has a detection limit in the low ppm range but allows us to quantify non-hydrocarbon components. We noted that the oxygen content of the headspace of the Bligh cores was often below that of air showing that oxygen had been consumed, presumably in the oxidation of organic matter.

Having determined that hydrocarbons were generally below the TCD detection limit we equipped our Varian 3400 GC with a Poraplot Q (porous polymer) capillary column so that the samples could be investigated using a more sensitive Flame Ionisation Detector (FID). The results of both analyses are shown in table 1.

We noted that samples with ppm range C7+ hydrocarbons showed a similar response to C7+ compounds by FID. We were able to also show that the major C7+ component was the aromatic hydrocarbon toluene. We also found low levels of hexane and heptane. We did not detect C1-C5 hydrocarbons as would normally be expected in an oil/gas seep environment. This may be due to a combination of the temperatures of the sediments, the degree of solubility of hydrocarbons at these temperatures and the relative rates of diffusion of the hydrocarbons through the sediments and the water column.

We conducted a GC-MS analysis of the headspace gas to identify the principal components by means of their mass spectra. We also confirmed the identity of the hexane and heptane by co-elution experiments with authentic standards.

Representative traces are appended to show this co-elution of hexane and to also show how a typical sample (14 bottom) was essentially devoid of C1-C5 components. Sample 13 bottom was typical of samples with a high level of toluene without much else. Gas analysis was completed 29 September 1993.

HEAVY HYDROCARBON ANALYSIS

Core tubes mostly contained a tightly compact grey-green mud. To facilitate extrusion, lengths of 15-20cm were cut from the main body of the core and the enclosed muds were transferred to a large beaker. The samples taken are listed in Table 2 and generally weighed about 1000g. One sample of an internal core was taken from the base of core 17 using a metal corer to remove mud that had not been in contact with the walls of the PVC core tube. Samples were then extracted by sonication with dichloromethane/methanol and this was then decanted and reduced to a small volume by rotary evaporation. This extracted organic matter (EOM) was first analysed by gas chromatography as if it was a "whole oil". These chromatograms appear as appendix 1.

SATURATED HYDROCARBON ANALYSIS

Subsequent to "whole oil" GC, the samples were separated into saturated, aromatic and polar/asphaltene fractions.

Approx 1.6 to 12 mg of the EOM was placed on a 129 silica gel column (pre-washed with petroleum spirit) and three fractions were collected (in 100ml round bottom flask) by eluting the column with:

- | | |
|---|-----------|
| 1. 40ml petroleum spirit | SATURATES |
| 2. 50ml petroleum spirit/dichloromethane(1:1) | AROMATICS |
| 3. 40ml chloroform/methanol (1:1) | POLARS |

Each fraction was reduced in volume on a rotary evaporator to approx 1 ml and transferred to a pre-weighed vial with dichloromethane (0.5ml 3x). The solvent was carefully removed by gentle exposure to a stream of dry nitrogen. Each fraction was weighed and labelled. Percent compositions were calculated on the basis of the original starting weights.

GC ANALYSIS

GC analysis of EOM and saturated hydrocarbon fractions was carried out using a HP 5890 Series II GC equipped with a fused silica capillary column (25m x 0.2mm) coated with cross-linked methylsilicone (HP Ultra-1). The samples, in hexane, were injected on-column at 60°C and held isothermal for 2 min using an HP autosampler. The oven was programmed to 300°C at 4'C/min with a hold period of 30 min. The carrier gas was hydrogen at a linear flow of 30cm/sec. Data was collected, integrated and manipulated using DAPA GC software.

Gas chromatograms of saturated hydrocarbon fractions are shown in the appendix,

GCMS ANALYSIS

GCMS analysis was carried out using a VG 70E instrument fitted with an HP 5790 GC and controlled by a VG 11-250 data system. The GC was equipped with an HP Ultra-1 capillary column (50m x 0.22 mm) and a retention gap of uncoated fused silica (0.5m x .53mm). The samples, in hexane were injected on-column at 50'C and the oven programmed to 150°C at 10'C/min then to 300°C at 3'C/min with a hold period of 30min. The carrier gas was hydrogen at a linear flow of 30cm/sec. The mass spectrometer was operated with a source temperature of 240'C, ionisation energy of 70eV and interface line and re-entrant at 31 0°C. In the multiple reaction monitoring (MRM) mode, the magnet current and ESA voltage were switched to sequentially sample for 26 selected parent -daughter pairs. The sampling time was 40 ms per reaction with 10 ms delay giving a total cycle time of 1.3s. The chromatograms are annotated with the diagnostic masses for parent and daughter and a five digit number indicating relative intensity. The names of specific molecules are also given where they have been identified.

GC-MS data varied little between samples and the MRM chromatograms of three samples (2 bottom, 6 bottom and 12 bottom) and the equivalent traces for the Tonga oil seep (Oil #742) are in appendix 3.

RESULTS AND DISCUSSION

Results of our initial screening of all samples are presented as a Table 1 showing the composition hydrocarbons in the headspace gas. There was considerable variability in the hydrocarbons detected. However, during biodegradation and weathering of oil in tropical waters, the light hydrocarbons are removed preferentially and the light aromatics are sufficiently soluble in water to be easily removed during prolonged contact. We were surprised that C1-C5 hydrocarbons were absent although the detection of C6 and C7 n-alkanes was suggestive of petroleum. A typical GC trace for the volatile hydrocarbons is shown in Figure 1. It seems probable that the original "pentane" anomaly may have been due to the same process of weathering except that differences in the way the samples were analysed or a seasonal difference cause the C5 to be detected more easily in the gas phase in the earlier work. Samples with moderately high levels were then selected for extraction.

Extactable organic matter was obtained from ten samples as indicated in Table 2. Gas chromatograms of the total extracts were dominated by peaks with a marked carbon number predominance, initially suggesting material of biogenic origin. Waxy n-alkanes in terrestrial muds, for example have a pronounced odd/even character. We carried our liquid chromatography to isolate the saturated, aromatic and polar compounds into separate fractions and this showed that the carbon chain preference was confined to compounds in the polar fraction and was probably due to waxy alcohols of biological origin. We did not analyse these further.

The saturated hydrocarbons themselves showed a pattern of n-alkanes without carbon number preference and these were predominantly in the range C21-C31 although we could detect C10-C20 compounds as well. This picture, exemplified by the trace shown in Figure 2 is consistent with an origin from mature petroleum. As well as the n-alkanes, we identified pristane, phytane, steranes and hopanes, all of which had similar distributions in all the cores.

The pattern of saturated hydrocarbons in the Bligh Water sediments closely resembled that seen in weathered petroleum which has been stranded on beaches and rocks in tropical Northern Australia (Summons et al., 1993). The lighter n-alkanes are increasingly removed as a function of increasing volatility, generally leaving an envelope starting from about n-C20. The oil has also probably been affected by microbial attack but this has not been severe enough to remove all the

n-alkanes and isoprenoids. A fixed aliquot of standard hydrocarbon (100ng of iso-C22) was added to the samples prior to GC. Using the peak of this compound as a benchmark, one can estimate the relative abundance of hydrocarbons remaining after weathering. Increasing height of the standard relative to the sample peaks indicates a low amount of n-alkanes and, if necessary, this could be calculated back using the original sample weight to obtain values for individual n-alkanes in ppb.

We used the distribution of biomarker hydrocarbons detected by GC-MS to obtain information about the source type, maturity and geological age of the source rocks. We based our interpretations on the guidelines used by Peters and Moldowan (1993) and the results of our own research.

Diagnostic hydrocarbons evident in the GC-MS traces shown in Figure 4a for the saturates from core #2 bottom include the C30 desmethylsterane (24-n-propylcholestane in 414-->217) and a mixture of C30 4-methylsteranes (in 414-->231) including dinosterane. Both of these compound classes are related to sterols confined to marine algae, in our experience, restricted to sediments with inputs of marine organic matter (Moldowan *et al.*, 1985, 1990). Moreover, the presence of dinosterane is restricted to sediments and oils of Mesozoic or younger age (Summons *et al.*, 1987). Other evidence of marine sedimentation of the source rock in Figure 4a is in the high abundance of C27 steranes relative to C29 steranes. C27 steranes are the most prominent biomarkers in the sample and the high ratio of steranes to hopanes (Fig. 4b) is another feature consistent with a marine source. Finally, the hopanes in Fig. 4b show the most abundant C28 compound is 28,30-bisnorhopane and this is typical of marine oils being abundant in the Santa Maria Basin oils of California and the Jurassic oils of the North Sea.

Features of the biomarker distributions strongly suggests that the oil was derived from a predominantly clastic source rock. Diasteranes, otherwise known as Ba-rearranged steranes are in high abundance compared to the regular aaa and aBB steranes. Diasteranes are produced by rearrangement of ordinary steranes on acidic catalytic surfaces of clay and hence they are generally absent from sediments of predominantly carbonate lithology as shown by empirical observation of low diasterane abundances in carbonate source rocks and their derived oils (e.g. Summons and Powell, 1987; Subroto *et al.*, 1992).

We also found similar patterns of triterpanes in all samples. There were low levels of diahopanes, neohopanes and 29,30-bisnorhopane in each sample and oleanane was detected in every one. There were also low levels of 2a(Me)-hopane and C30 hopane and C29 hopane peaks were roughly equivalent in abundance except that internal core #17 has a higher C29/C30 hopane ratio than the others. This could reflect partial removal of C30 through biodegradation. The samples had similar ratios of Ts/Tm and C29 norneohopane/C29 aB-hopane and these parameters reflect the similarity in maturity of the samples. Only traces of 25-norhopanes could be detected. If present, these compounds would have signified a highly biodegraded oil (Alexander et al., 1984; Volkman et al., 1983). Thus the hydrocarbons probably originate from a reservoir horizon that is not in contact with meteoric water.

Diasteranes are not very abundant and panes are abundant in the Pili seep oil (#741, #742). This oil affords a comparative example of one with a major carbonate contribution to the source rock, but one where the composition of the organic matter is roughly equivalent.

The isomer distributions of the steranes in some samples shows a slight preference for 20R over 20S isomers ($20S/20S+20R=0.55$ and the end-point of approx. 1.1 for this ratio has not been reached). This suggests that the samples could be in the early half of the "normal" window for oil generation. This ratio is, however, sensitive to the effects of biodegradation with the 20R being preferentially attacked (and Moldowan, 1979). The high ratio for parameter 2 in cores #13 top and #25 top may be due to the effects of increased degradation. The ratios of Ts/Tm and S/S+R C31 triterpanes are consistent with the hydrocarbons being derived from a mature oil and certainly enable the sample to be distinguished from samples of biogenic hydrocarbons. In fact, compounds such BB-hopanes which are characteristic of immature sediments or biogenic samples were not undetected in the Bligh Water sediments.

We did not detect bicadinanes or botryococcane in any of the samples. These compounds can be prominent in the Tertiary oils of South East Asia and are present in many of the beach-stranded oils which have been transported to Australia on ocean currents. The appearance of the hydrocarbon patterns and the similarity of the signature in all the samples is that of a weathered seep oil. There are no inconsistencies or anomalies in the ratios of the peaks, other than some minor ones attributable to differential weathering. Anomalous ratios for different

biomarkers could suggest pollution from a refined petroleum comprising oils from multiple sources and maturities. For these reasons we believe that the hydrocarbons probably come from a single local source.

The dinosteranes constrain the age of the Bligh Water sediment hydrocarbons to Triassic age or younger. Oleanane, almost certainly constrains the age to Late Cretaceous or Tertiary. We believe the peak we have denoted as oleanane is that compound but there are reports in interferences in the detection. Also, there are conflicting literature reports about the earliest appearance of this compound in sediments and oil (See Peters and Moldowan, 1993). As a result, we must remain somewhat cautious in the interpretation of this compound.

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TABLE 1

Sample No.	Depth (m)	• a GC-TCD DETECTION					•• b GC-FID DETECTION			
		O2	N2	CO2	C1-C6	C7+	C1-C5	Hexane	Heptane	Toluene
		mole %					ppb			
2 BOTTOM	383	22.6	77.4	nd	nd	nd	nd	0.2	0.4	60.8
2 TOP	283	2.9	97.0	nd	nd	0.1	nd	0.2	0.6	0.7
4 BOTTOM	378	5.2	94.5	nd	nd	0.3	nd	nd	nd	137.9
4 TOP	378	22.6	77.3	nd	nd	nd	nd	0.3	0.5	0.9
5	439	22.3	77.3	nd	nd	0.3	nd	2.9	5.4	183.8
6 BOTTOM	475	8.0	91.6	nd	nd	0.4	nd	0.2	0.5	63.1
6 TOP	475	19.6	80.4	nd	nd	nd	nd	0.1	0.4	0.9
12 BOTTOM	315	4.3	95.6	nd	nd	0.1	nd	0.1	0.1	0.3
12 TOP	315	22.4	77.4	0.1	nd	nd	nd	1.3	1.9	0.3
13 BOTTOM	320	2.7	96.4	nd	nd	0.4	nd	nd	0.3	278.2
13 TOP	320	6.3	93.7	nd	nd	nd	nd	0.5	0.7	1.4
14 BOTTOM	329	1.7	98.3	nd	nd	nd	nd	1.7	8.1	5.9
14 TOP	329	12.2	87.8	nd	nd	nd	nd	nd	nd	0.3
15 BOTTOM	317	3.1	96.8	nd	nd	0.1	nd	nd	0.2	14.5
15 TOP	317	10.9	88.9	0.1	nd	nd	nd	nd	nd	0.1
17 BOTTOM	366	22.6	77.4	nd	nd	nd	nd	1.0	0.4	11.2
17 TOP	366	2.0	98.0	nd	nd	nd	nd	nd	nd	0.1
18 BOTTOM	355	6.4	93.5	nd	nd	0.1	nd	nd	nd	45.3
18 TOP	355	22.4	77.6	nd	nd	nd	nd	nd	nd	0.2
20	366	15.6	84.2	0.1	nd	0.1	nd	nd	nd	0.6
21(A)	366	7.7	92.3	nd	nd	nd	nd	nd	nd	3.9
21(B)	366	20.0	79.8	0.2	nd	nd	nd	nd	nd	nd
21 (B) **	366	22.6	77.3	nd	nd	nd	nd	0.1	nd	1.1
21C BOTTOM	366	1.8	97.8	nd	nd	0.4	nd	1.6	6.3	333.0
21C TOP	366	22.6	77.9	0.2	nd	nd	nd	0.4	0.7	0.8
22 BOTTOM	402	2.1	97.9	nd	nd	nd	nd	nd	nd	7.2
22 TOP	402	3.4	96.3	nd	nd	nd	nd	0.1	0.3	1.0
23 BOTTOM	399	3.0	97.0	nd	nd	nd	nd	nd	nd	0.9
23 TOP	399	22.3	77.5	0.2	nd	nd	nd	0.4	0.8	0.5
24 BOTTOM	470	6.1	93.9	nd	nd	nd	nd	nd	nd	30.2
24 TOP	470	6.5	93.5	nd	nd	nd	nd	0.1	0.2	0.4
25 BOTTOM	350	10.5	89.4	nd	nd	0.1	nd	0.3	0.6	21.8
25 TOP	350	2.0	98.0	nd	nd	nd	nd	0.8	1.0	0.7
Ltka BOTTOM	-	22.7	77.3	nd	nd	nd	nd	nd	nd	6.9
Ltka TOP	-	3.2	96.8	nd	nd	nd	nd	0.3	0.7	0.6

nd NOT DETECTED

• GAS WAS WITHDRAWN FROM HEAD SPACE OVER FROZEN SEDIMENT (GC-TCD)

** SEDIMENT ALLOWED TO THAW TO ROOM TEMPERATURE THEN HEATED

TO 60 deg C FOR 4 HOURS BEFORE SAMPLING HEAD SPACE.

*** CONCENTRATION OF INDIVIDUAL GASEOUS HYDROCARBONS SAMPLED AT ROOM TEMPERATURE

a DETECTION LIMIT = sub ppm

b DETECTION LIMIT = 0.1 ppb (Note ppb = 1/1,000,000,000)

Table 1.

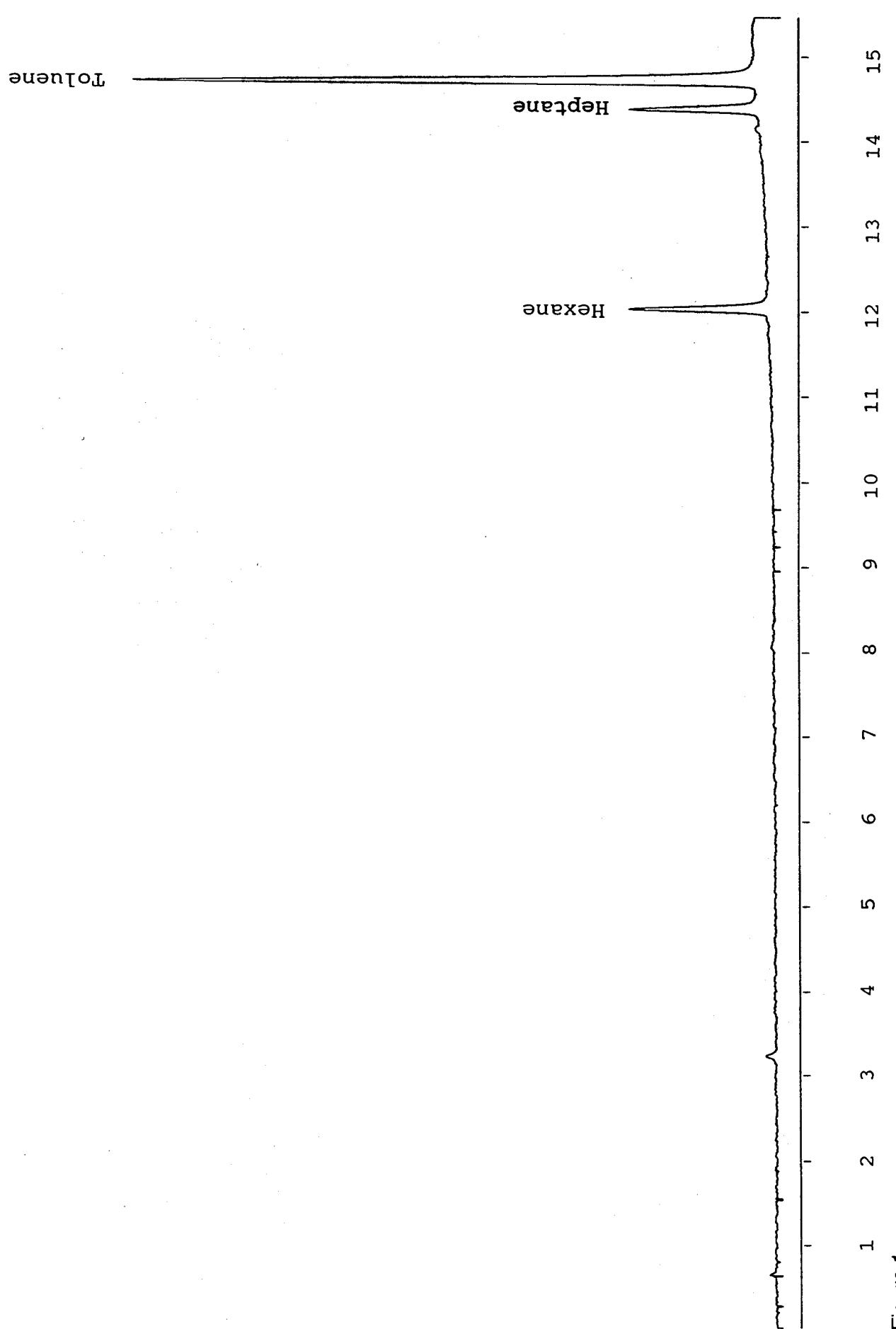
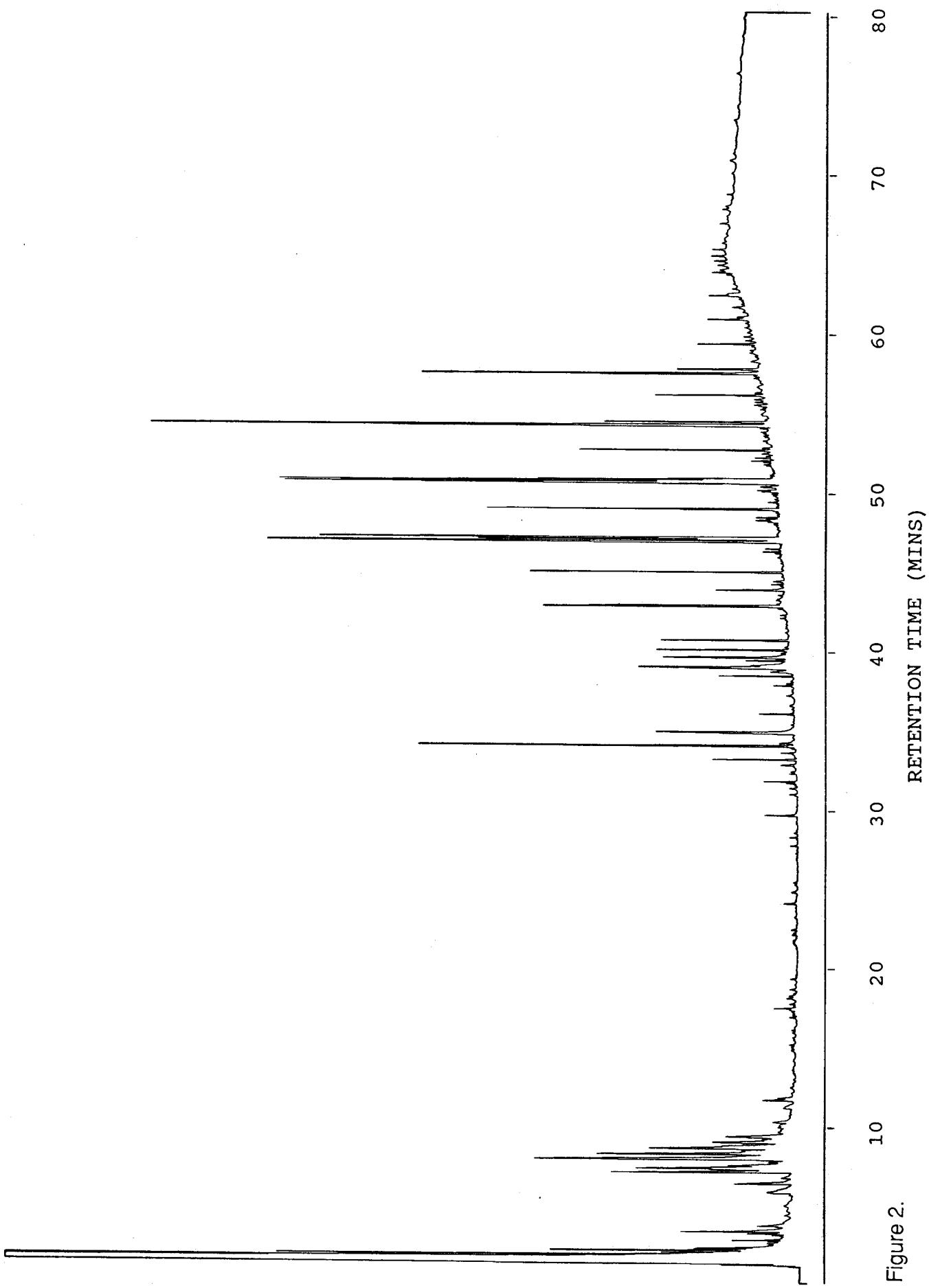


Table 2: Bligh Water sediment extract data

Core No.	Wt extracted (g)	EOM (mg)	ppm EOM	Saturates (mg)	Aromatics (mg)	ONS Comp. (mg)
2 Bottom	958	4.0	4.2	1.3	0.1	2.4
6 Bottom	1023	12.0	11.7	1.2	0.2	7.7
12 Bottom	664	3.1	4.7	0.9	0.2	1.9
13 Bottom	1025	2.9	2.8	0.7	0.1	1.7
13 Top	523	1.5	2.9	0.6	0.1	0.6
17 Bottom	631	2.6	4.1	0.6	0.1	1.7
21 Bottom	994	3.3	3.3	1.2	0.2	1.7
23 Top	1438	2.3	0.7	0.4	0.2	1.4
25 Top	745	2.3	3.1	0.9	0.3	0.9
Internal Core 17 Bottom	150	1.6	10.7	0.3	0.1	1.0

2 BOTTOM TOTAL EXTRACT



2 BOTTOM SATURATES

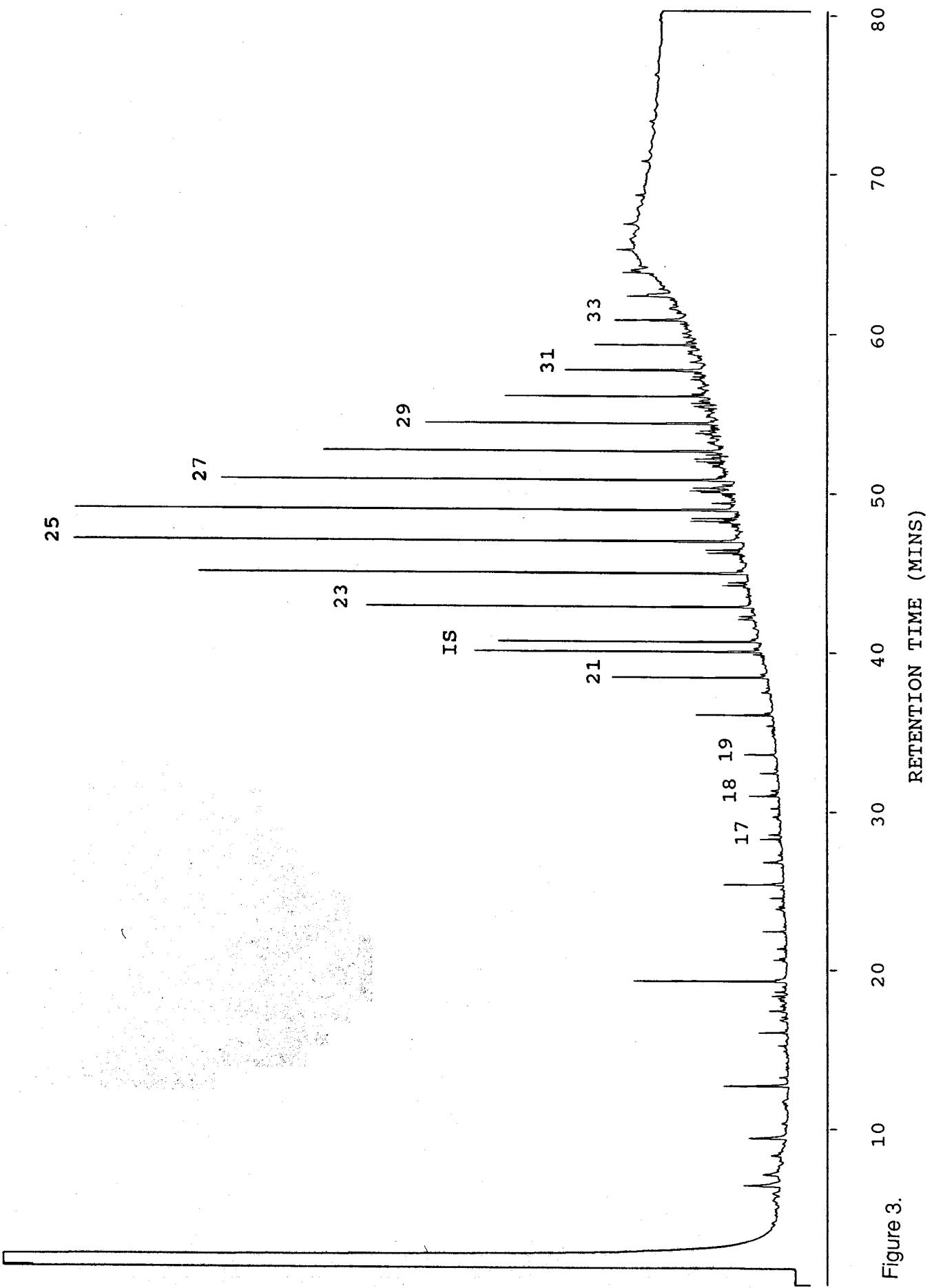


Figure 3.

Sterane GCMS fingerprint : Bligh Water sediment #2 (bottom).

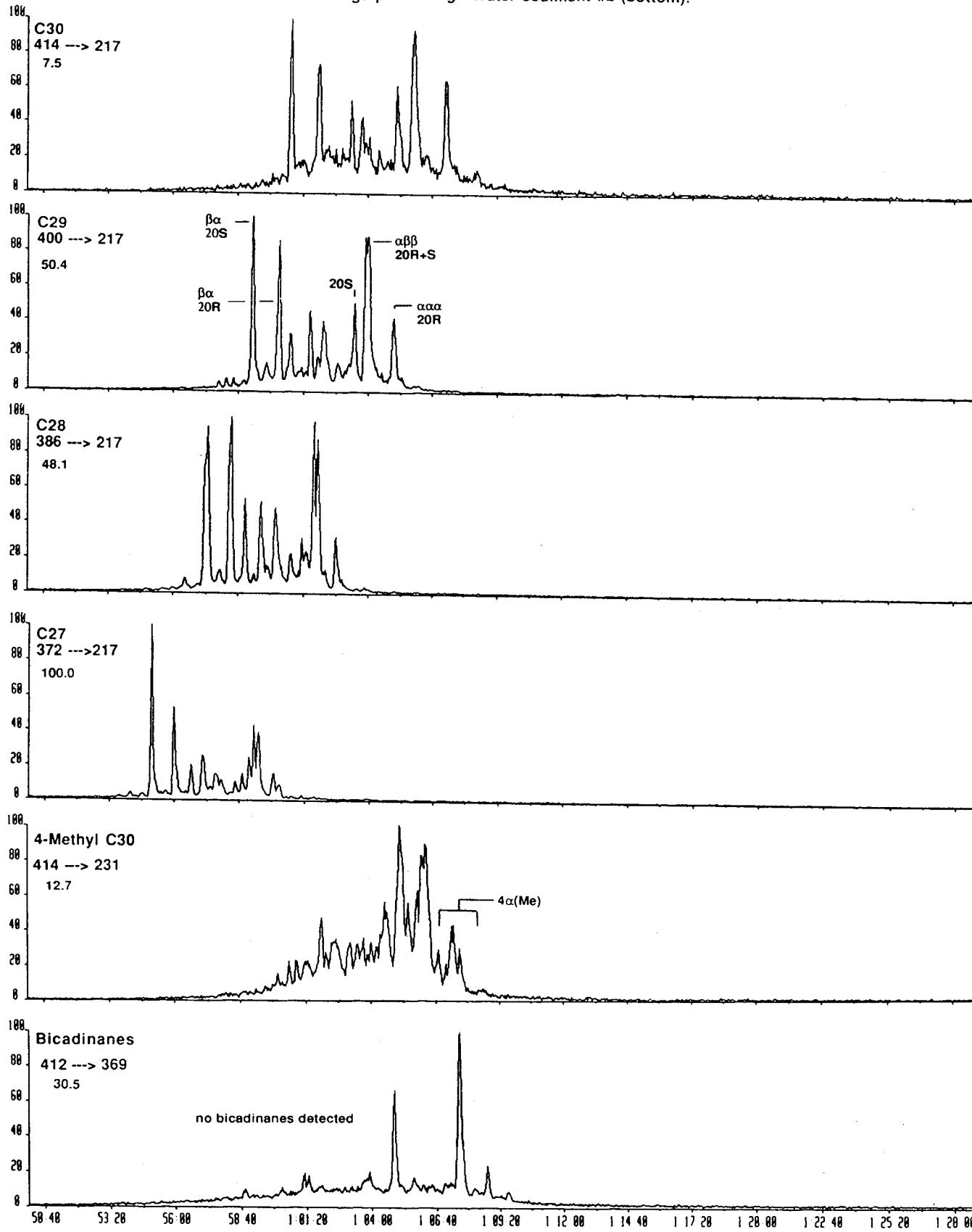


Figure 4a.

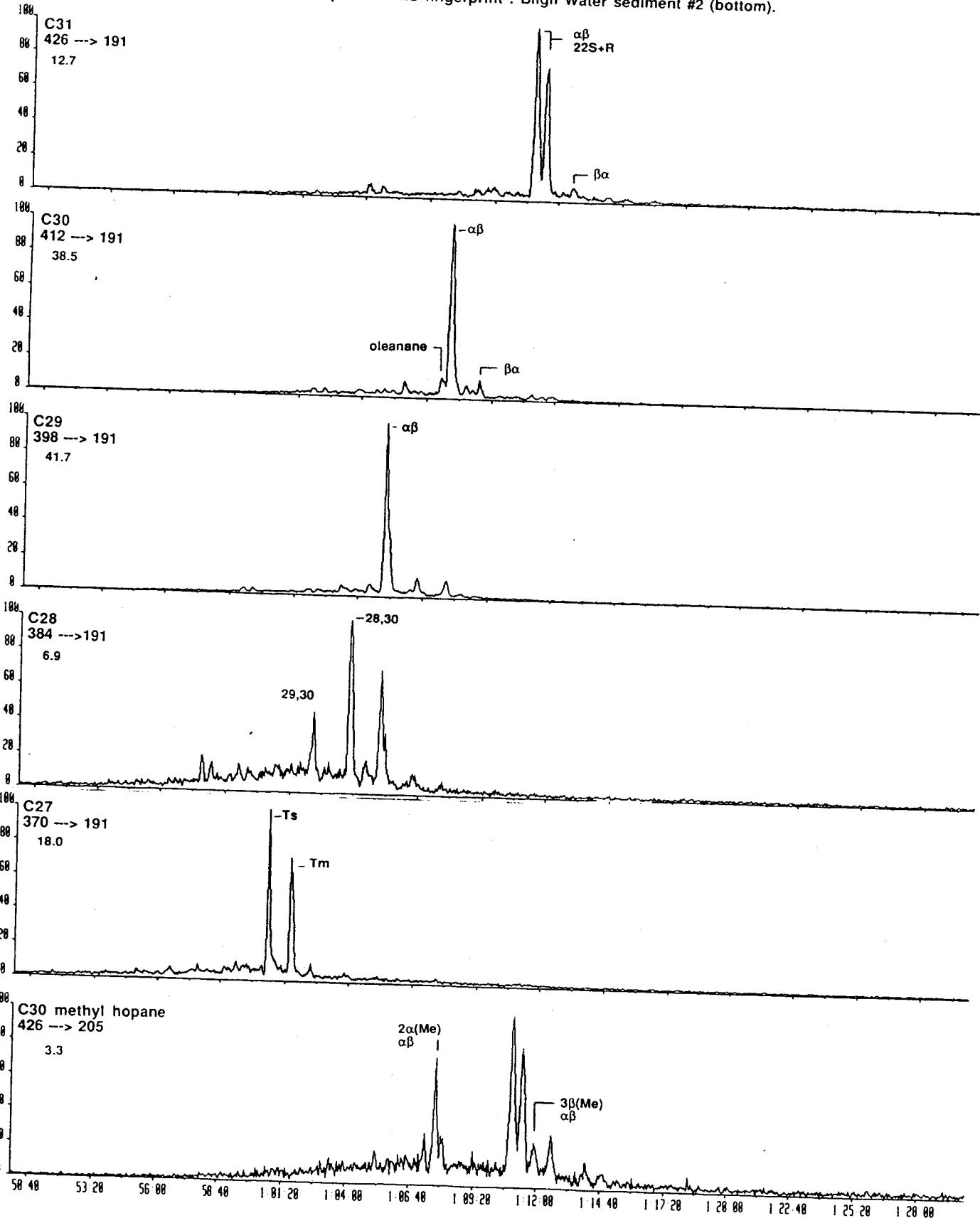


Figure 4b.

Table 3.

SOPAC Samples : GCMS data

	Tonga Seep	Tonga Seep	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	Bligh Water	
ID Trace	AGSO No 741 2OC26A-6	AGSO No 742 2OC26A-7	2 BOTTOM 3OC20A-27	6 BOTTOM 2OC26A-8	12 BOTTOM 2OC26A-1	13 BOTTOM 2OC26A-9	17 BOTTOM 3OC20A-28	CORE 2OC26A-2	21 BOTTOM 2OC26A-19	13 TOP 2OC26A-4	23 Top 3OC24A-24	25 TOP 3OC20A-22	
SOURCE-SPECIFIC BIOMARKER PARAMETERS													
27:28:29 reg steranes	a ****	1 : .53 : .53	1 : .67 : .83	1 : 1.08 : 1.41	1 : .94 : 1.4	1 : .94 : 1.4	1 : 1.1 : 1.79	1 : .92 : 1.33	1 : .85 : 1.38	1 : .84 : 1.08	1 : .94 : 1.14	1 : .71 : 1.12	1 : 1.17 : 1.87
27:28:29 dia steranes	b	1 : .76 : .59	1 : .77 : .7	1 : .98 : .73	1 : .7 : .45	1 : .87 : .41	1 : .74 : .50	1 : .99 : .79	1 : .95 : .82	1 : 1.03 : .65	1 : .94 : .64	1 : 1.06 : .74	1 : .61 : .40
27:29 reg steranes	c	2.27	1.48	0.57	0.48	0.48	0.37	0.69	0.79	0.69	1.09	0.94	0.42
27:29 dia steranes	d	1.61	1.14	1.16	2.12	2.38	1.66	1.01	1.19	1.46	1.24	1.16	2.21
28:29 reg steranes	e	1.15	1.10	0.62	0.60	0.54	0.59	0.61	0.69	0.45	0.86	0.70	0.67
28:29 dia steranes	f	1.38	0.97	1.31	1.41	1.91	1.35	1.22	1.02	1.71	1.28	1.40	1.25
30:29 reg steranes	g	0.16	0.14	0.24	0.27	0.30	0.35	0.22	0.19	0.31	0.27	0.23	0.29
hop/ster	h	0.85	0.86	1.24	1.00	0.89	0.96	0.94	0.82	0.93	0.73	0.93	0.92
31/30 hopanes	i	0.68	0.48	0.57	0.65	0.80	0.59	0.50	0.61	0.54	0.80	0.76	0.41
29/30 hopanes	j	1.36	1.60	0.96	1.10	1.12	1.18	1.06	2.04	1.15	1.68	0.91	0.89
29bis/hop	k	0.11	0.12	0.05	0.05	0.05	0.04	0.07	0.13	0.06	0.10	0.10	0.03
28bis/hop	l	0.28	0.34	0.15	0.17	0.14	0.23	0.17	0.26	0.19	0.25	0.30	0.11
Ts/ hopane	m	0.54	0.41	0.28	0.31	0.33	0.32	0.33	0.45	0.37	0.71	0.36	0.22
diahop/hop	n	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2aMe hop/abC30 hop		0.10	0.09	0.05	0.06	0.07	0.06	0.05	0.13	0.07	0.07	0.00	0.08
30Me hop/abC30 hop		0.01	0.01	0.02	0.00	0.03	0.01	0.01	0.01	0.03	0.04	0.00	0.02
oleanane/17a(H)-hopane		0.11	0.11	0.08	0.08	0.14	0.11	0.10	0.06	0.16	0.09	0.13	0.06
MATURITY-SPECIFIC BIOMARKER PARAMETERS													
S/S+R C27dia ster	1 ****	0.55	0.56	0.61	0.59	0.57	0.65	0.63	0.55	0.61	0.62	0.60	0.60
S/S+R C29 reg ster	2	0.60	0.53	0.49	0.55	0.56	0.52	0.53	0.58	0.55	0.65	0.56	0.61
$\alpha\beta/\alpha\beta+\text{aaa}$ ster	3	0.56	0.56	0.64	0.62	0.58	0.64	0.62	0.61	0.58	0.62	0.62	0.61
dia/reg c29 ster	4	0.46	0.33	2.03	1.85	1.85	1.71	1.98	1.73	2.05	2.10	1.73	1.14
Ts/Tm	5	0.57	0.46	1.15	1.18	1.02	1.02	1.11	0.81	0.93	1.16	0.98	0.91
S/S+R C31 triterp	6	0.56	0.56	0.59	0.59	0.54	0.59	0.57	0.58	0.56	0.57	0.56	0.52
$\beta\alpha/\beta\alpha+\text{aa}$ triterp	7	0.06	0.05	0.05	0.05	0.10	0.05	0.05	0.05	0.02	0.06	0.07	0.06

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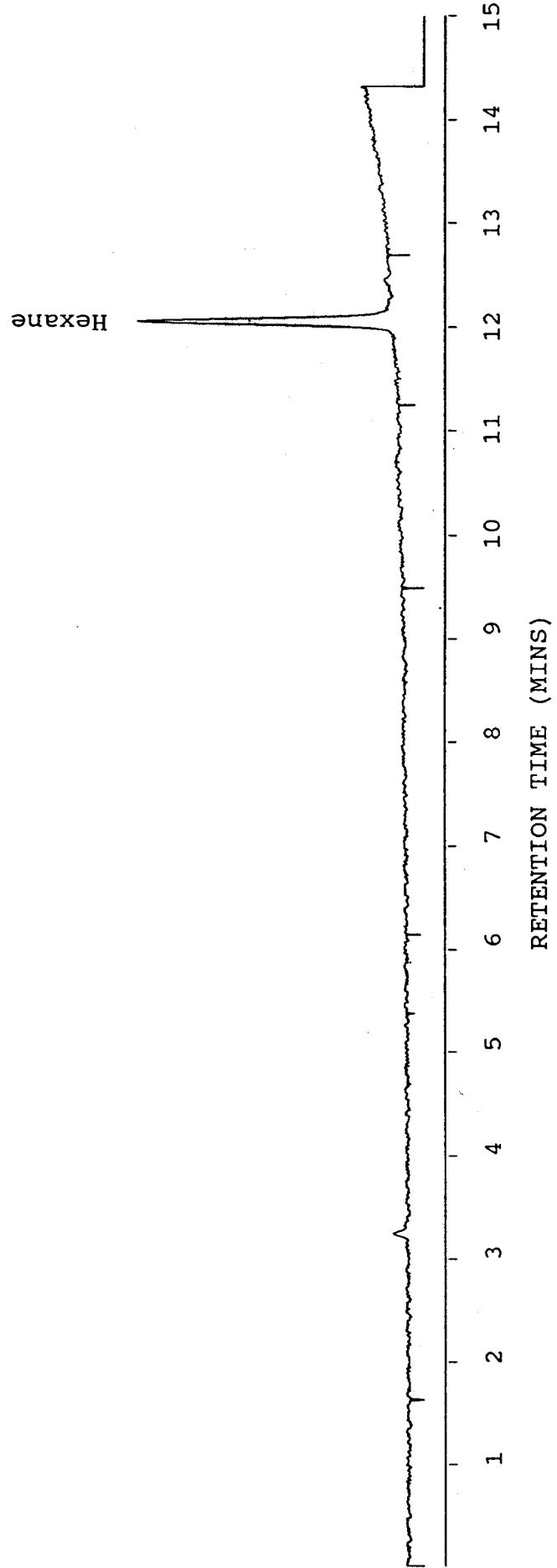
Table 4.

Key to maturity-dependent biomarker parameters

	Biomarker Parameter	Specificity	m/z	Reference
	<i>Steranes</i>			
1	C ₂₇ 13β(H)17α(H) 20S diasterane / C ₂₇ 13β(H)17α(H) 20R + 20S diasterane	Maturity: immature to early oil generation, 0 - 0.55	259	Ensminger <i>et al.</i> , 1978
2	C ₂₉ 5α(H)14α(H)17α(H) 20S sterane / C ₂₉ 5α(H)14α(H)17α(H) 20R + 20S sterane	Maturity: immature to early oil generation, 0 - 0.55. Biodegradation: R is consumed in preference to S	217	Mackenzie <i>et al.</i> , 1980; Seifert & Moldowan, 1986
3	C ₂₉ 5α(H)14β(H)17β(H) 20R sterane / C ₂₉ 5α(H)14β(H)17β(H) 20R + C ₂₉ 5α(H)14α(H)17α(H) 20R steranes	Maturity: immature to optimum hydrocarbon generation, variable - 0.7.	217	Mackenzie <i>et al.</i> , 1980; Seifert & Moldowan, 1986
4	C ₂₉ 13β(H)17α(H) diasteranes (20R + 20S) / C ₂₉ 5α(H) steranes (20R + 20S)	Maturity: early mature to late oil/early gas generation, 0 - 100. Biodegradation: 20R is consumed in preference to 20S. Source: high for clastics, low for carbonates.	217, 259	Alexander <i>et al.</i> , 1983
	<i>Triterpanes</i>			
5	C ₂₇ 18α(H)-22,29,30-trisnorhopane (Ts) / C ₂₇ 17α(H)-22,29,30-trisnorhopane (Tm)	Maturity: immature to post mature, 0-100. Source: Tm derived from bacteria.	191	Seifert & Moldowan, 1978
6	C ₃₁ 17α(H)21β(H) 22S homohopane / C ₃₁ 17α(H)21β(H) 22S + 22R homohopane	Maturity: immature to onset of oil generation, 0 - 0.6.	191	Mackenzie <i>et al.</i> , 1980; Seifert & Moldowan, 1986; Zumberge, 1987
7	C ₃₀ 17β(H)21α(H) moretane / C ₃₀ 17β(H)21α(H) moretane + 17α(H)21β(H) hopane	Maturity: immature to early oil generation, 0 - 0.5.	191	Seifert & Moldowan, 1980

Key to source-specific biomarker parameters

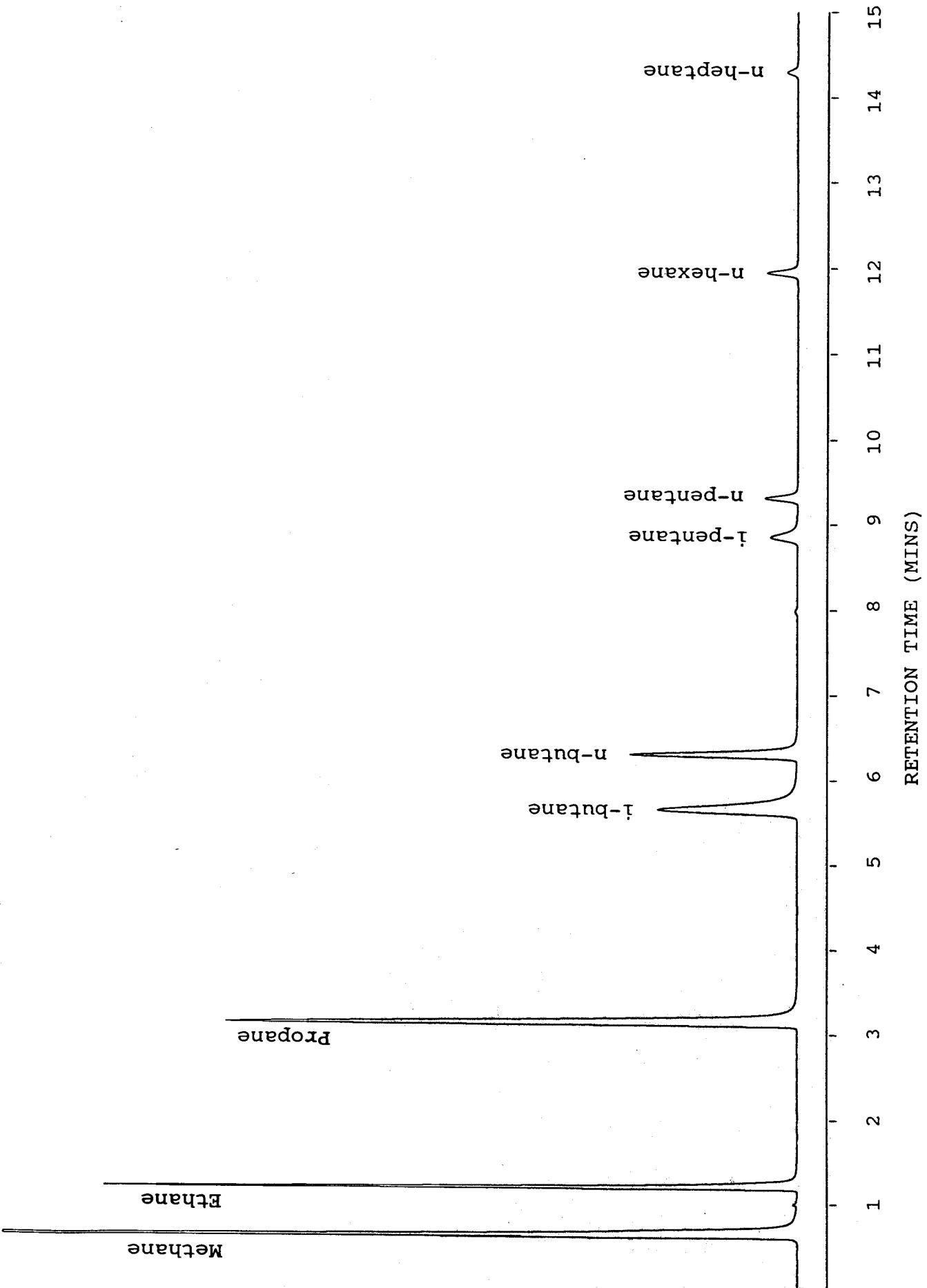
	Biomarker Parameter	Specificity	Diagnostic Ion	References
a	<i>Steranes</i> C ₂₇ : C ₂₈ : C ₂₉ 5α(H)14α(H)17α(H) 20R steranes	Source: C ₂₇ and C ₂₈ dominant algal input. C ₂₉ dominant indicative of terrestrial plants.	217	Mackenzie <i>et al.</i> , 1983; Moldowan <i>et al.</i> , 1985
b	C ₂₇ : C ₂₈ : C ₂₉ 13β(H)17α(H) 20R diasteranes	As above	259	
c	C ₂₇ 5α(H)14α(H)17α(H) 20R sterane / C ₂₉ 5α(H)14α(H)17α(H) 20R sterane	Source: high values algal material, low values (< 1) terrestrial plants.	217	Seifert <i>et al.</i> , 1983
d	C ₂₇ 13β(H)17α(H) 20R diasterane / C ₂₉ 13β(H)17α(H) 20R diasterane	As above	259	
e	C ₂₈ 5α(H)14α(H)17α(H) 20R sterane / C ₂₉ 5α(H)14α(H)17α(H) 20R sterane	As above	217	
f	C ₂₈ 13β(H)17α(H) 20R diasterane / C ₂₉ 13β(H)17α(H) 20R diasterane	As above	258	
g	C ₃₀ 24-n-propyl-sterane (ααα 20R) / C ₂₉ sterane (ααα 20R)	Source: C ₃₀ sterane present in marine algal organic matter.	217, 231	
<i>Triterpanes</i>				
h	C ₃₀ 17α(H)21β(H) hopane / C ₂₉ 5α(H) steranes 20R + 20S	Source: high for prokaryote, low (< 1) for eukaryote.	191, 217	
i	C ₃₁ 17α(H)21β(H) hopanes 22R + 22S / C ₃₀ 17α(H)21β(H) hopane	Source: high values anoxic during deposition & early diagenesis.	191	
j	C ₂₉ 17α(H)21β(H) hopane / C ₃₀ 17α(H)21β(H) hopane	Source: values > 1 anoxic, marine, carbonate environment.	191	
k	C ₂₈ 29, 30-bisnorhopane / C ₃₀ 17α(H)21β(H) hopane	Source	191	
l	C ₂₈ 28, 30-bisnorhopane / C ₃₀ 17α(H)21β(H) hopane	Source	191	
m	C ₂₇ 18α(H)-22,29,30-trisnorhopane (Ts) / C ₃₀ 17α(H)21β(H) hopane	Source; Maturity.	191	Volkman <i>et al.</i> , 1983
n	C ₃₀ 17α(H)21β(H) diahopane / C ₃₀ 17α(H)21β(H) hopane	Source	191	

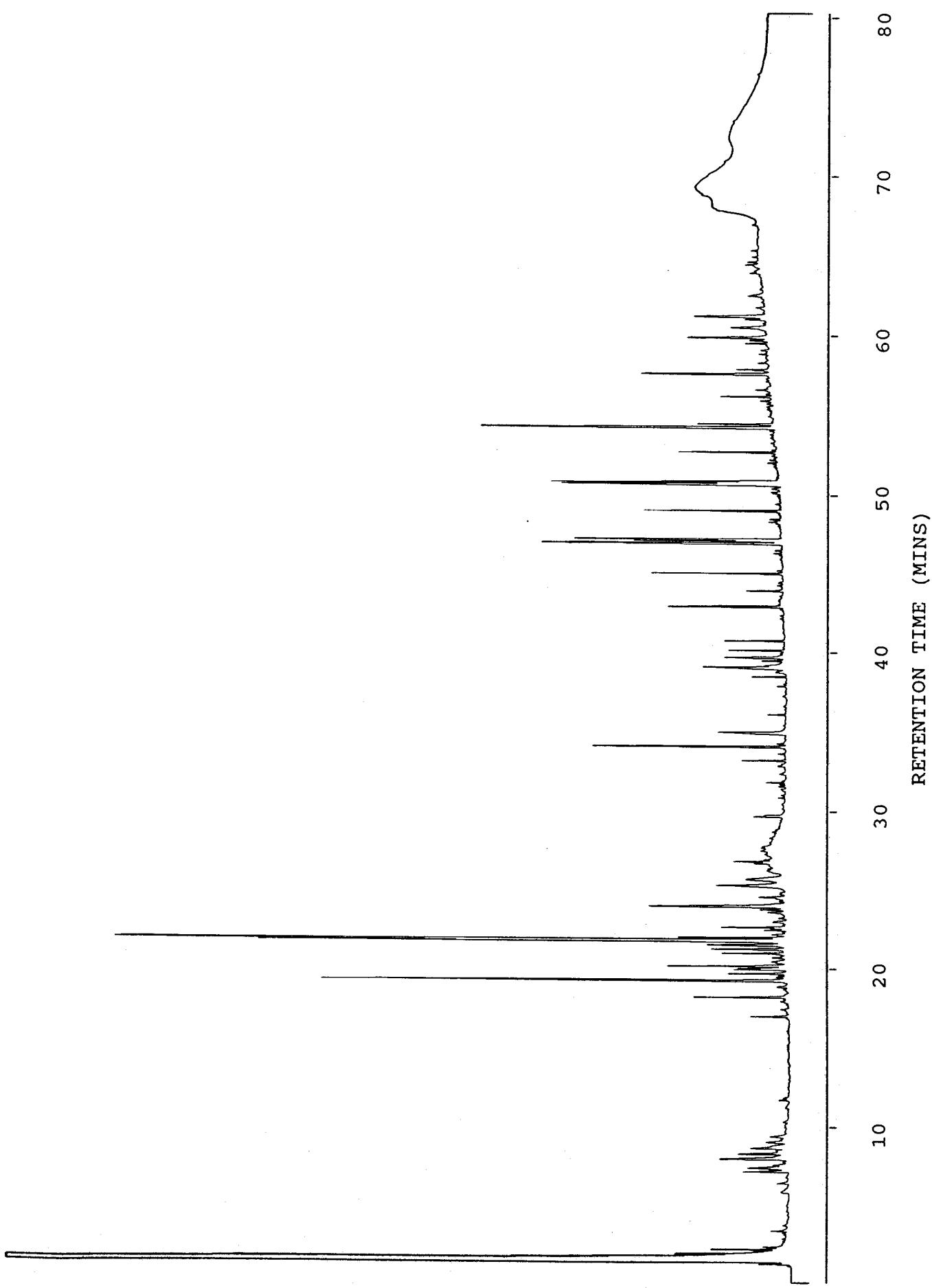


Toluene

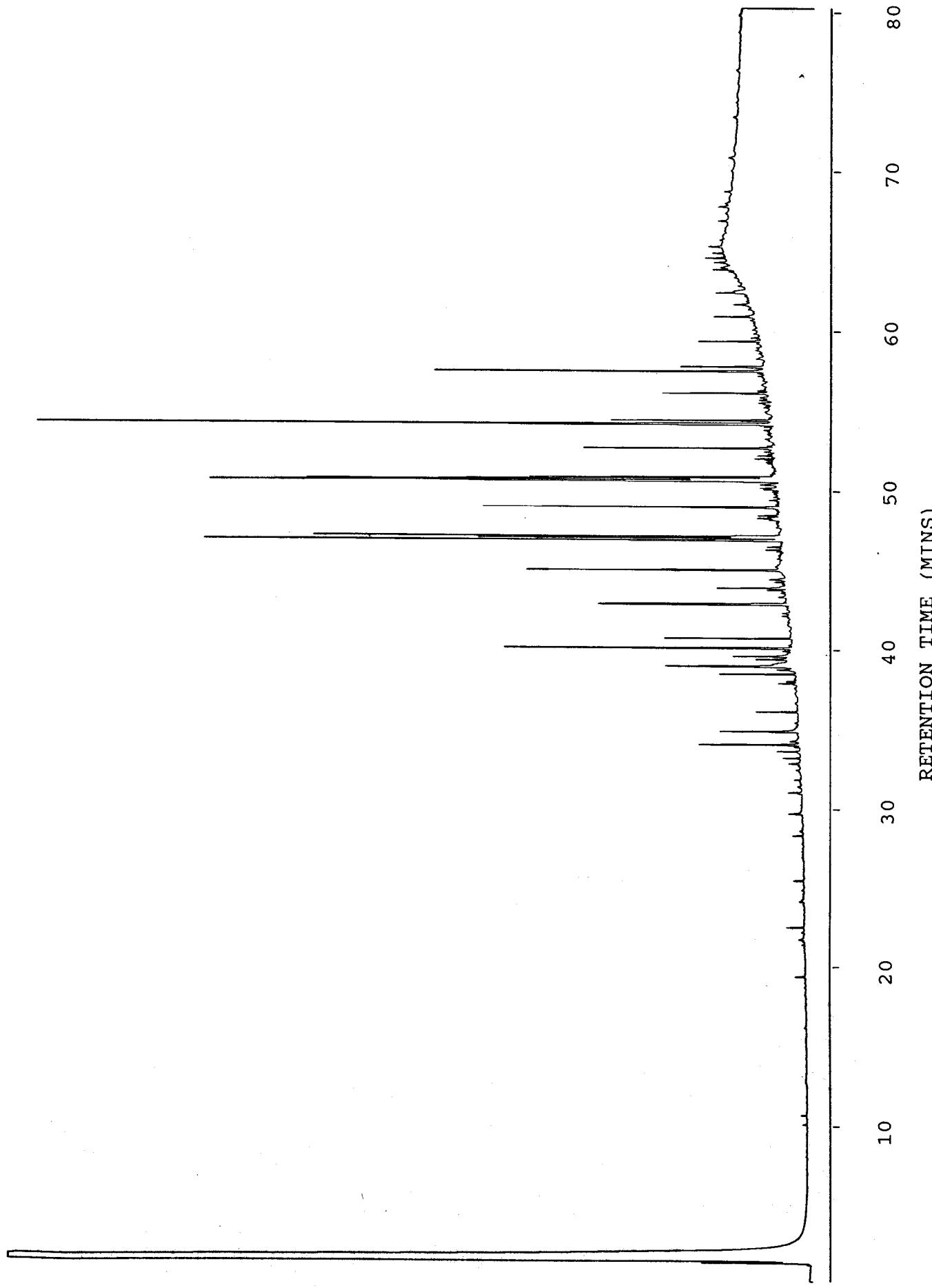
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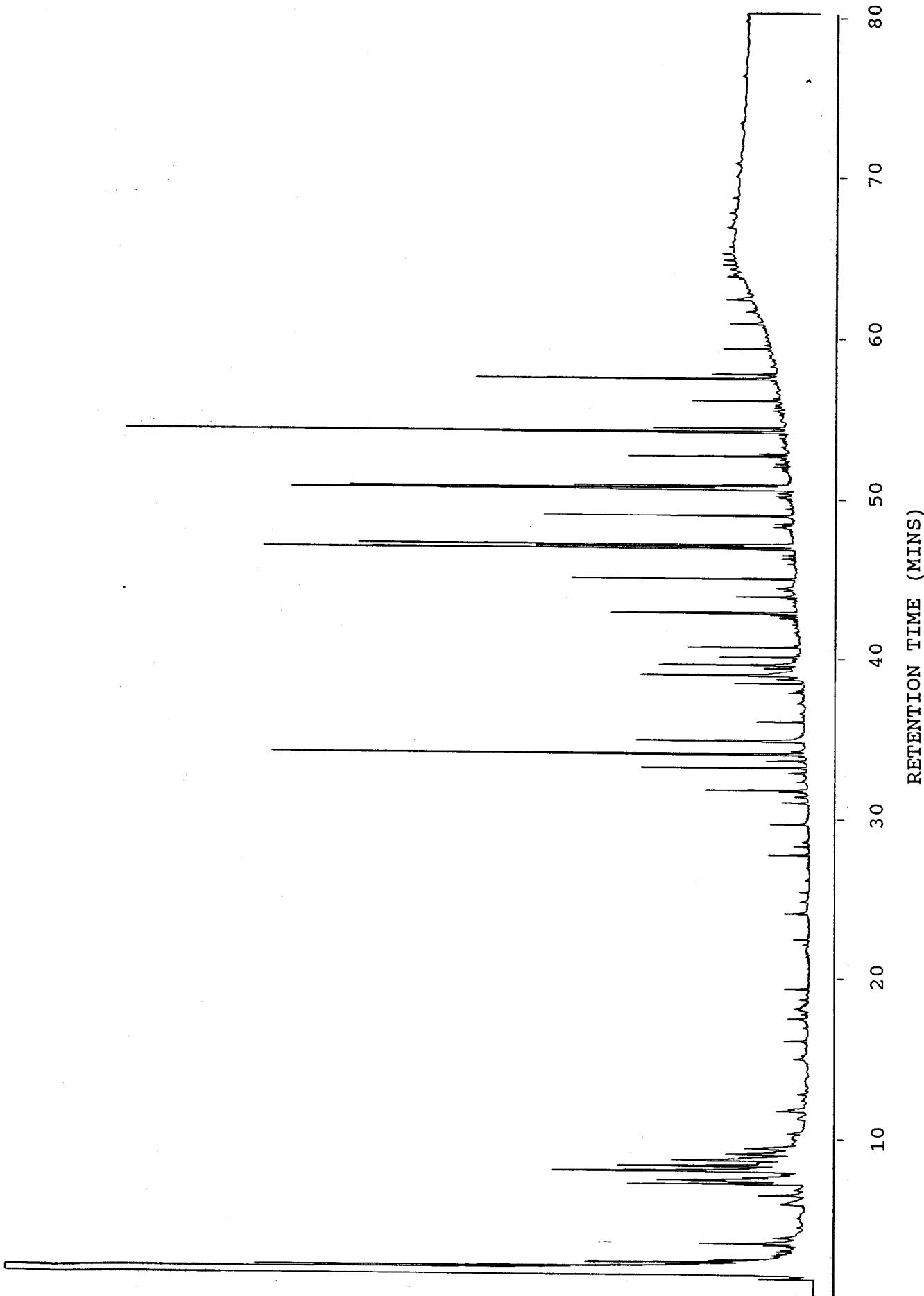
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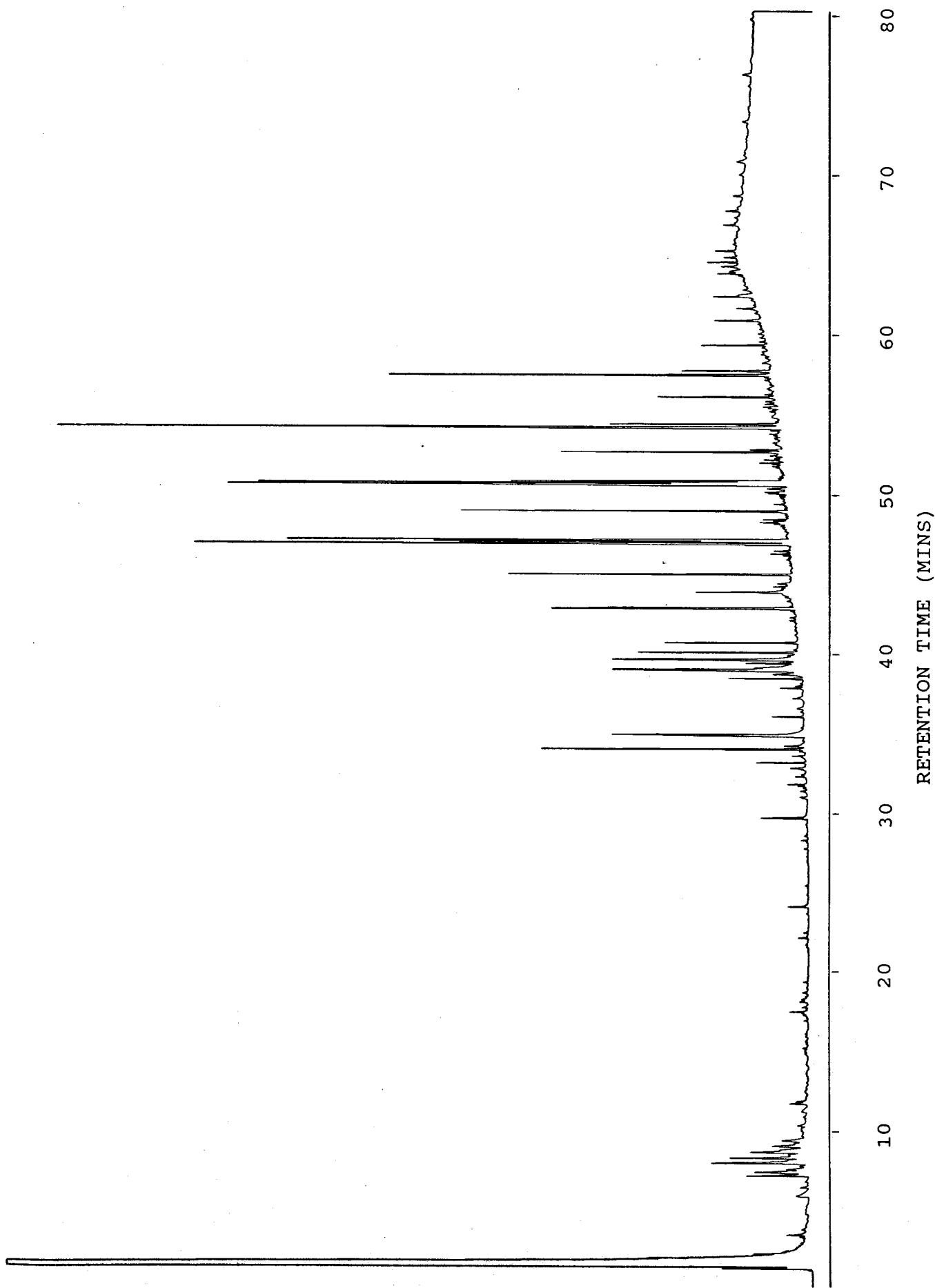


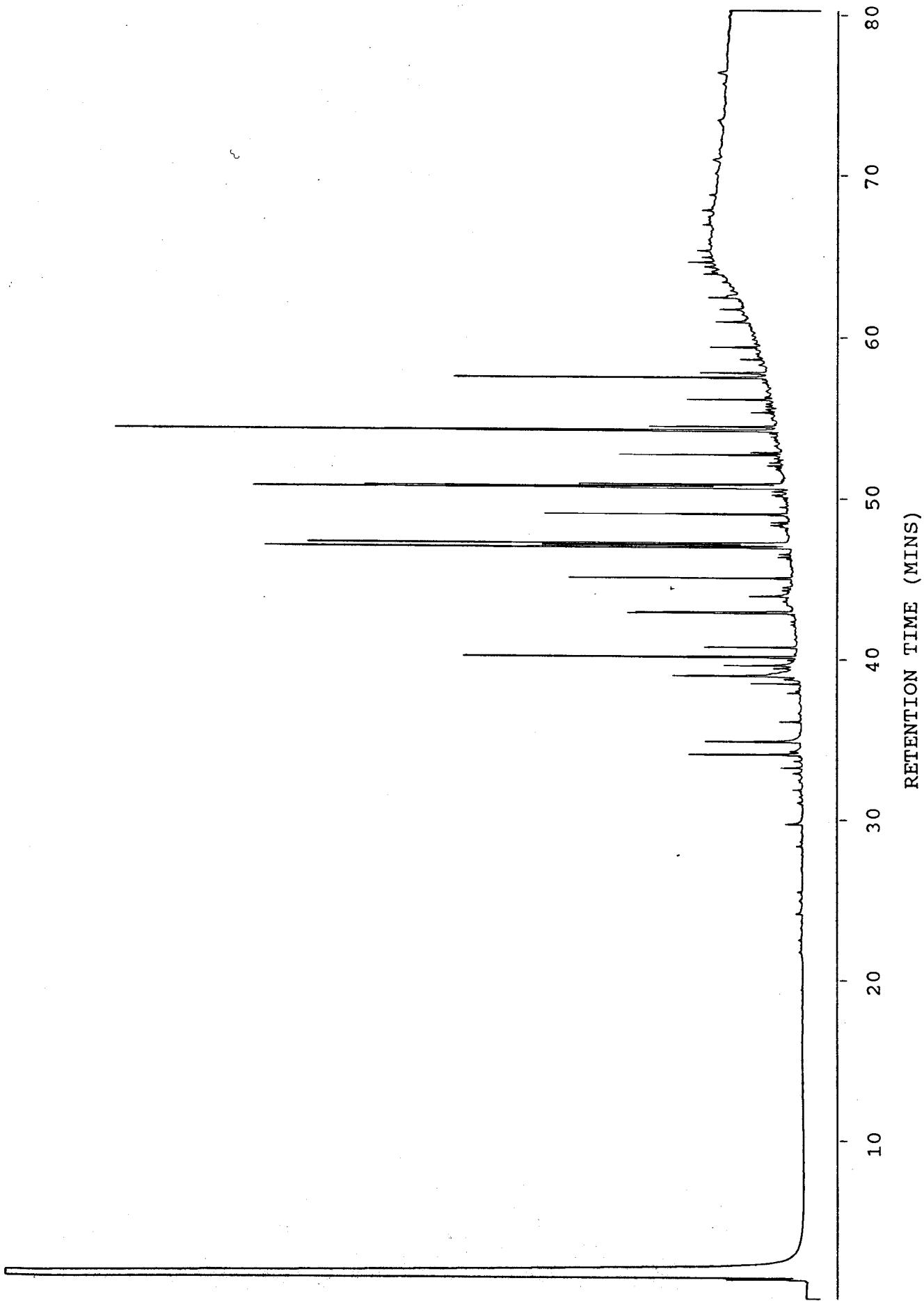
13 TOP TOTAL EXTRACT

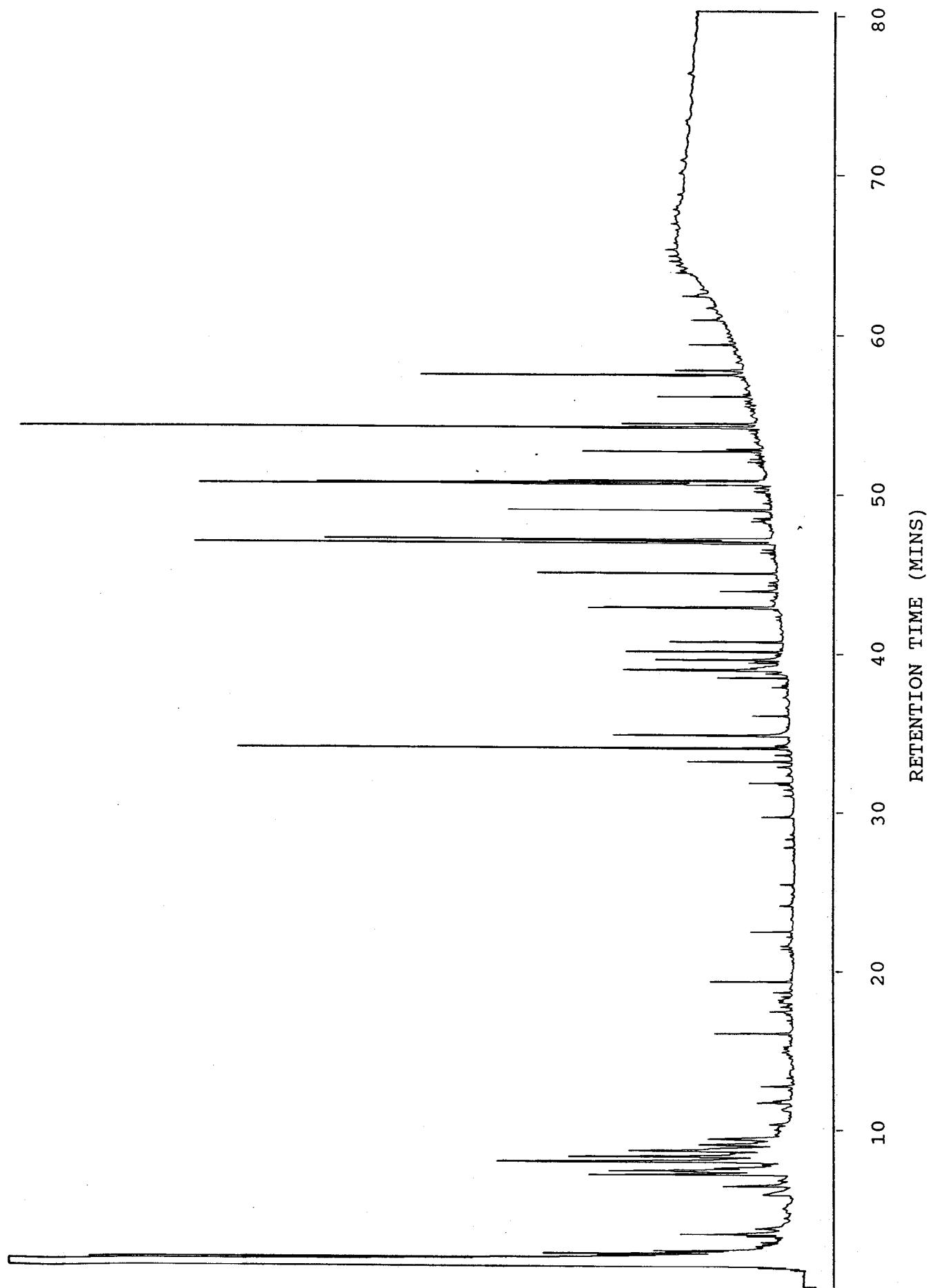


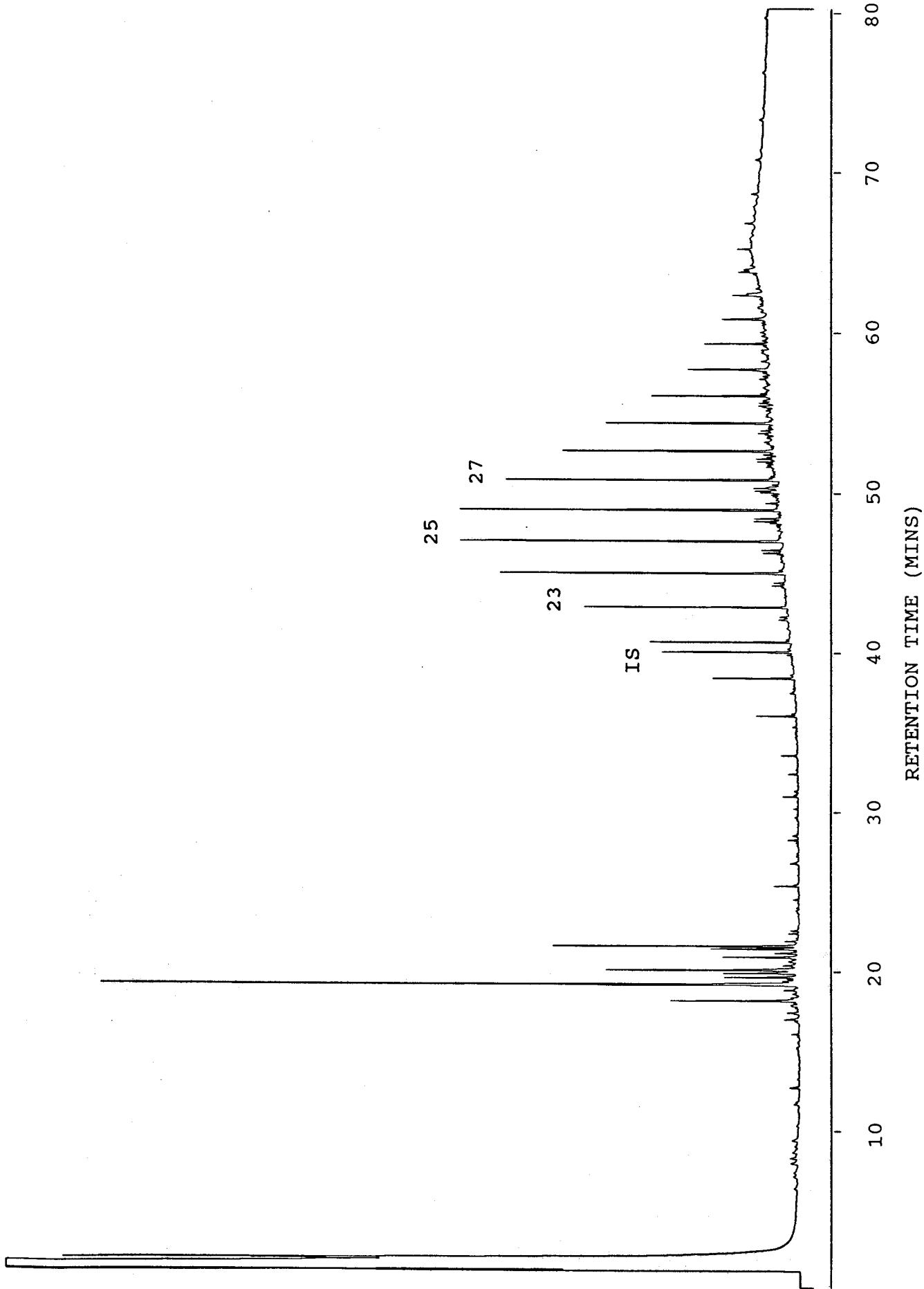


21 BOTTOM TOTAL EXTRACT

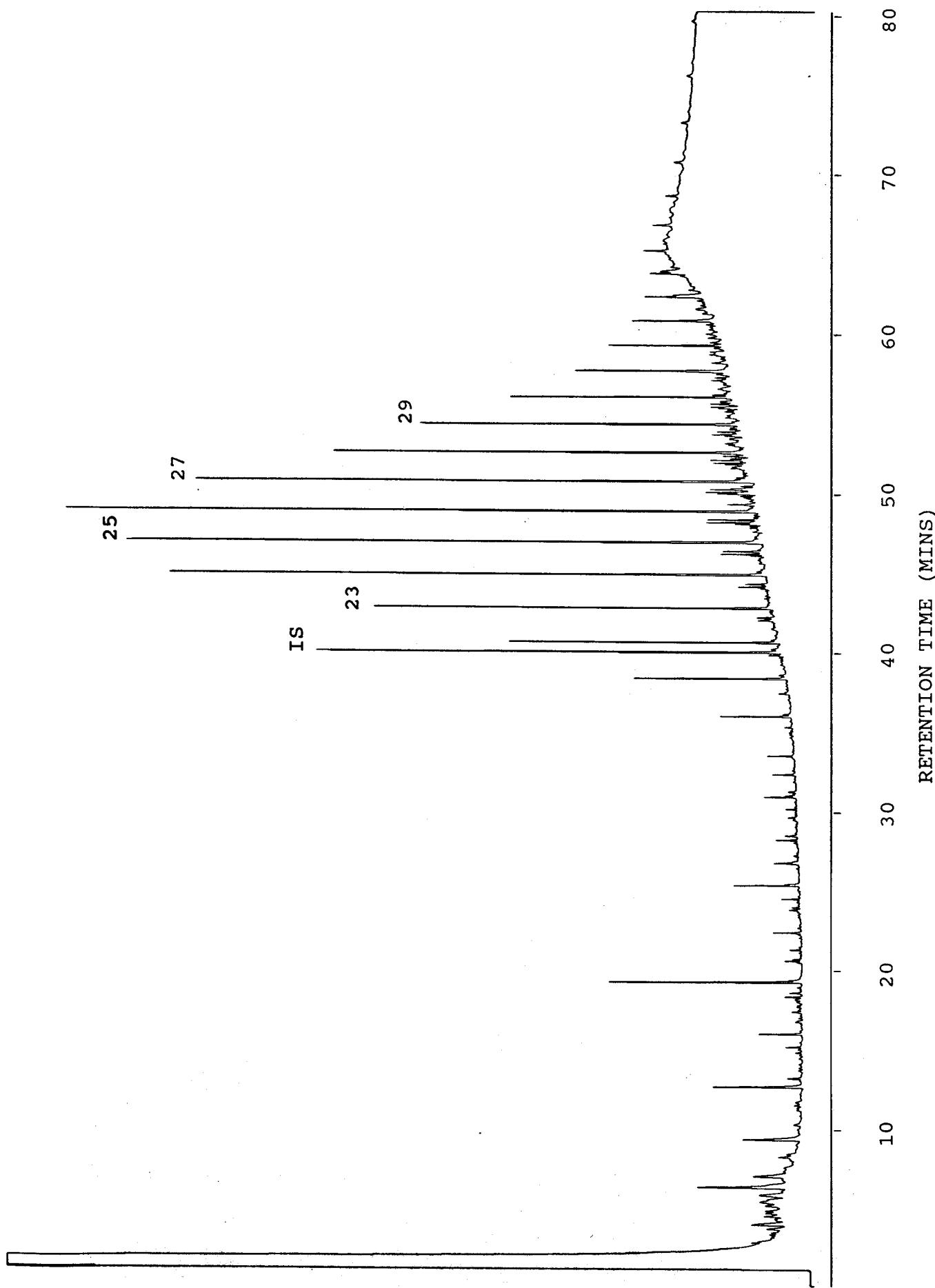


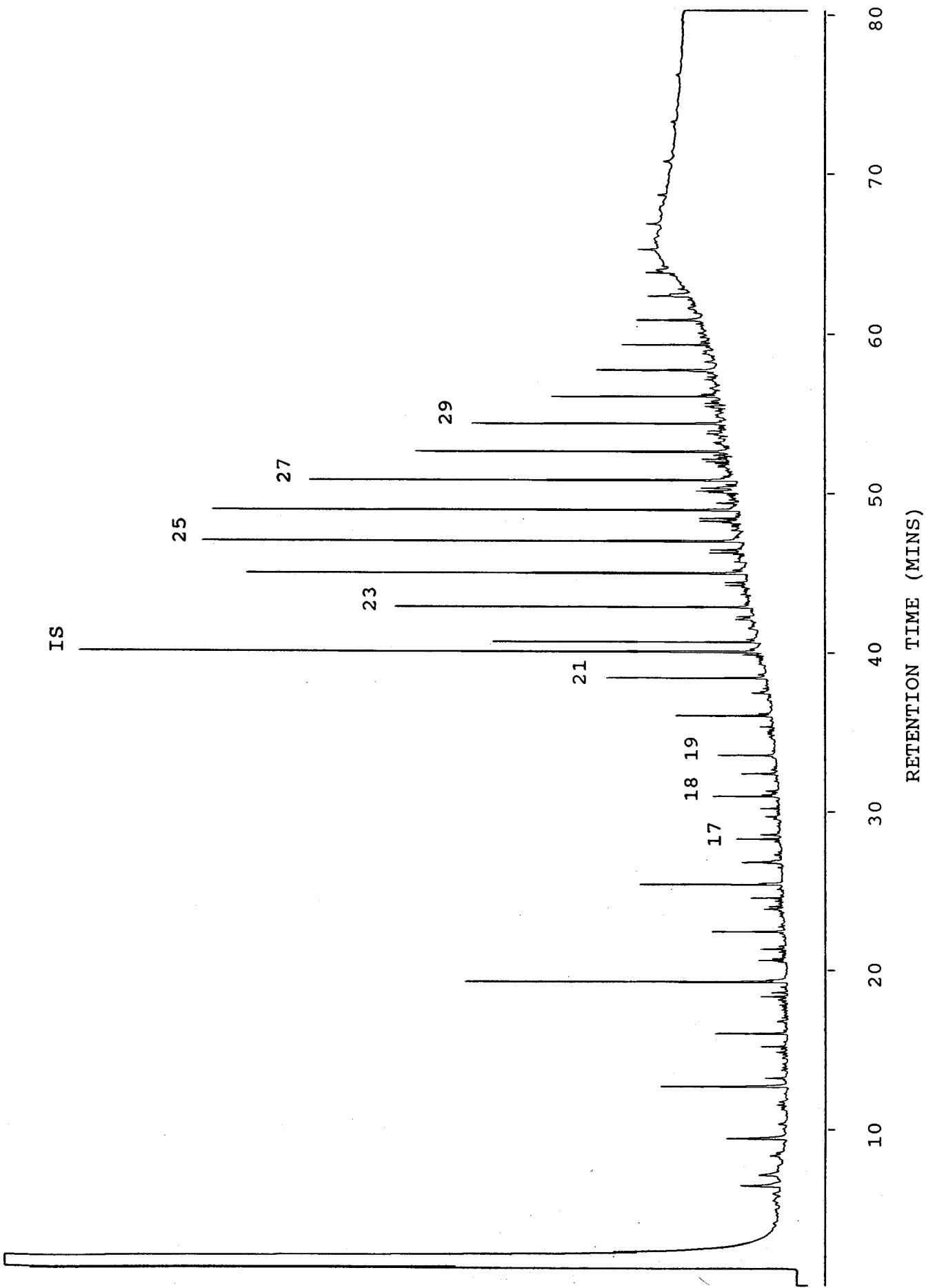


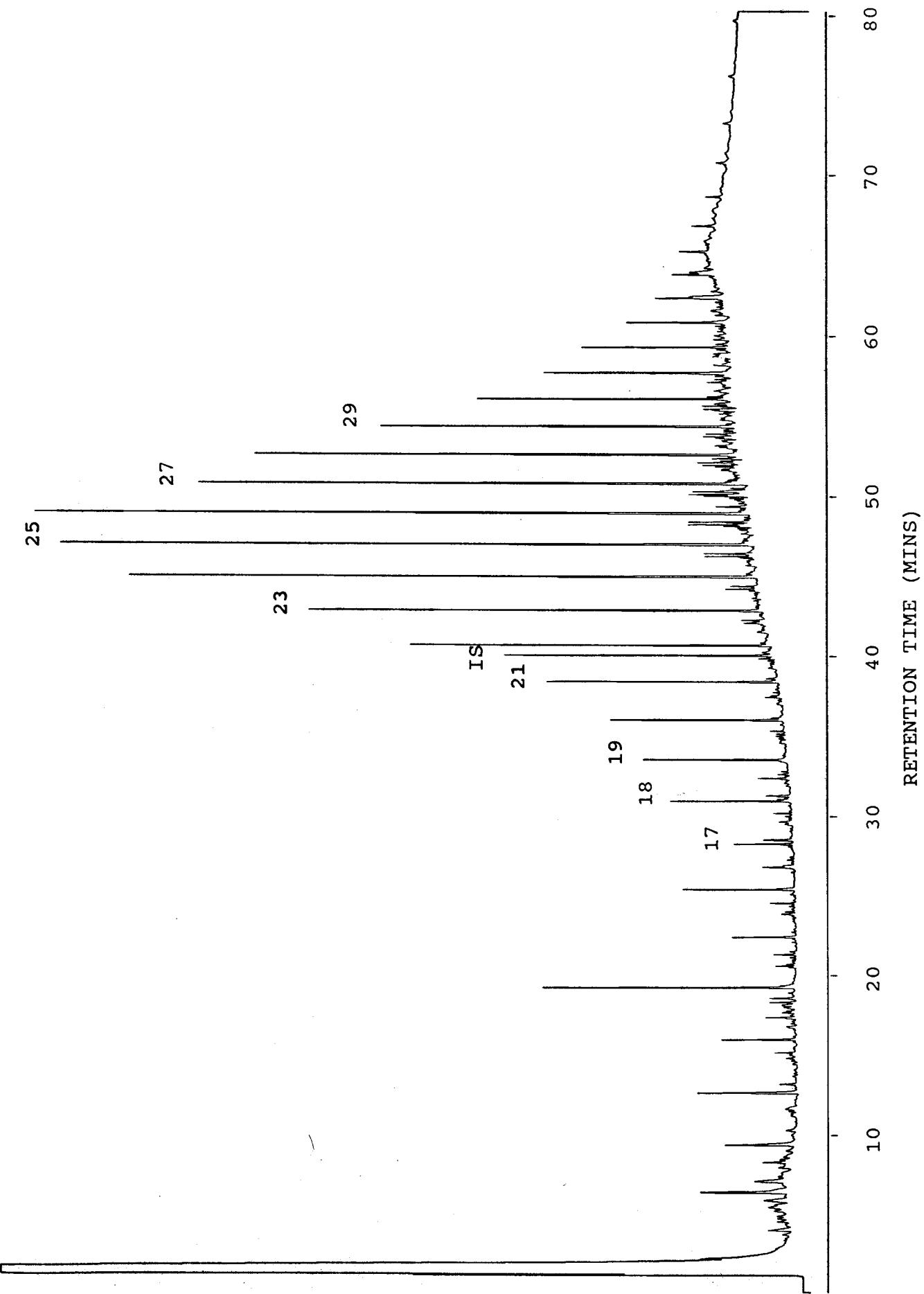




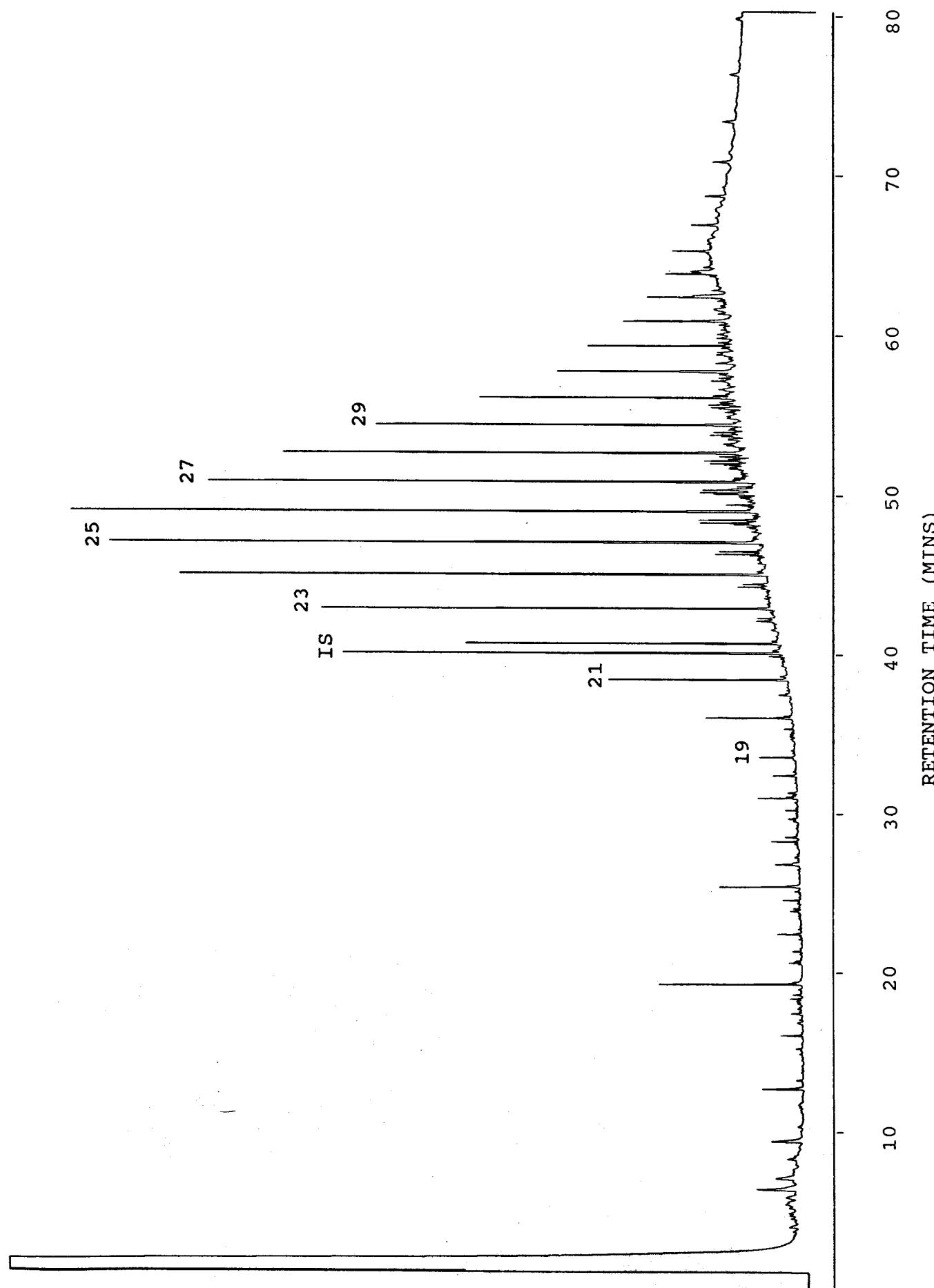
12 BOTTOM SATURATES



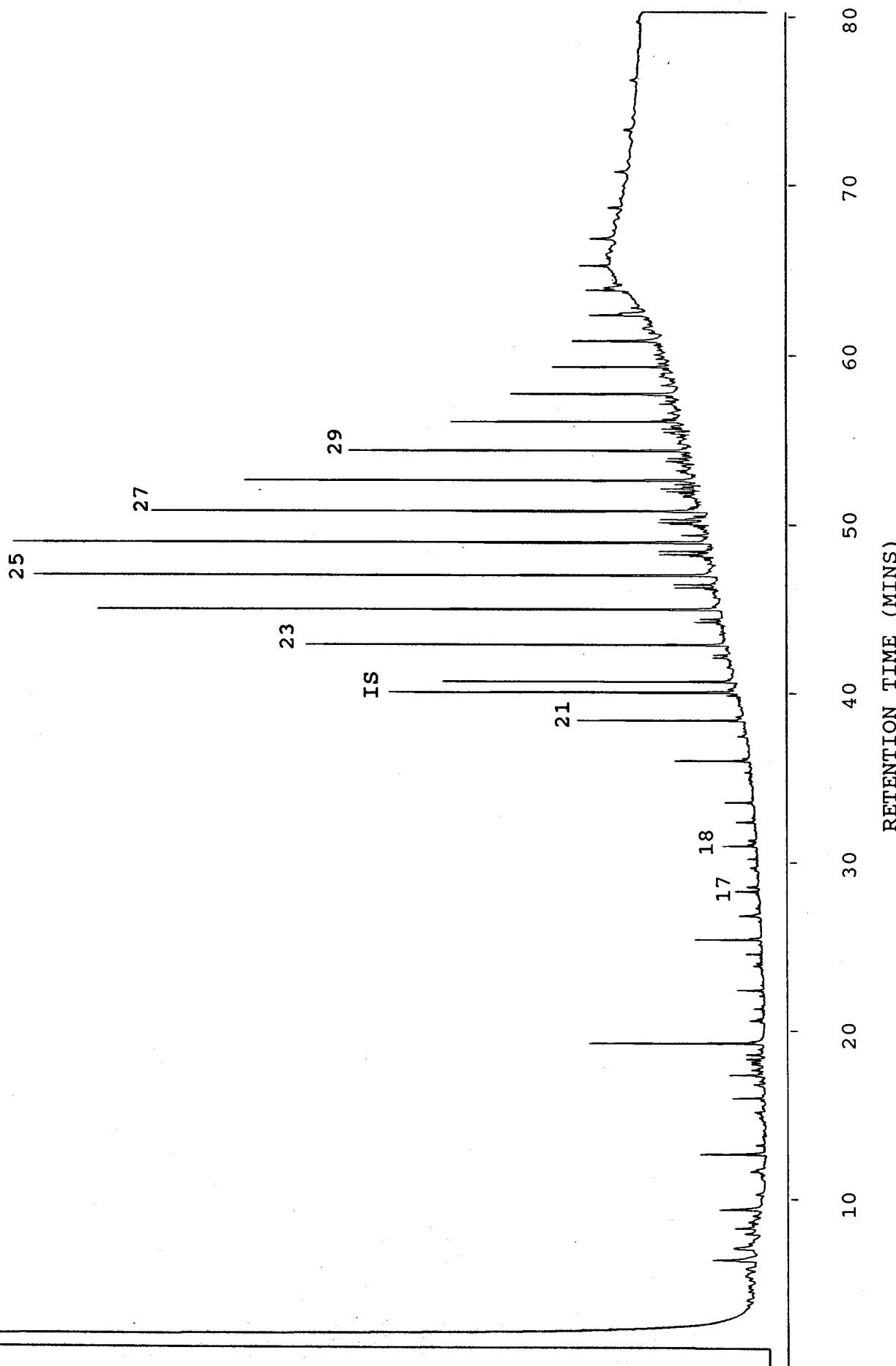


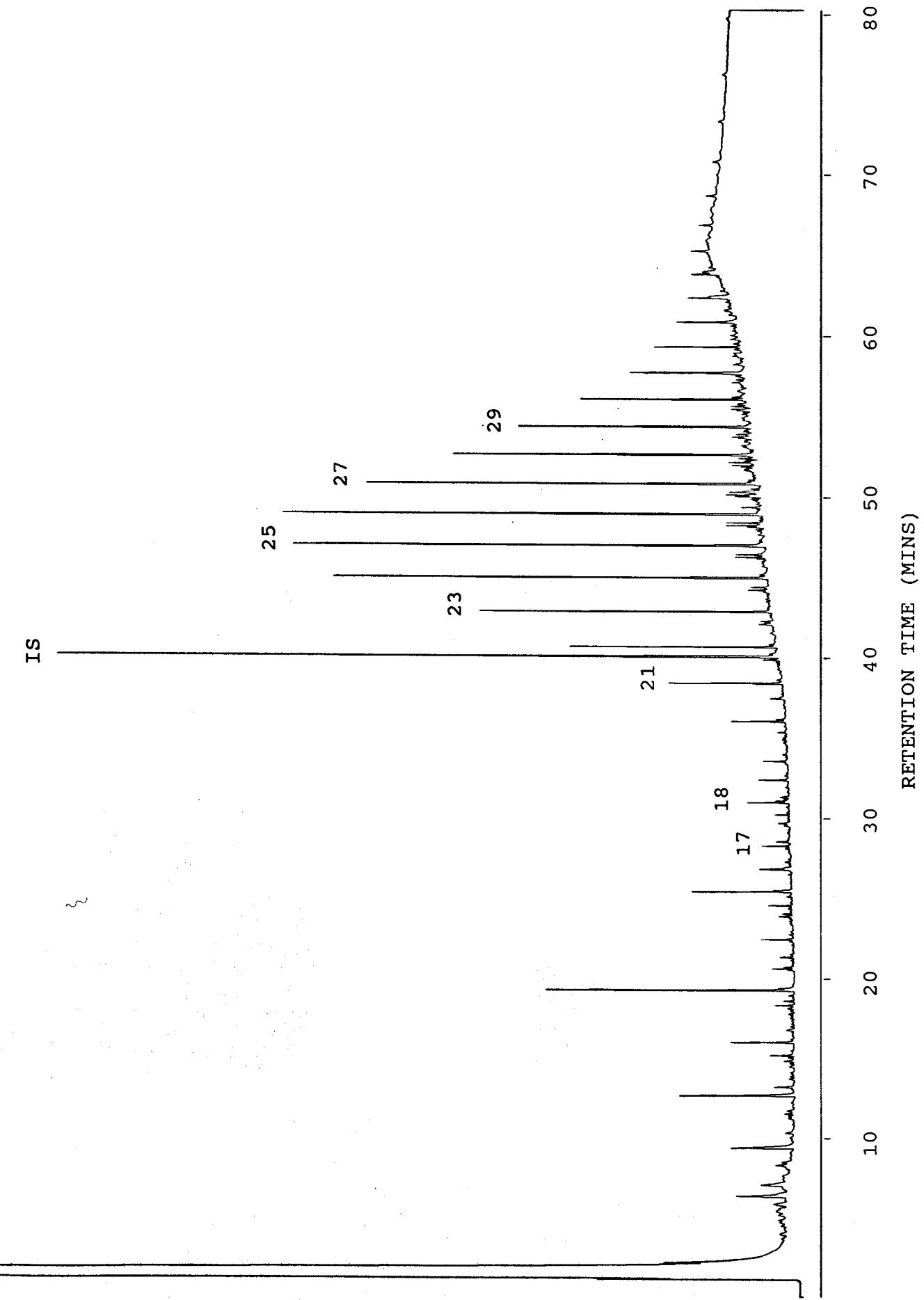


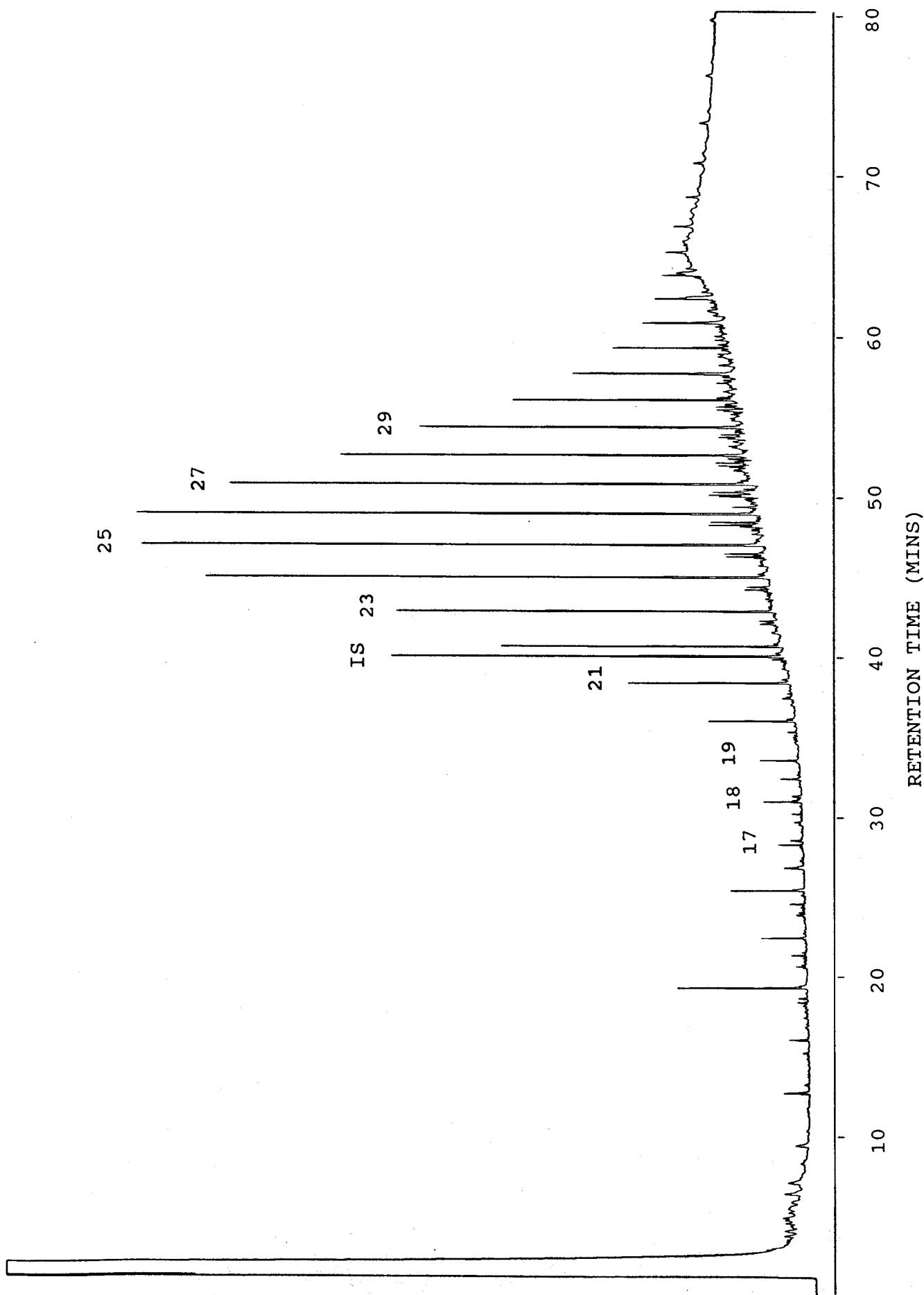
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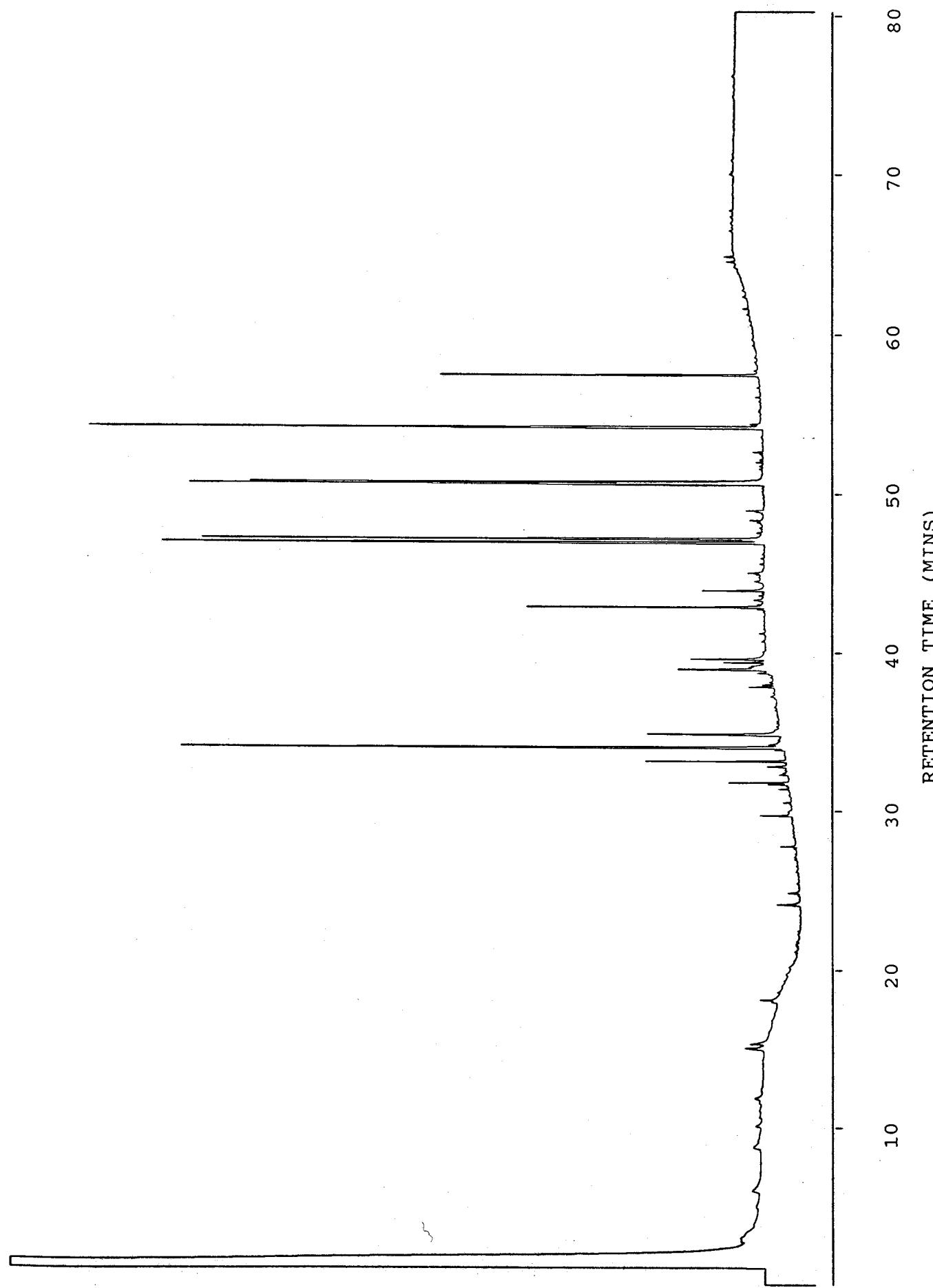
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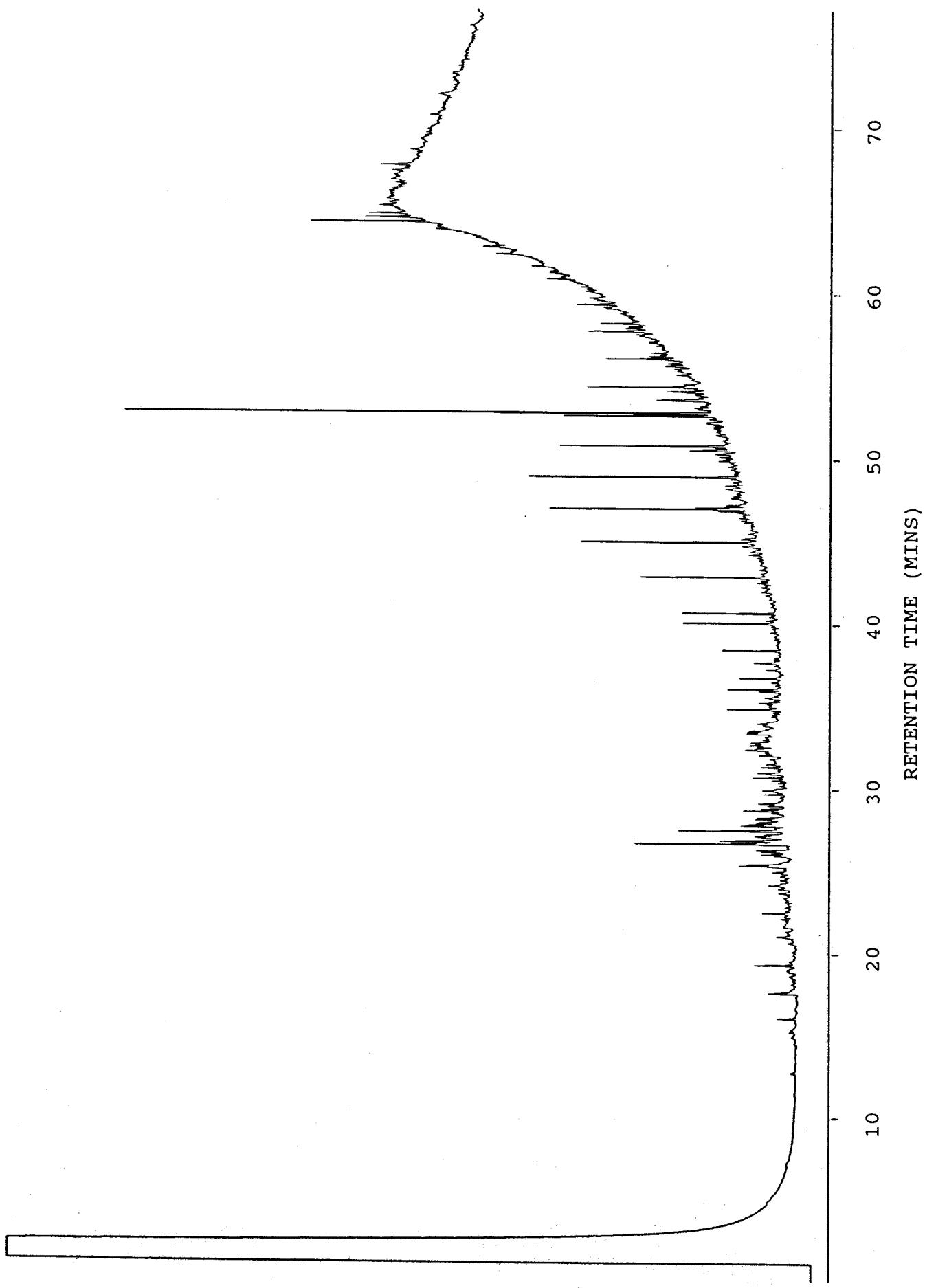




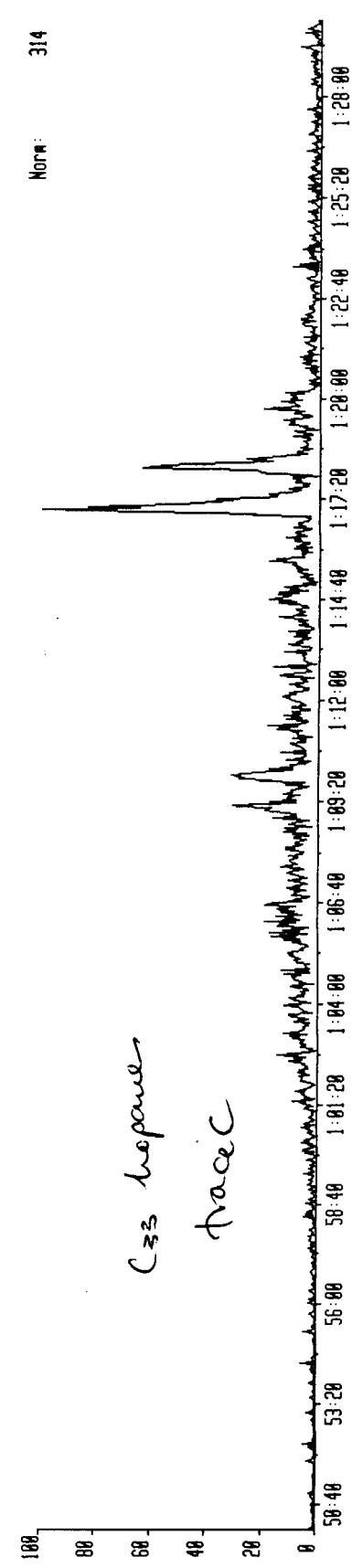
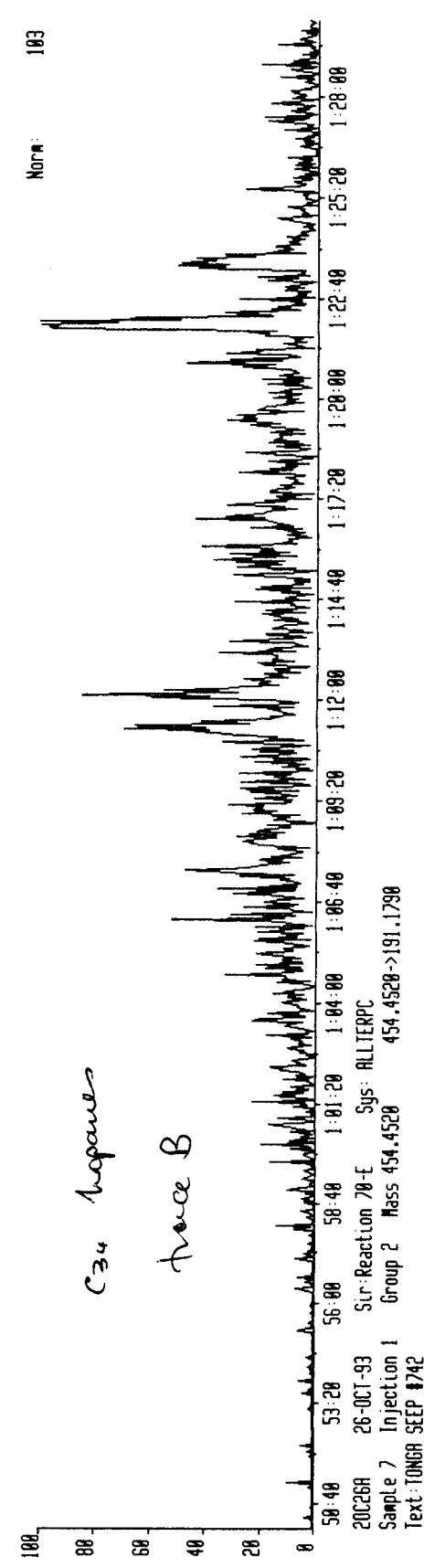
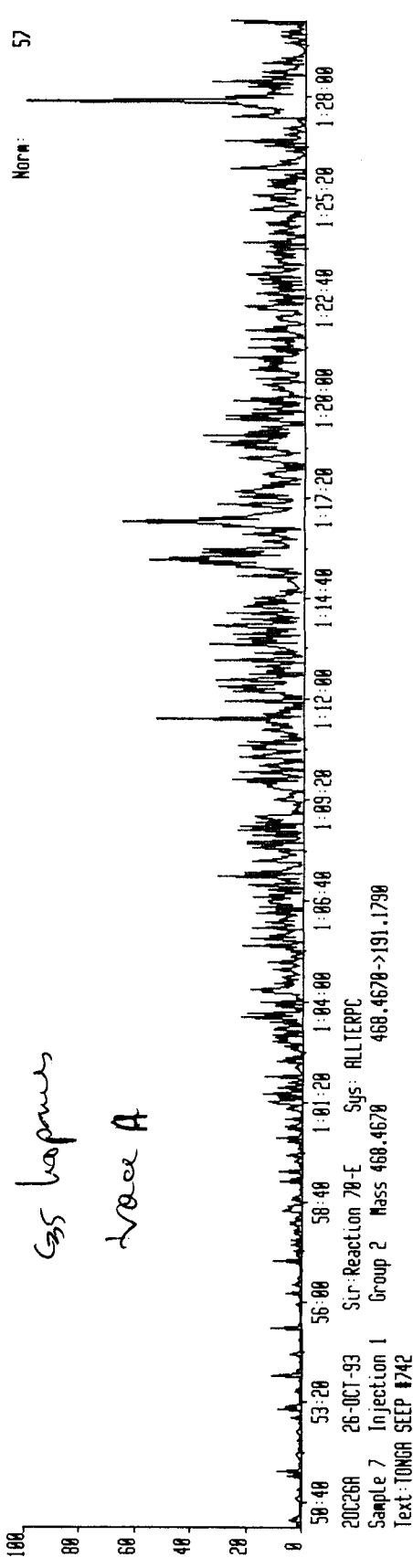


25 TOP POLARS

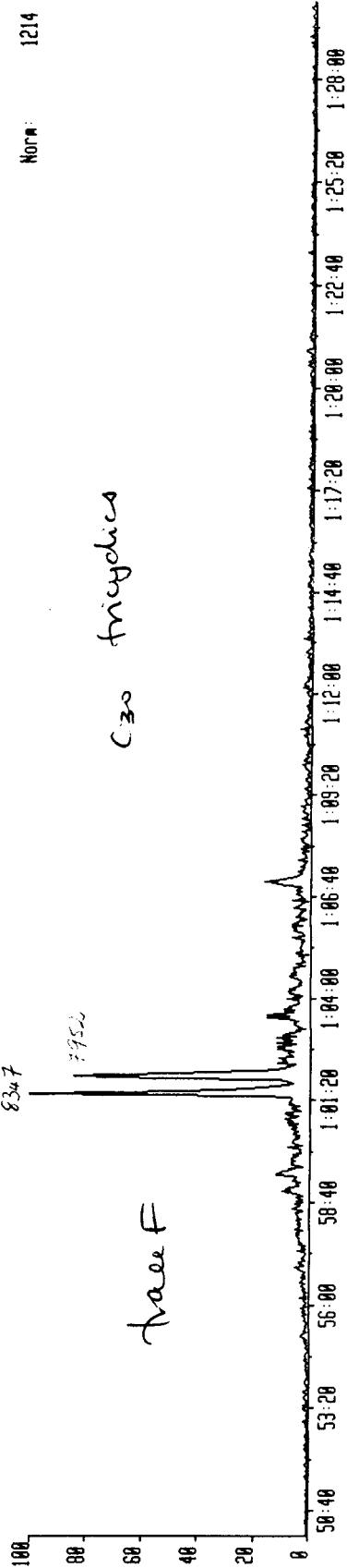
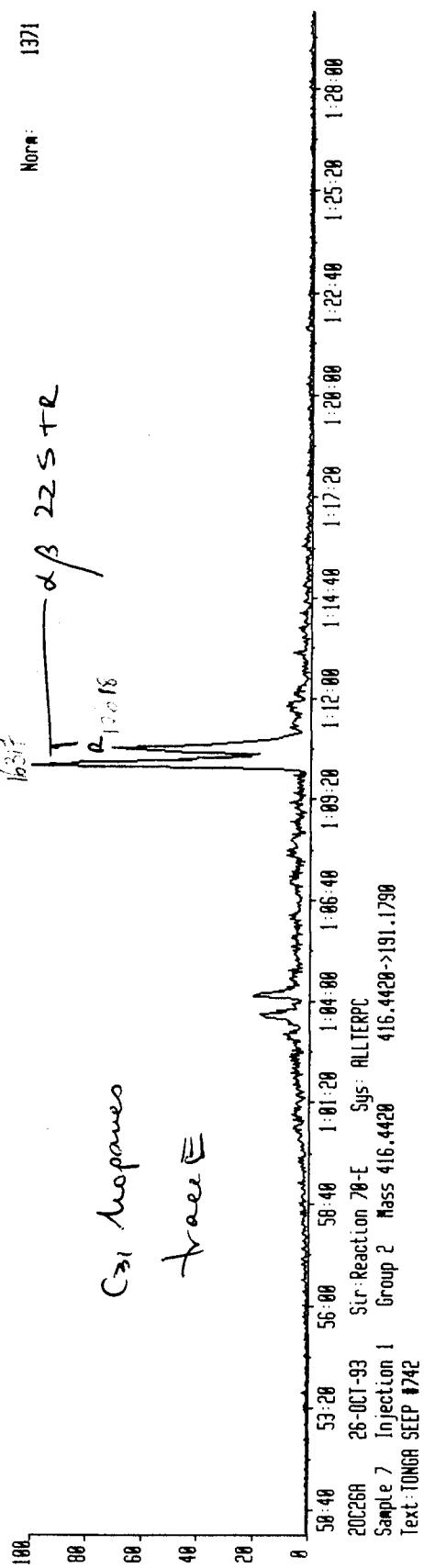
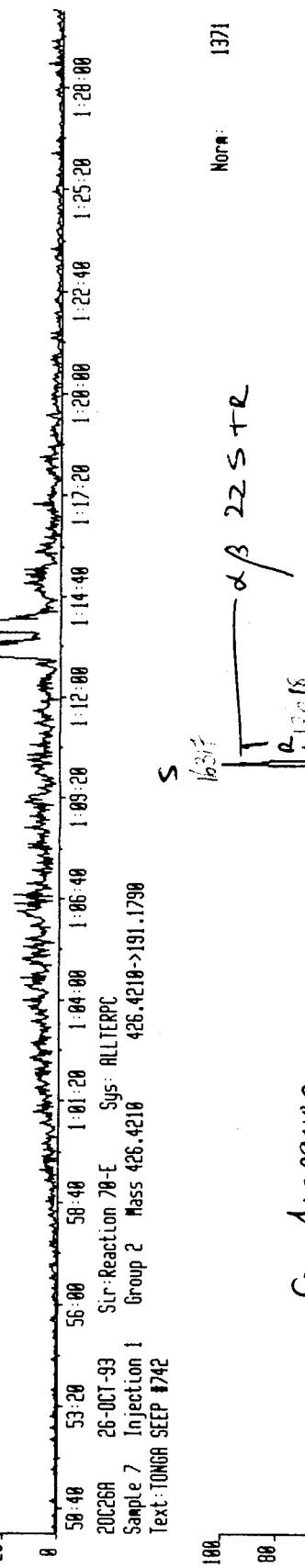




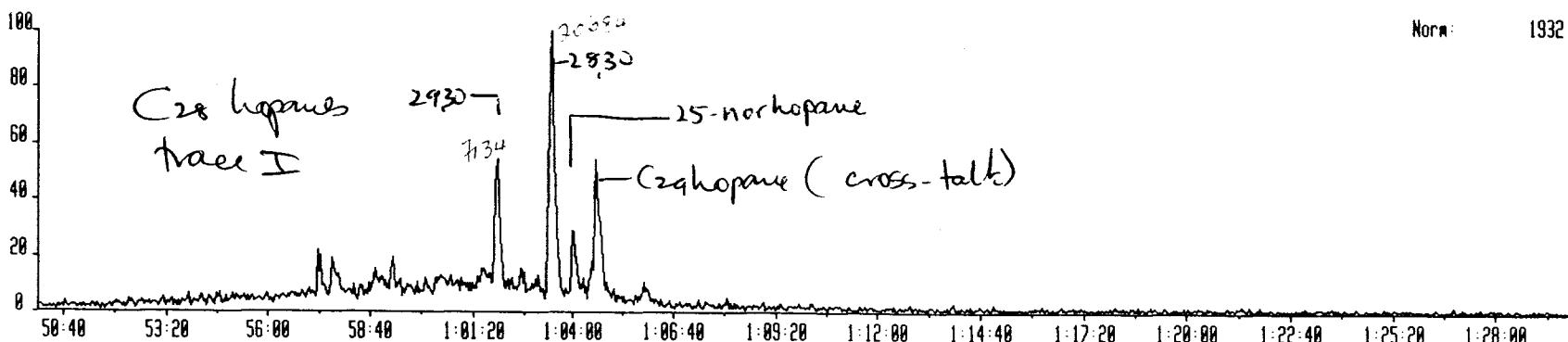
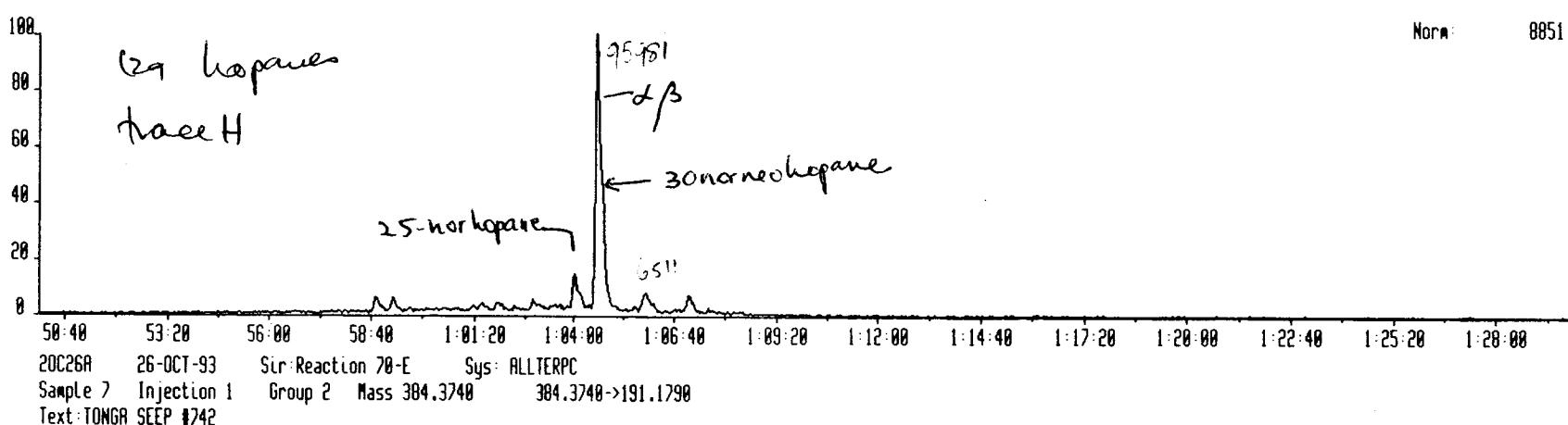
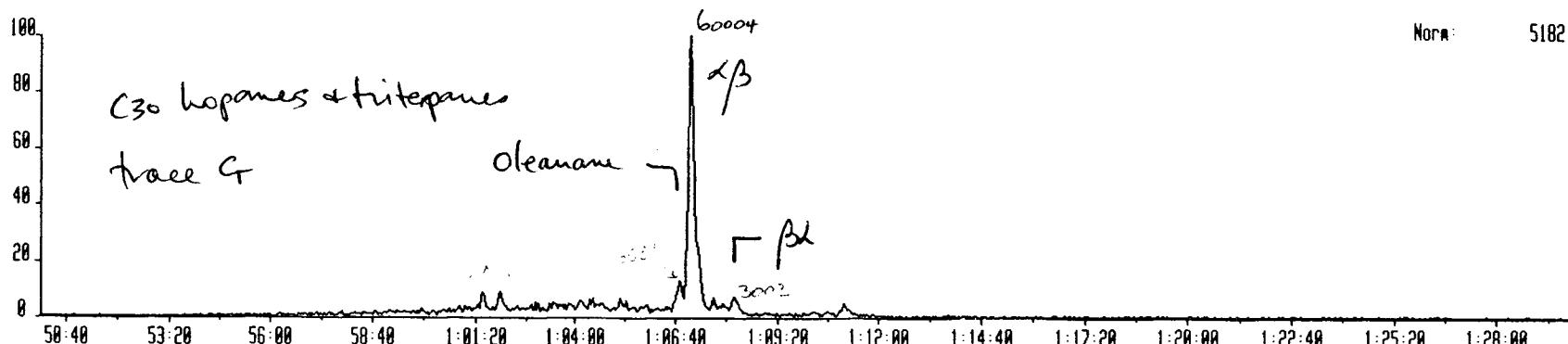
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Sample 7 Injection 1 Group 2 Mass 462.4828 462.4628->191.1790
Text: TONGA SEEP #742



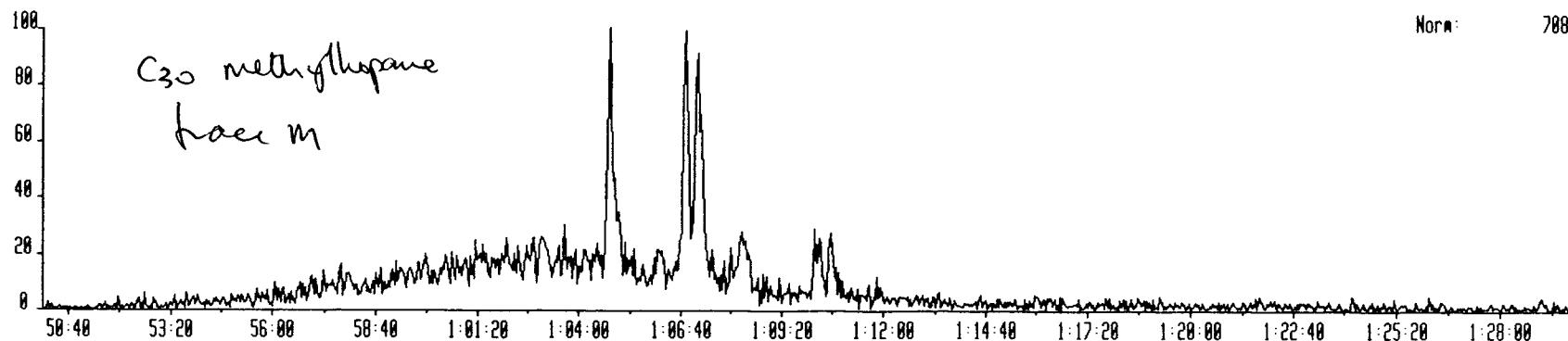
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Sample 7 Injection 1 Group 2 Mass 448.4370 448.4370->191.1790
Text: TONGA SEEP #742



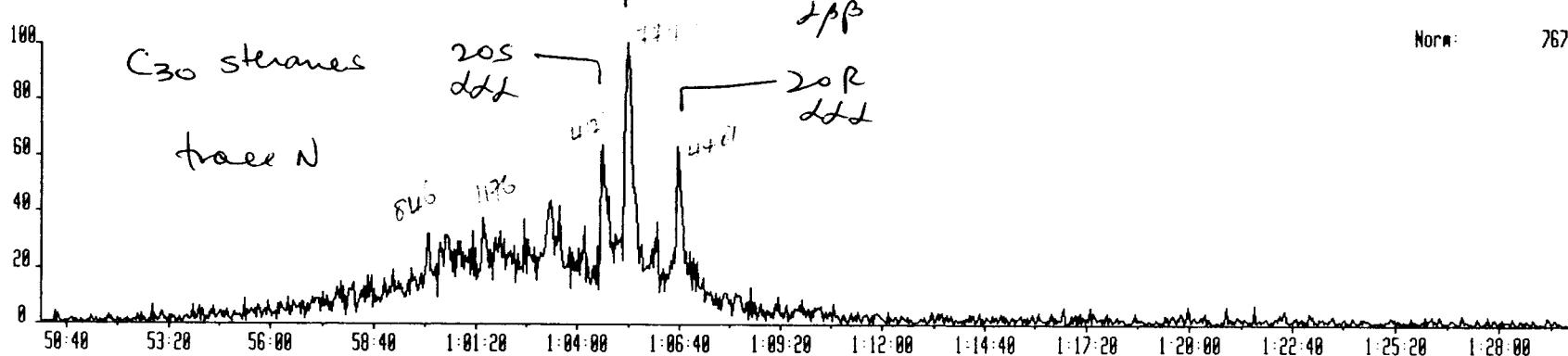
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Sample 7 Injection 1 Group 2 Mass 412.4060 412.4060->191.1790
Text: TONGA SEEP #742



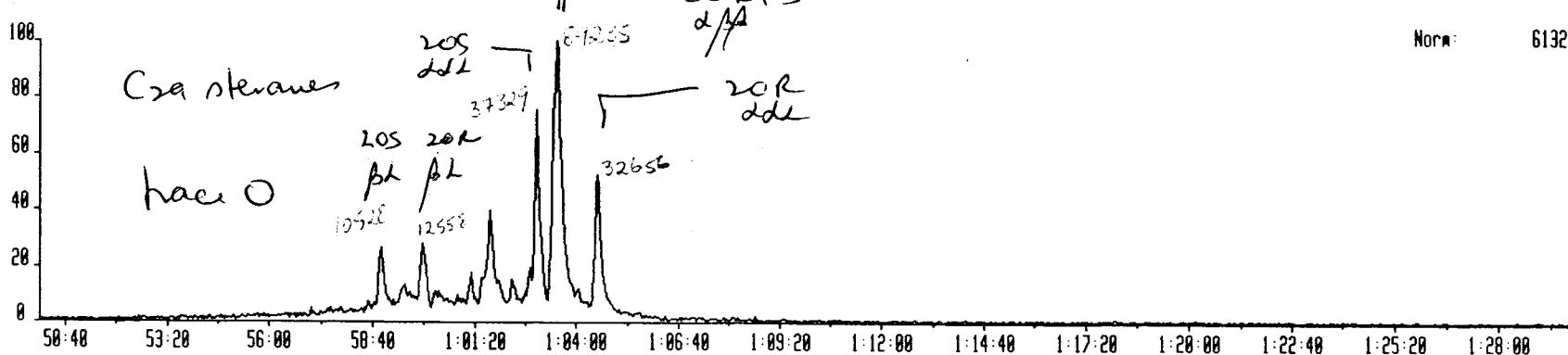
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Sample 7 Injection 1 Group 2 Mass 412.4060 412.4060->205.1940
Text:TONGA SEEP #742



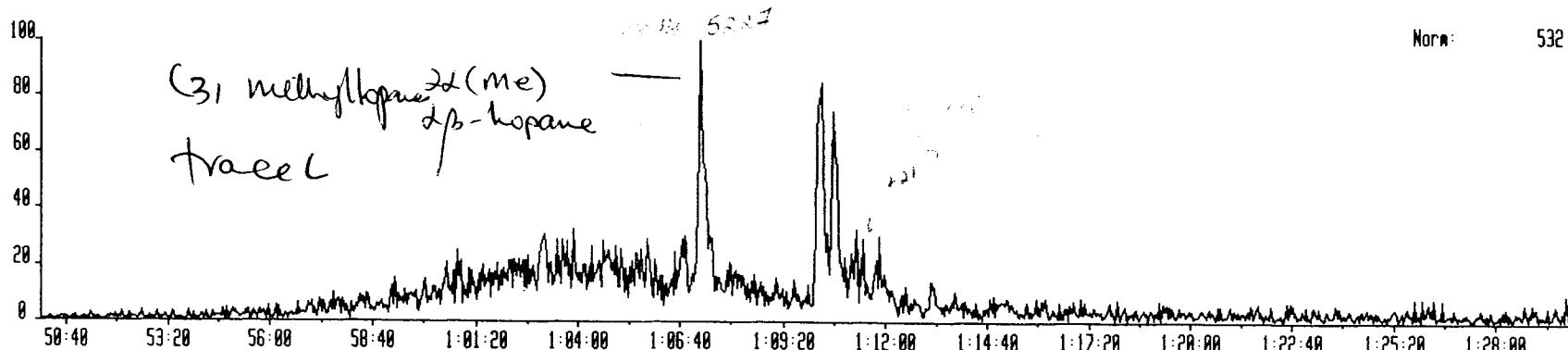
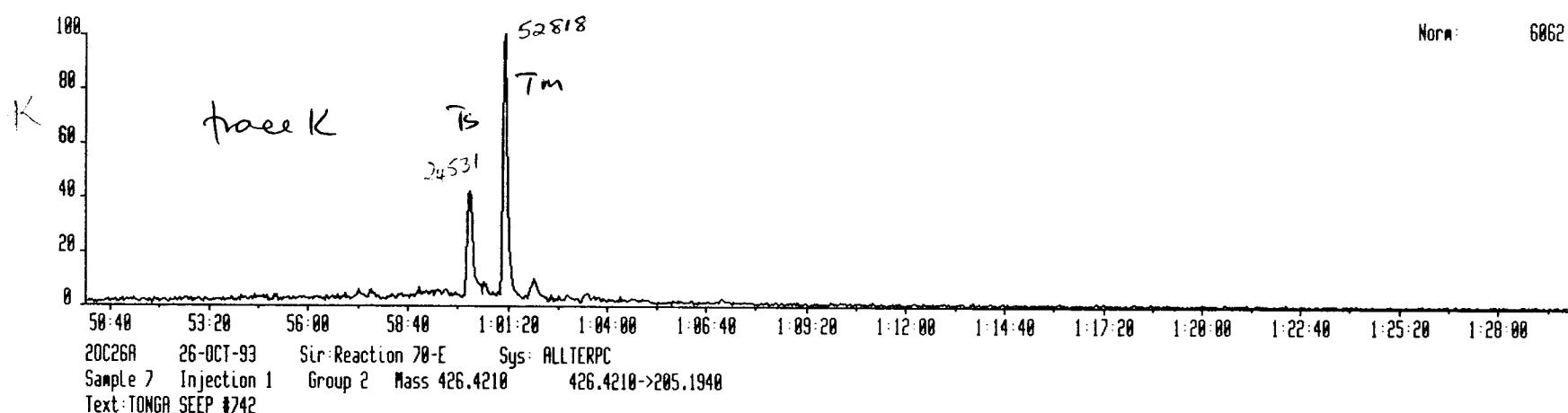
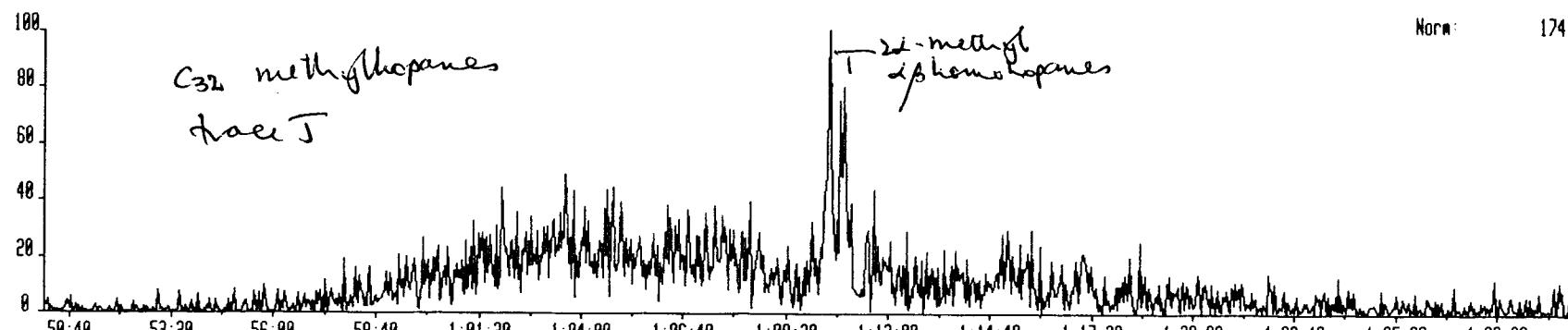
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Sample 7 Injection 1 Group 2 Mass 414.4230 414.4230->217.1960
Text:TONGA SEEP #742



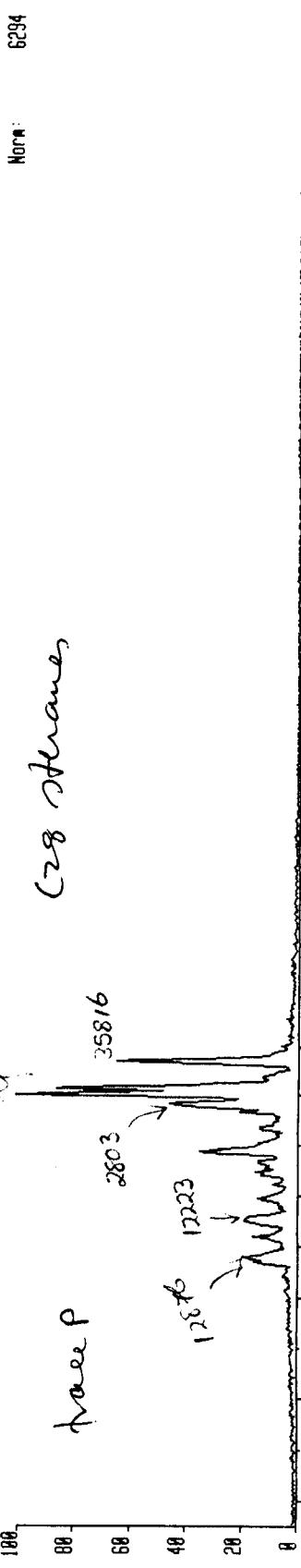
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Sample 7 Injection 1 Group 2 Mass 400.4078 400.4078->217.1960
Text:TONGA SEEP #742



20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 7 Injection 1 Group 2 Mass 440.4370 440.4370->205.1940
Text:TONGA SEEP #742



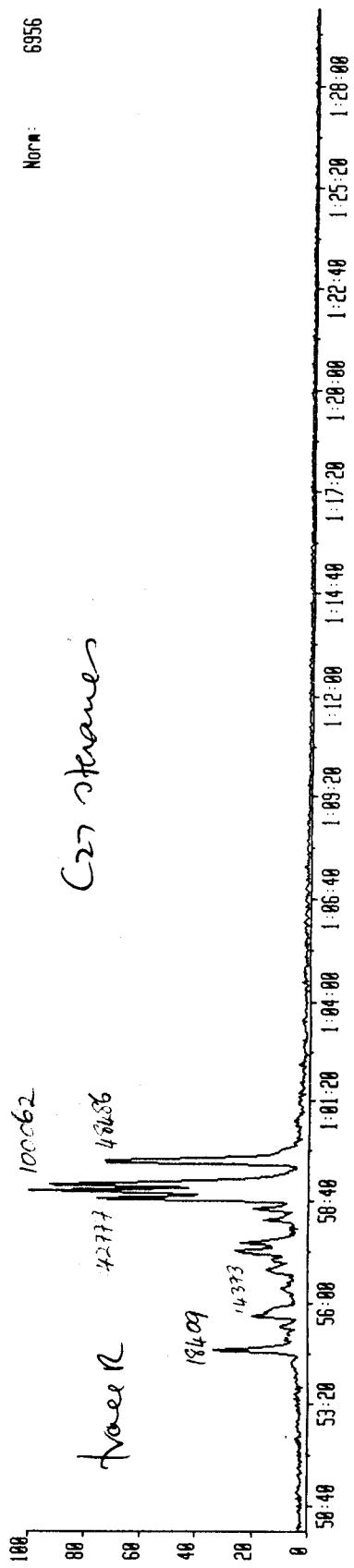
Sample 7 Infection 1
Group 2 Mass 396.3918
Surveillance W-1
July 29, 2011
Sample 7 Infection 1
Group 2 Mass 396.3918
Surveillance W-1
July 29, 2011
Expt 1003 SEP 14
396.3918 >217.1998



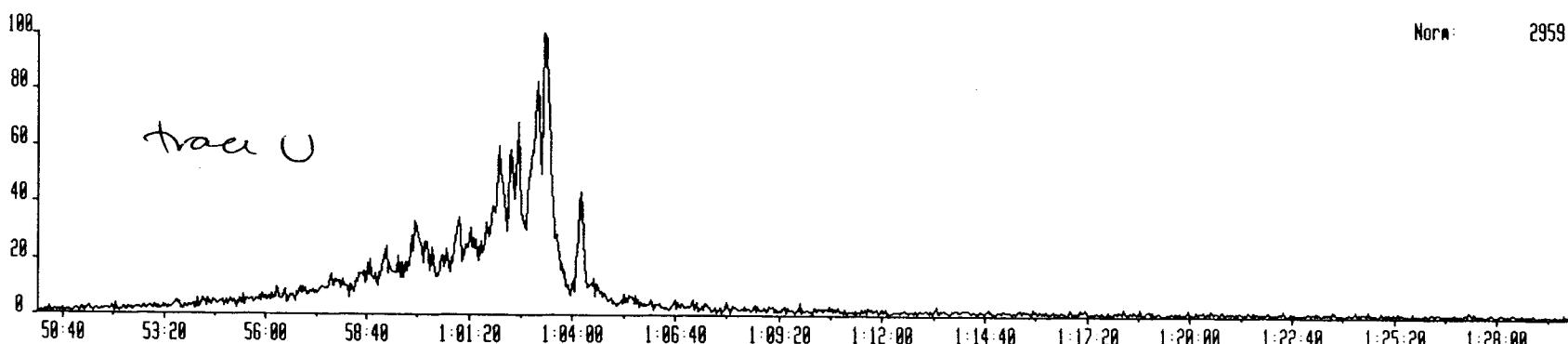
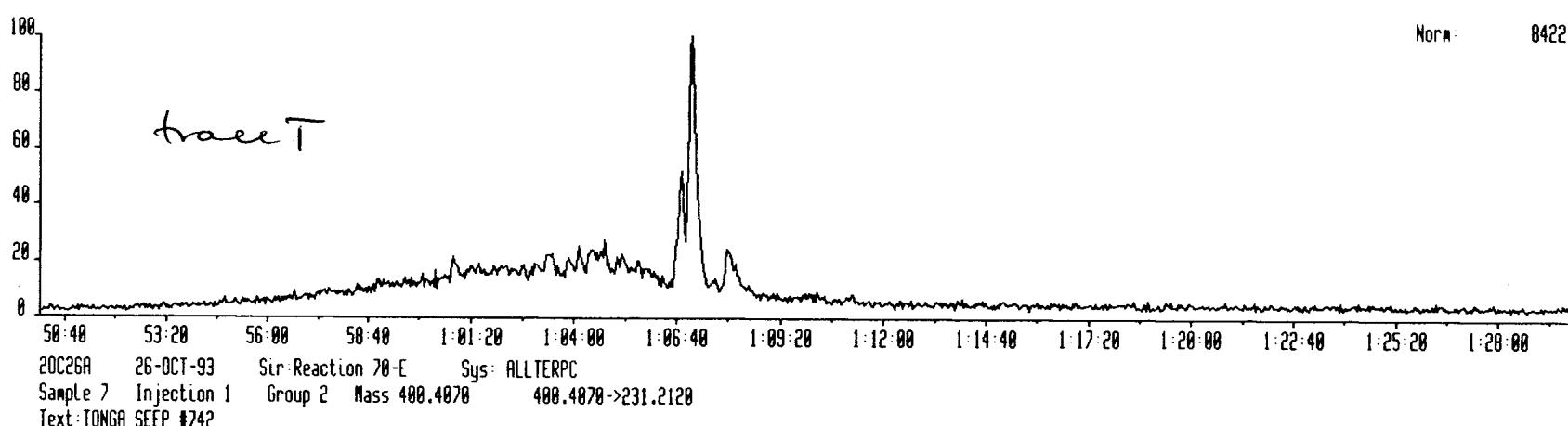
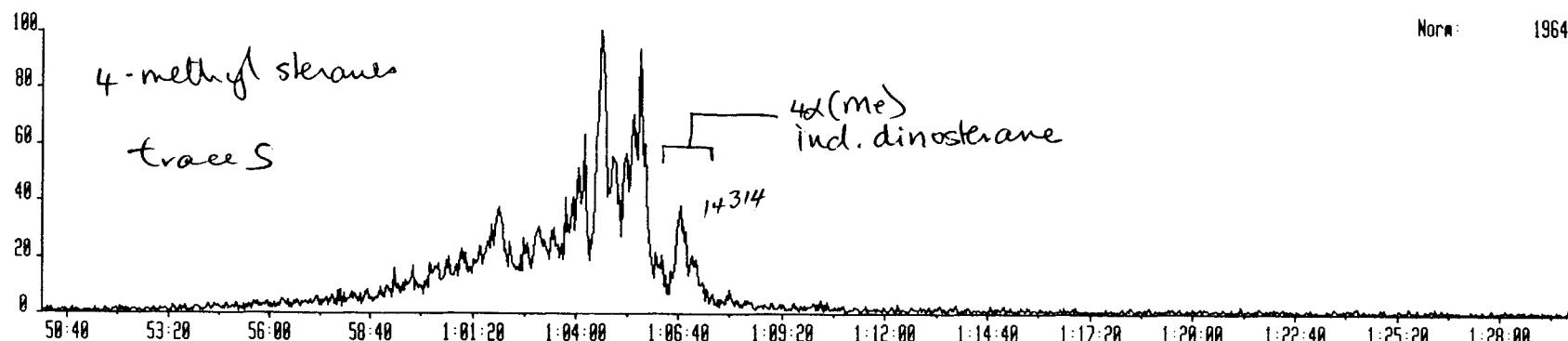
20268A 26-0C1-93 Sir Reaction 78-F Sys: ALLTERPC
 Sample 7 Injection 1 Group 2 Mass: 428,4308 428,4308->231,2129
 Test: TONGA SEEP #742



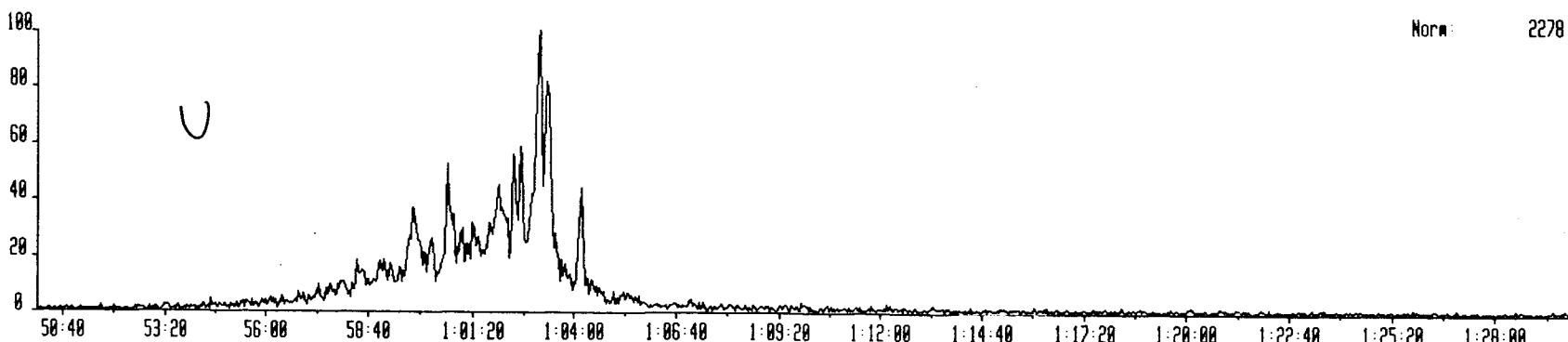
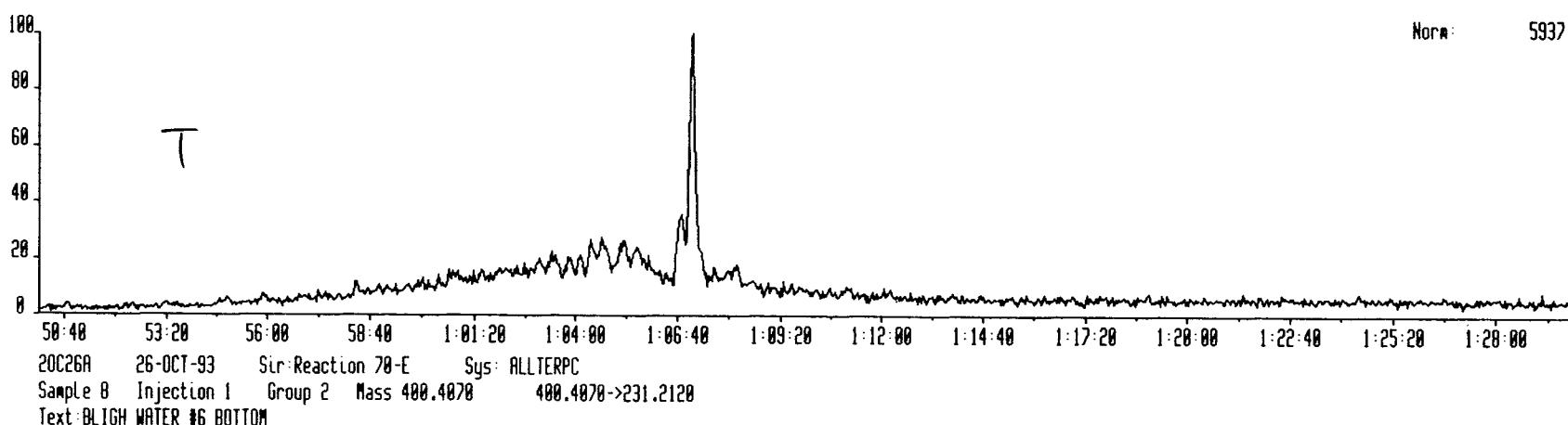
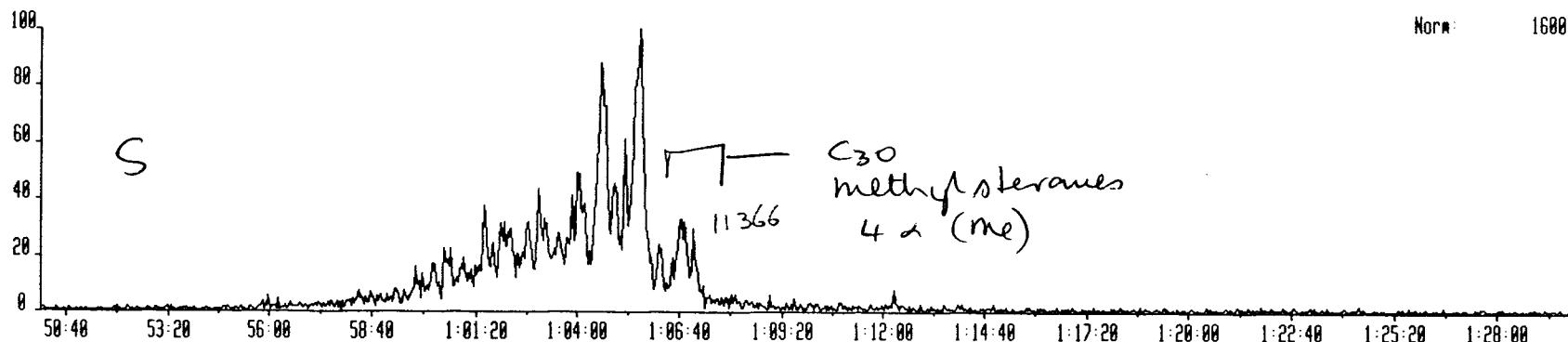
20269 26-007-93 Sir: Reaction 70-E Sus: ALLIERPC
Sample 7 Injection 1 Group 2 Mass 372.3768 >217.1968
Text: TONGA SEEP #742



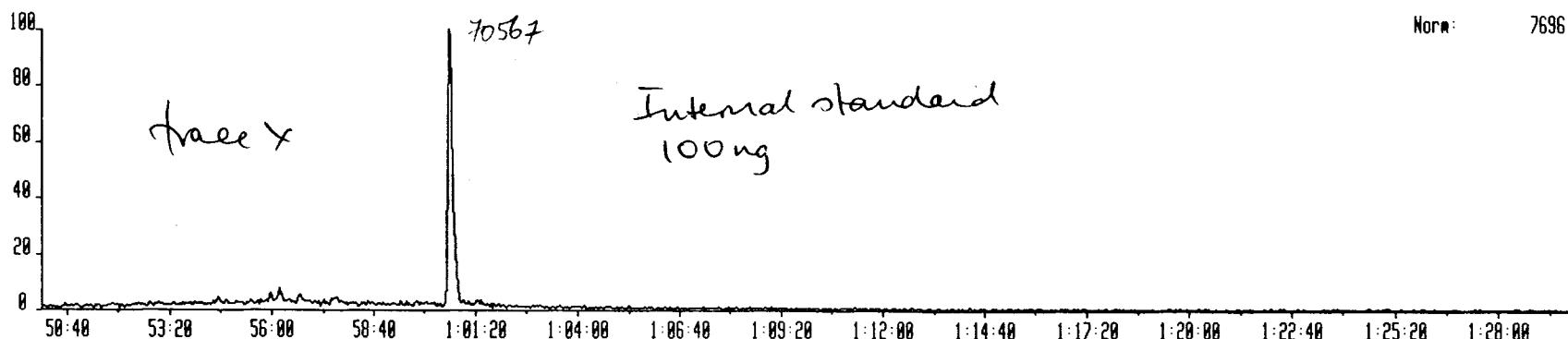
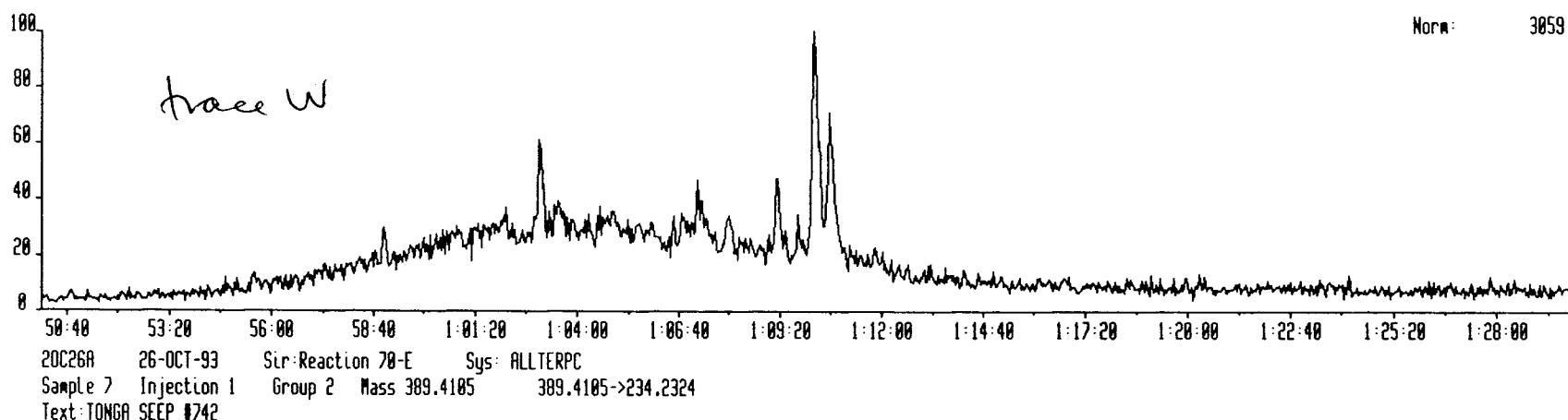
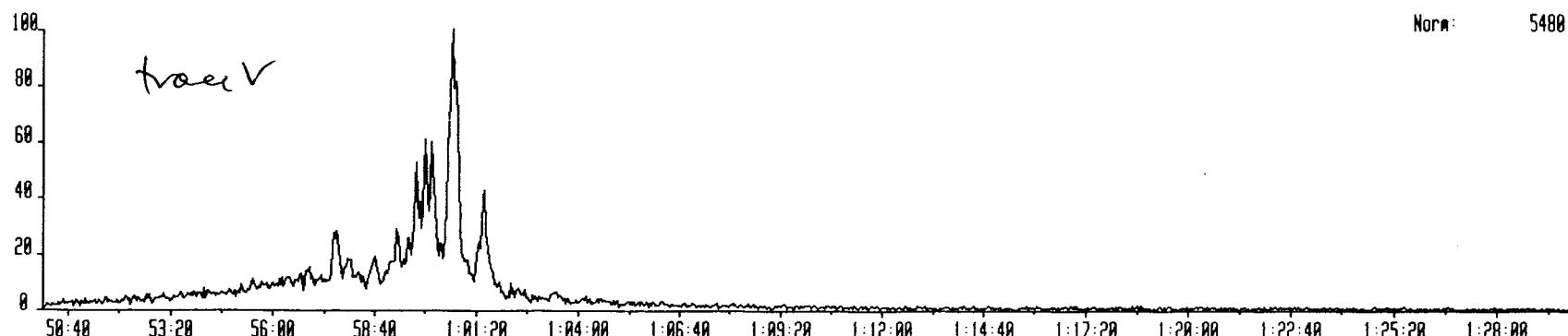
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Sample 7 Injection 1 Group 2 Mass 414.4230 414.4230->231.2120
Text: TONGA SEEP #742



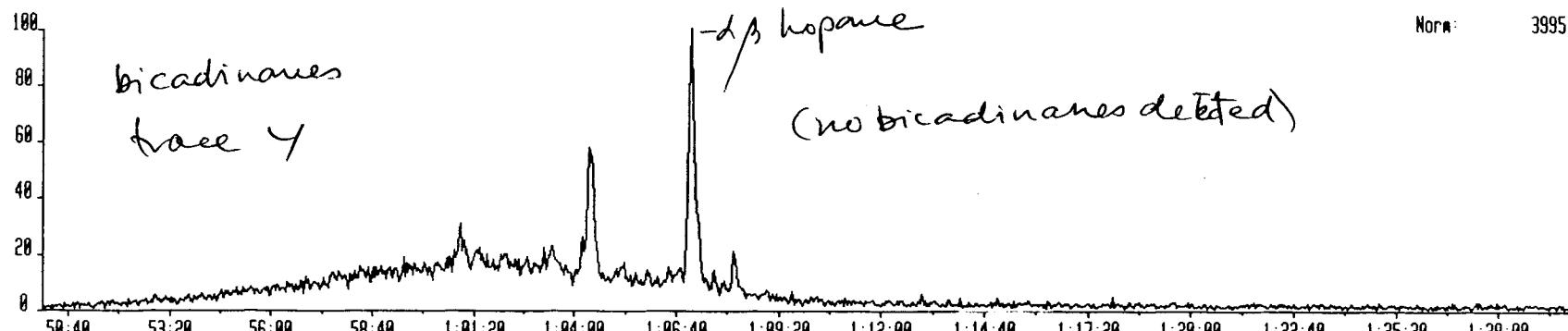
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Sample 8 Injection 1 Group 2 Mass 414.4230 414.4230->231.2120
Text:BLIGH WATER #6 BOTTOM



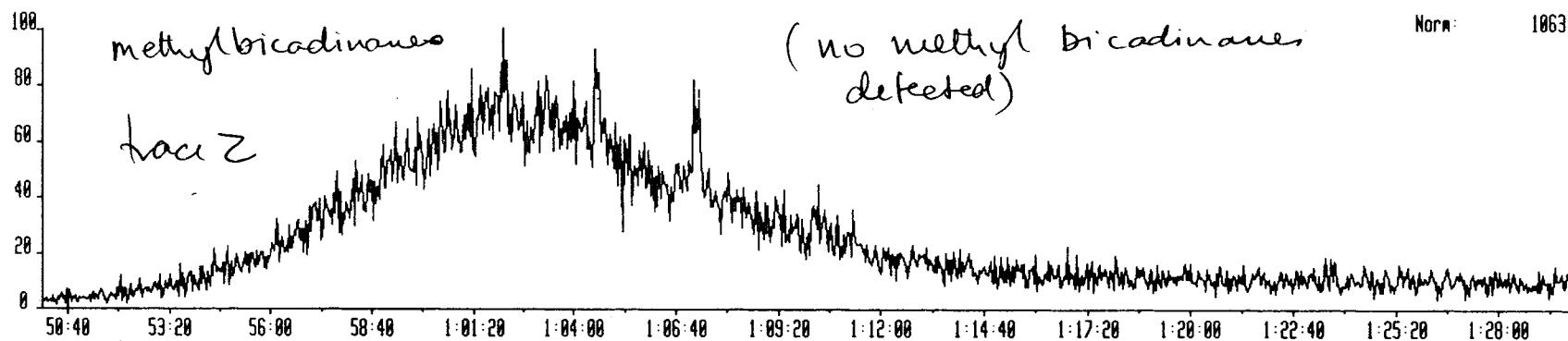
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Sample 7 Injection 1 Group 2 Mass 386.3910 386.3910->231.2120
Text:TONGA SEEP #742



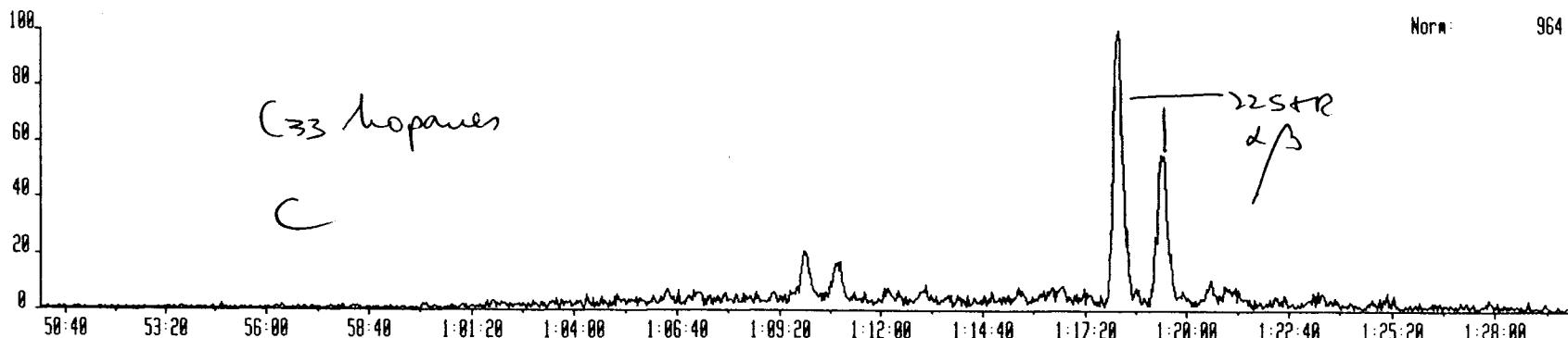
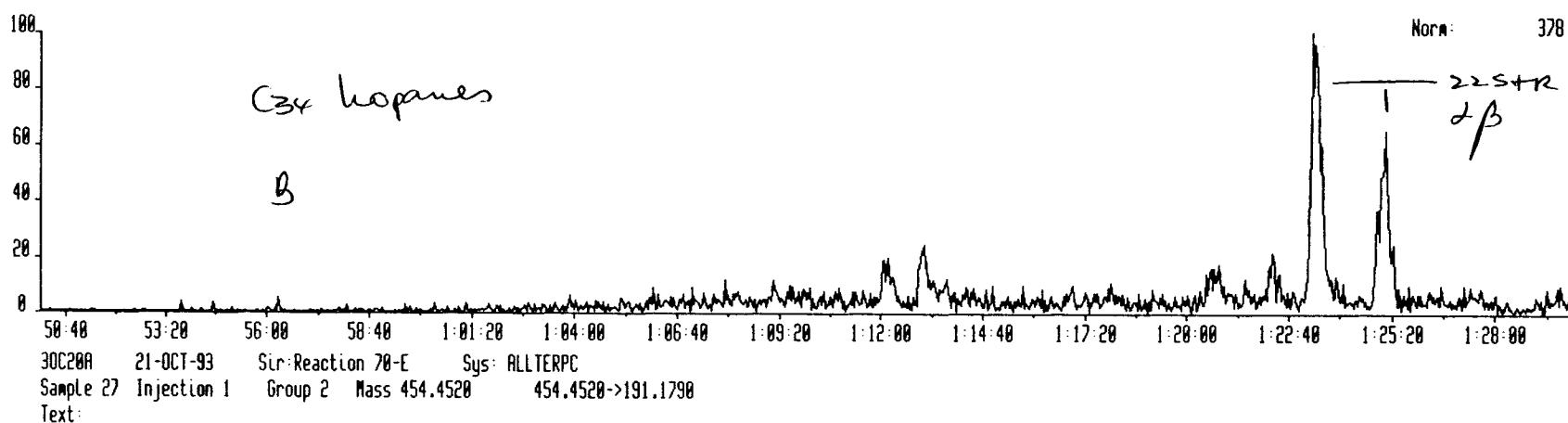
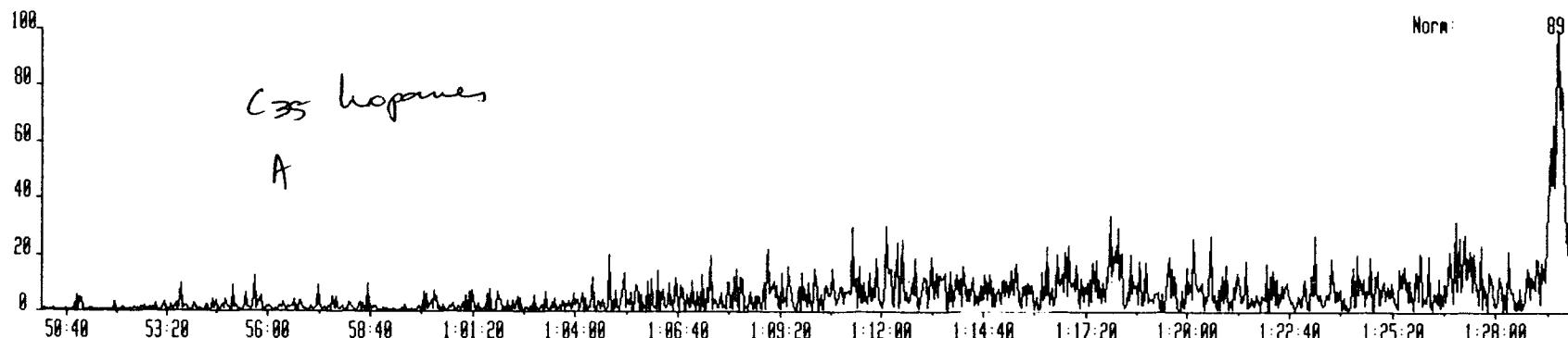
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Sample 7 Injection 1 Group 2 Mass 412.4060 412.4060->369.3400
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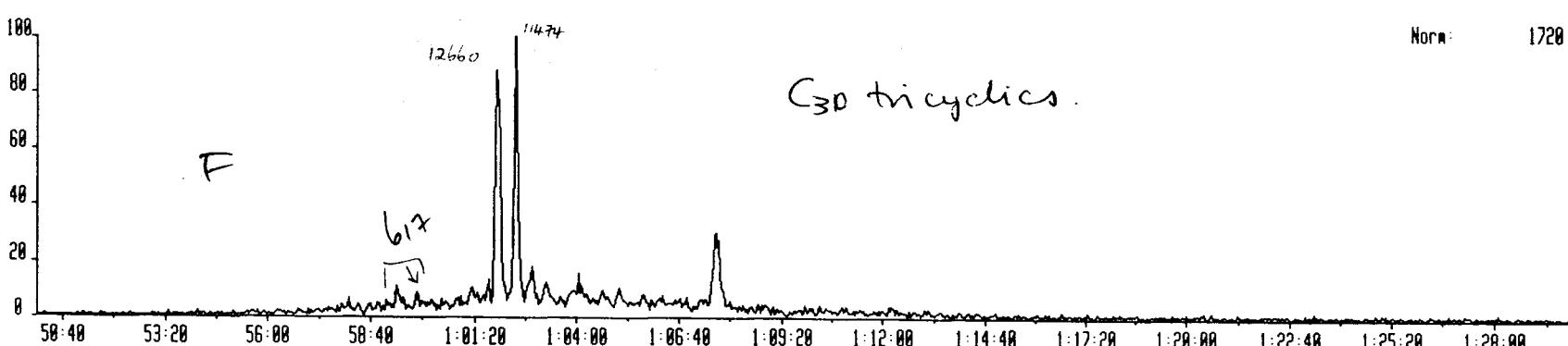
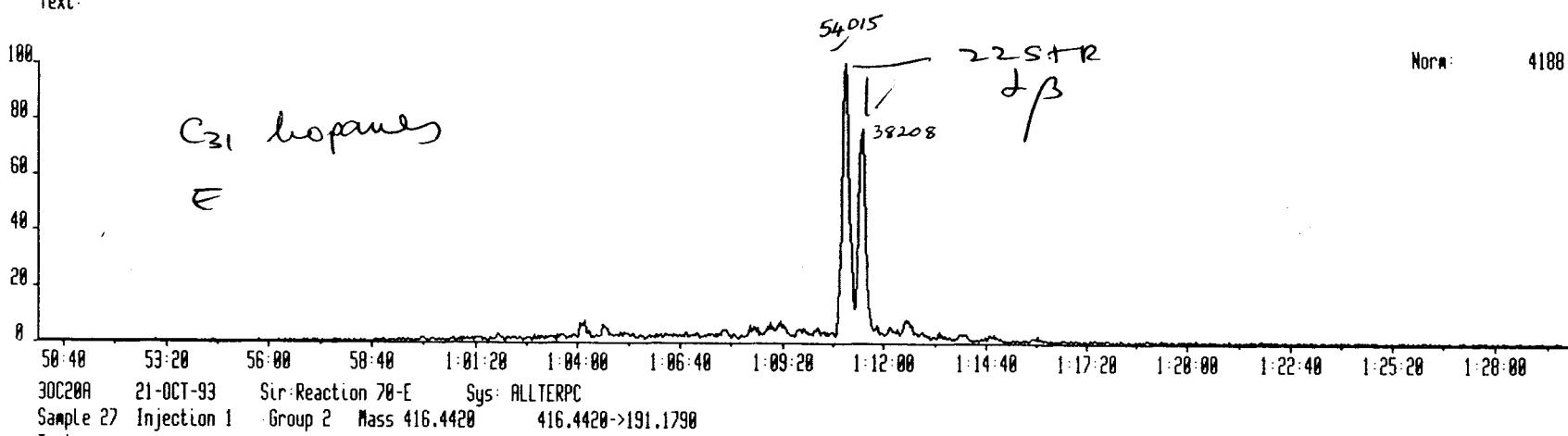
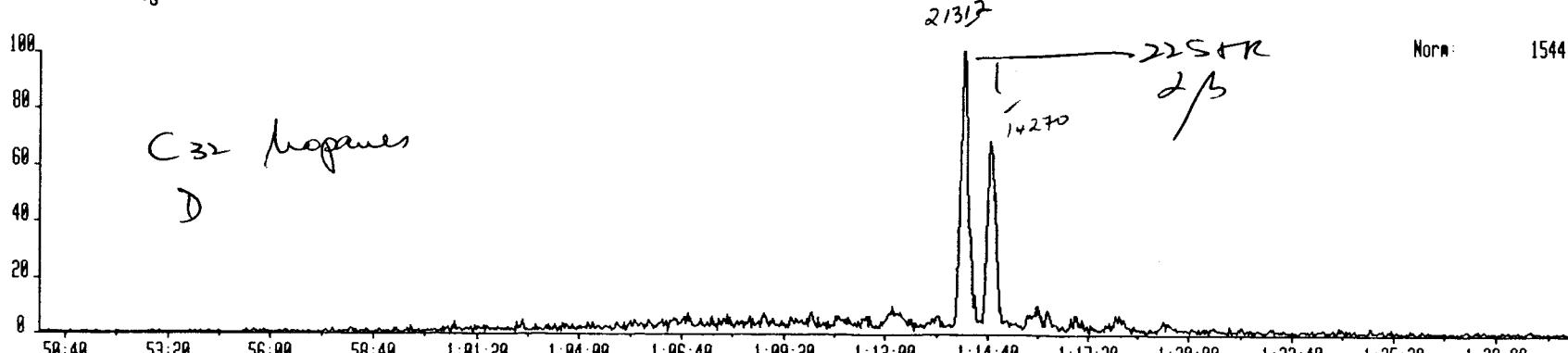
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 7 Injection 1 Group 2 Mass 426.4230 426.4230->383.3550
Text:TONGA SEEP #742



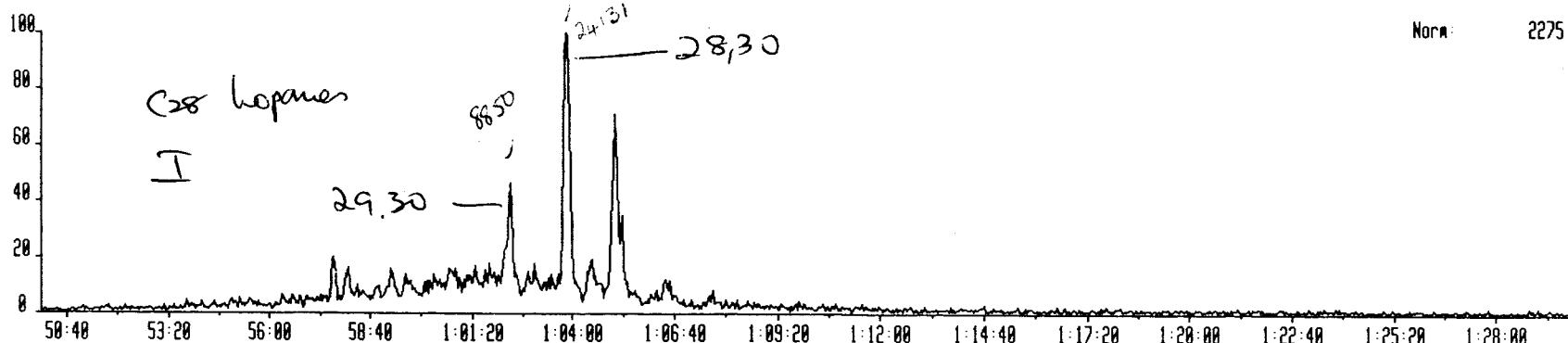
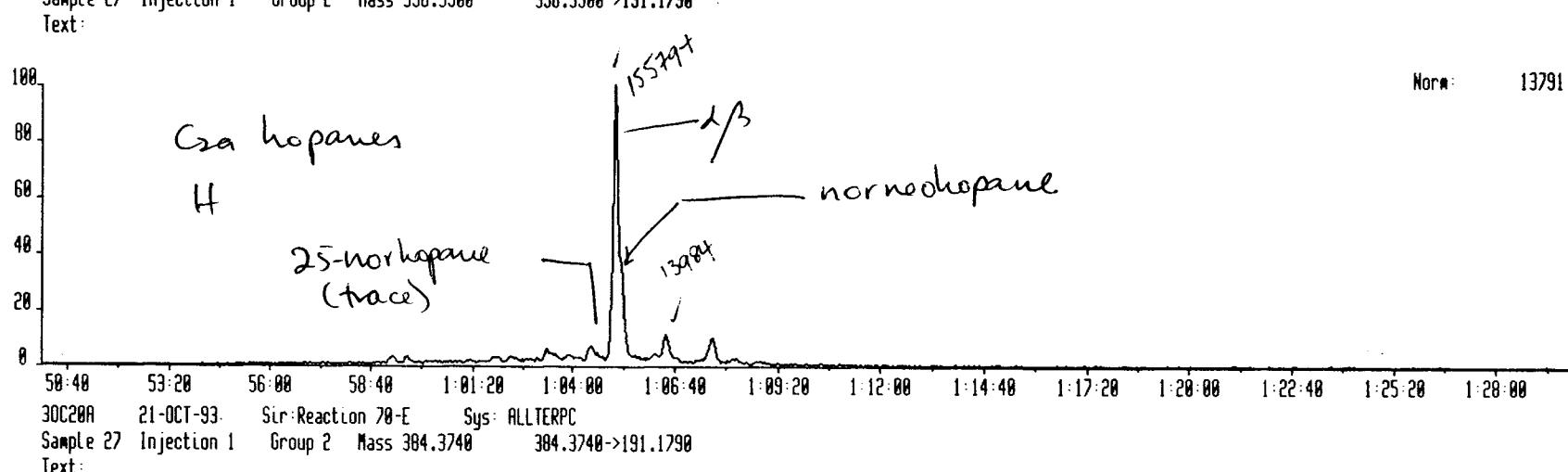
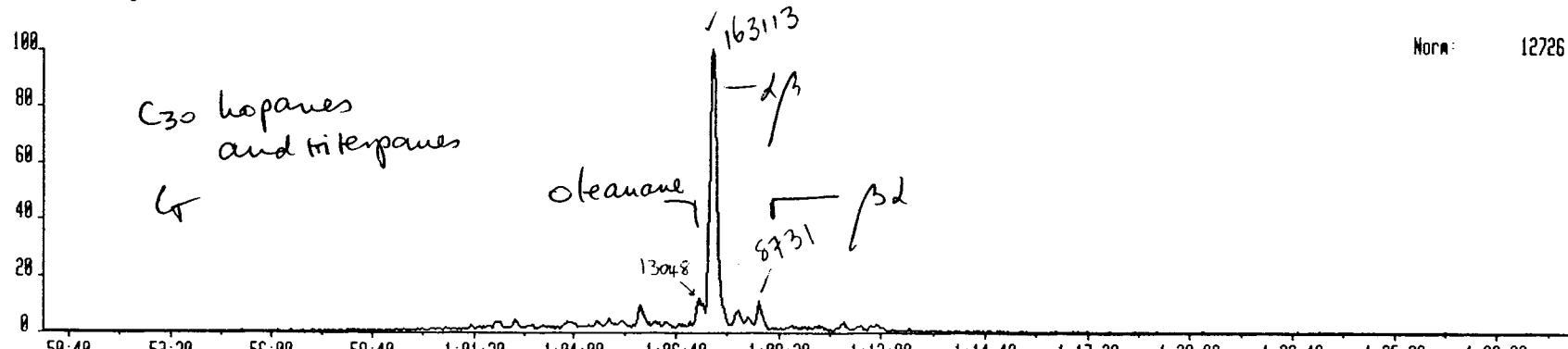
30C20A 21-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 482.4820 482.4820->191.1790
Text: High wave : 2 bottom



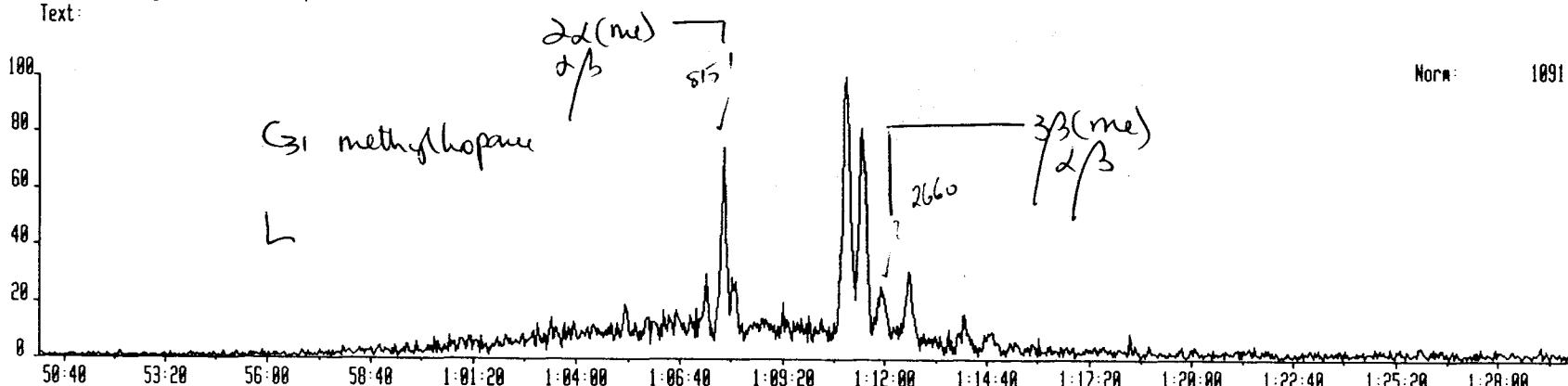
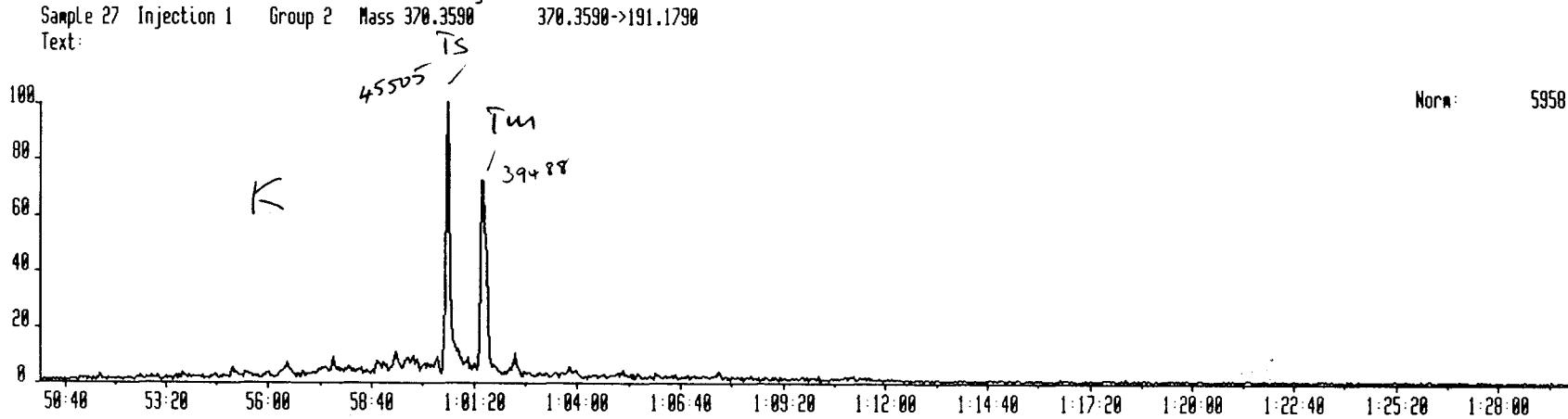
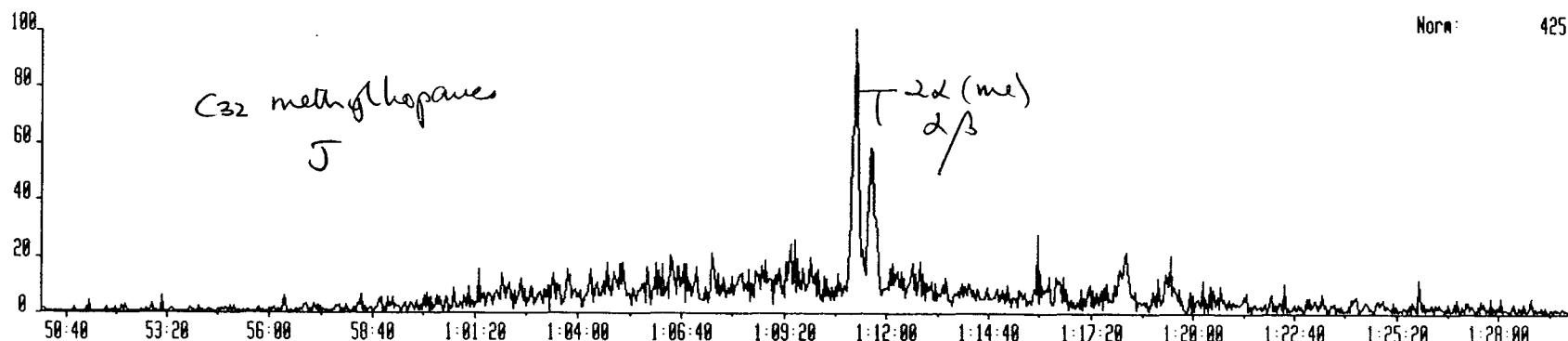
30C20A 21-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 440.4370 440.4370->191.1790
Text: Black water 2 bottom



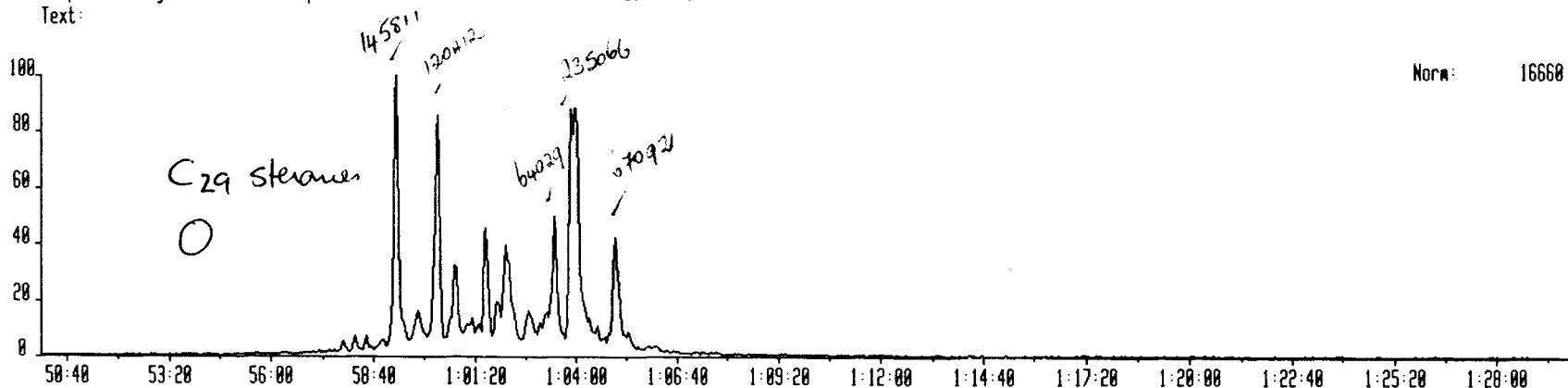
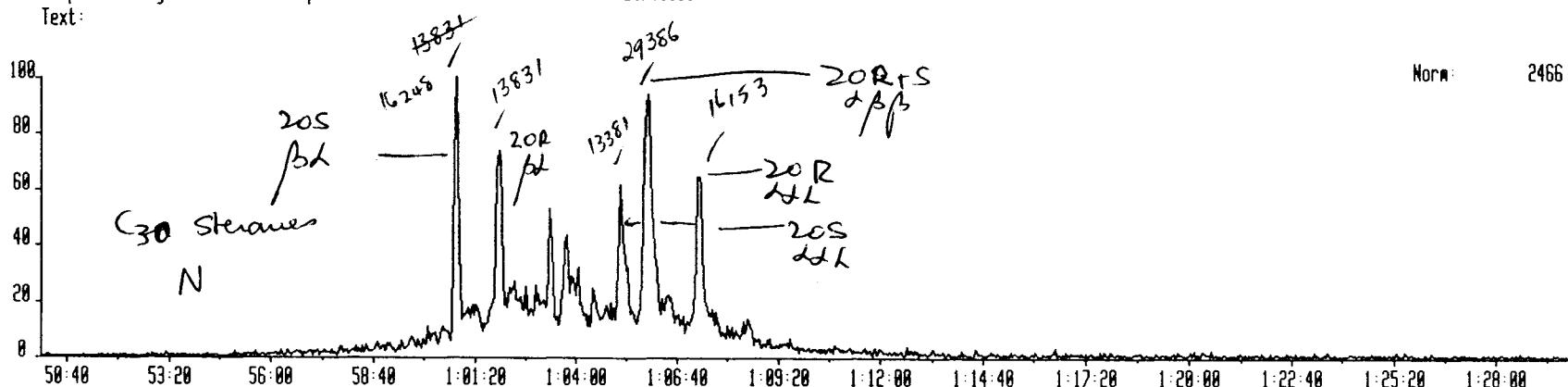
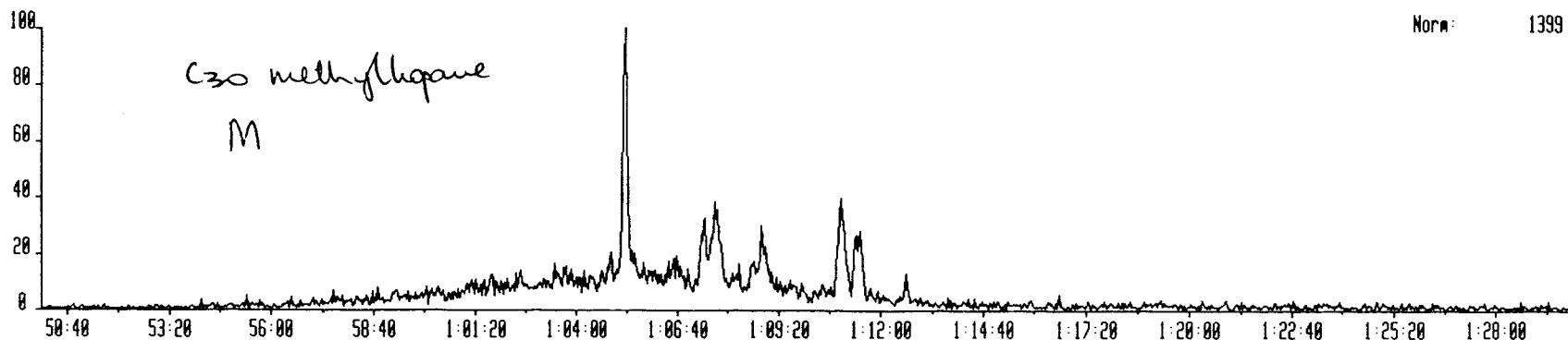
30C20A 21-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 412.4060 412.4060->191.1790
Text: Bligh water 2 Bottom



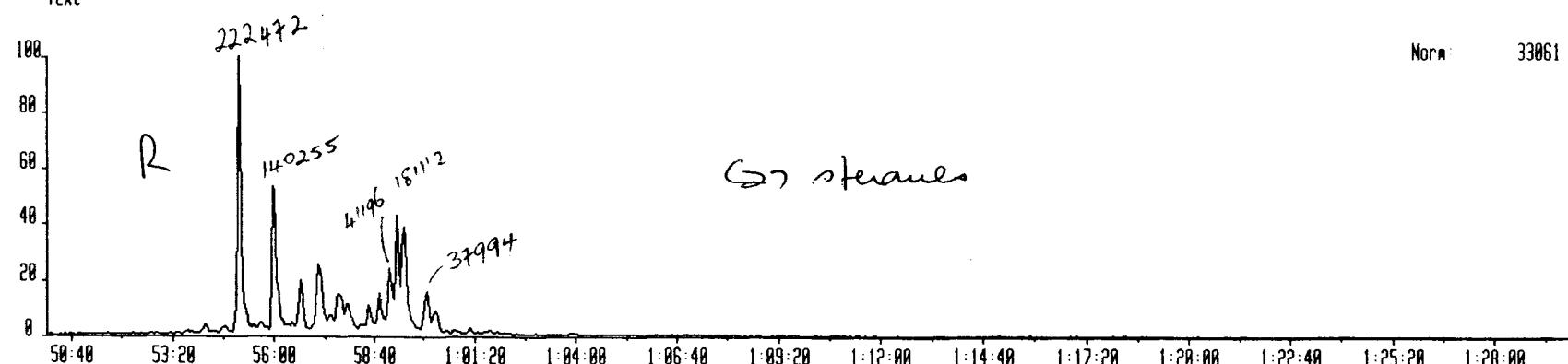
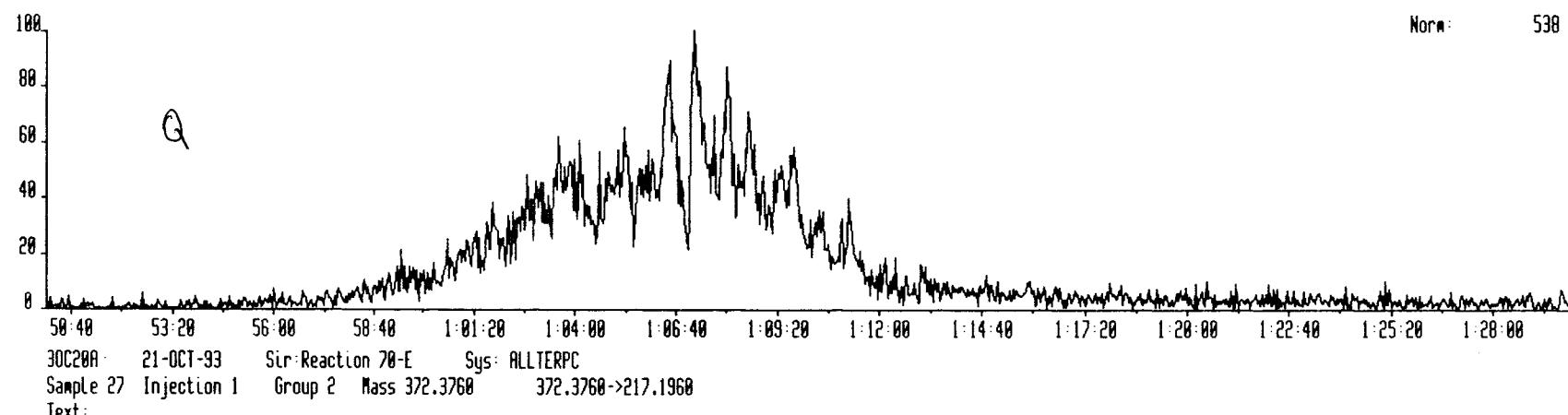
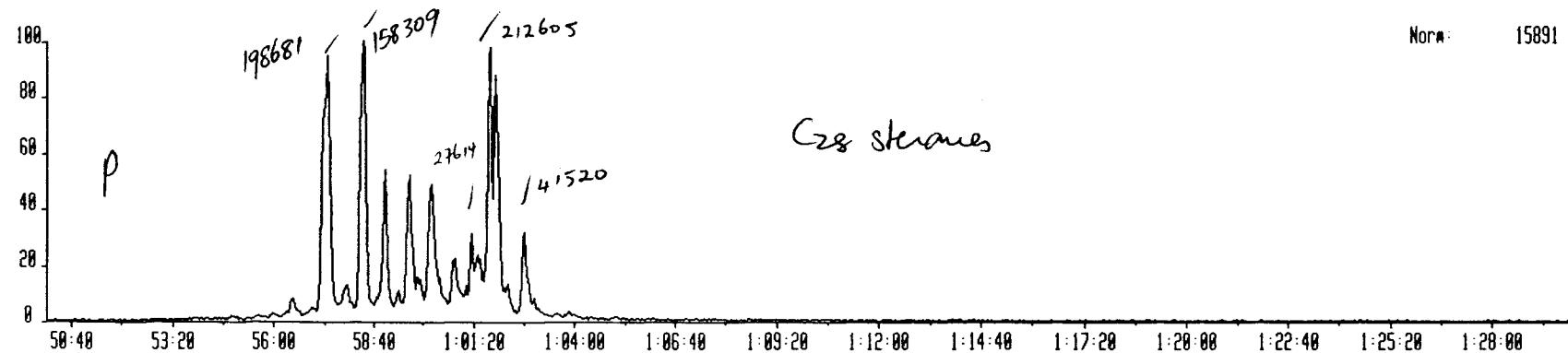
30C20H 21-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 440.4370 440.4370->205.1940
Text: Bligh water : 2 bottom



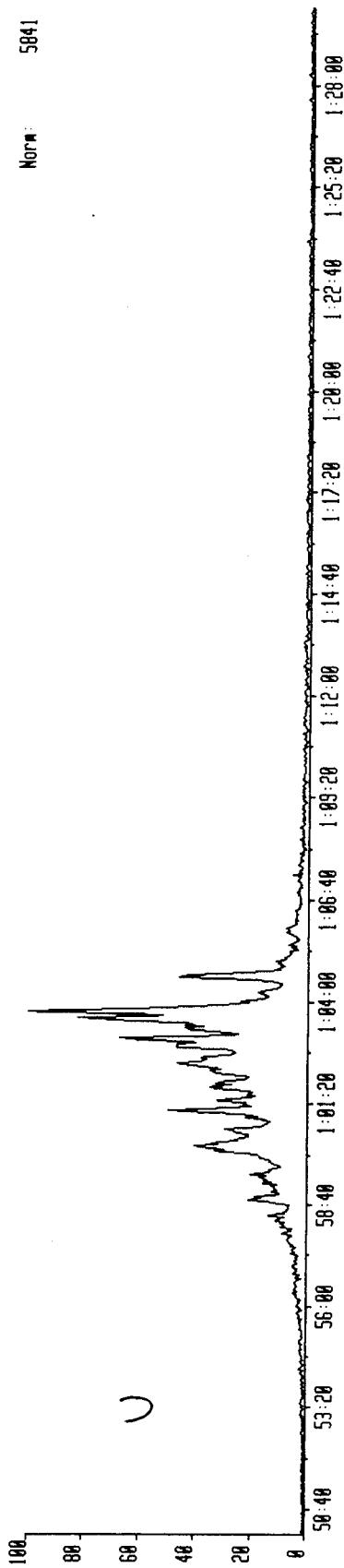
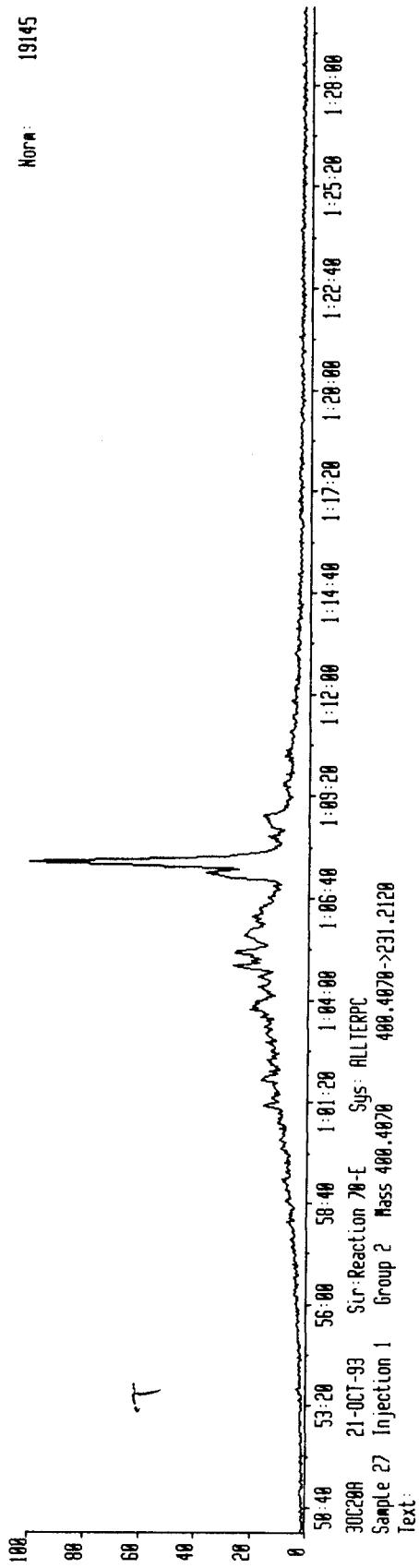
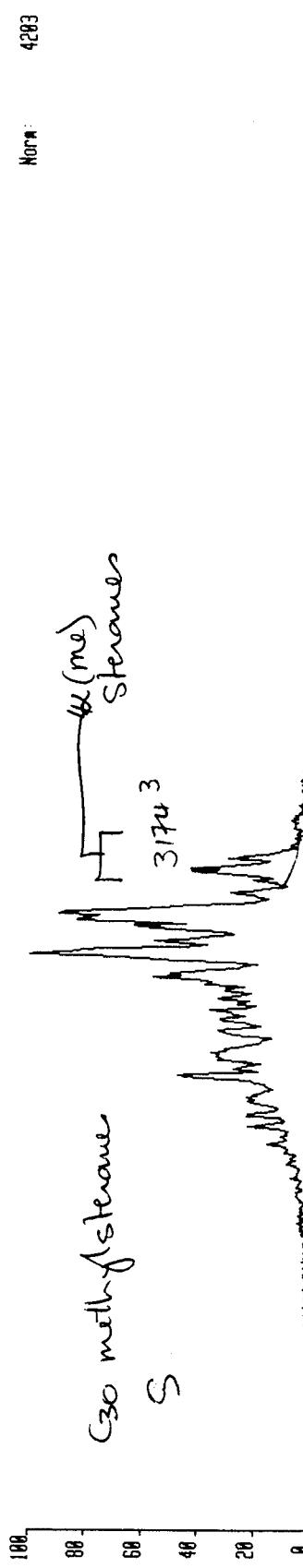
30C20A 21-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 412.4060 412.4060->205.1940
Text: Bligh water: 2 bottom



Section 21-001-05 Ser: Reaction 70-E Log: NELTERPC
Sample 27 Injection 1 Group 2 Mass 386.3910 386.3910->217.1960
Text: B (high water: 2 bottom)

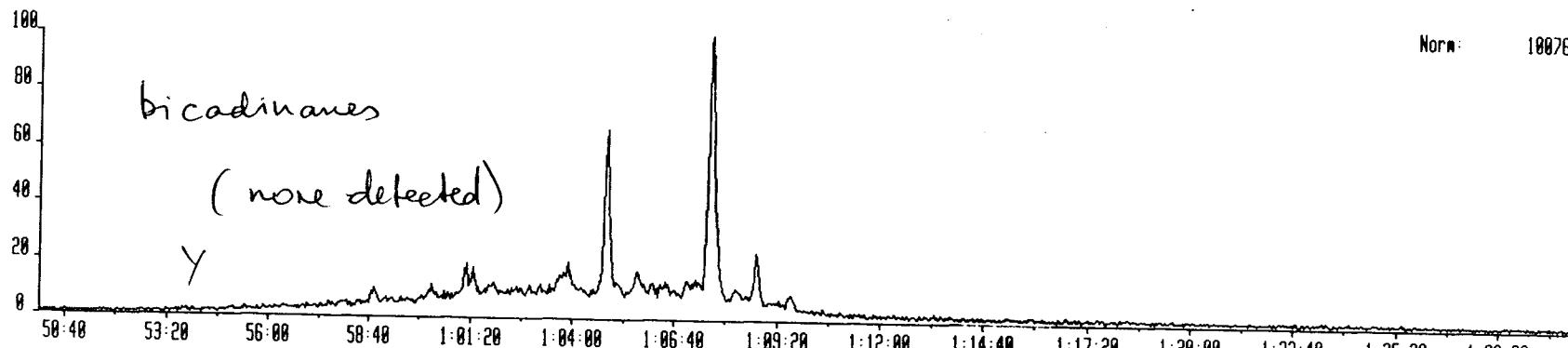


Solution d1-IL-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 27 Injection 1 Group 2 Mass 414.4230 414.4230->231.2120
Text: ~~Brug water~~ 2,5-dione



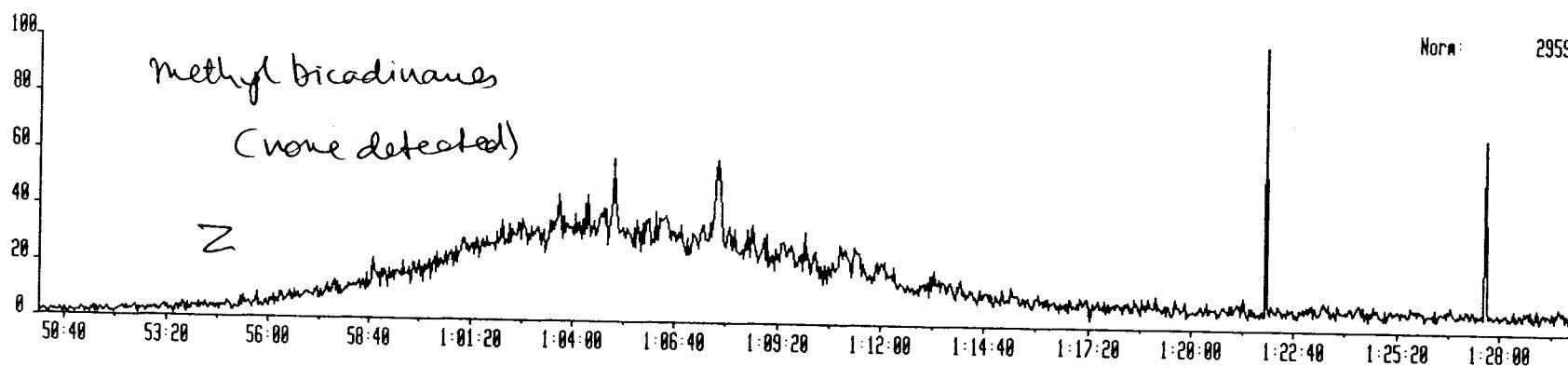
Sample 27 Injection 1 Group 2 Mass 412.4060 412.4060->369.3400
Text: Bligh water z bottom

Norm: 18076

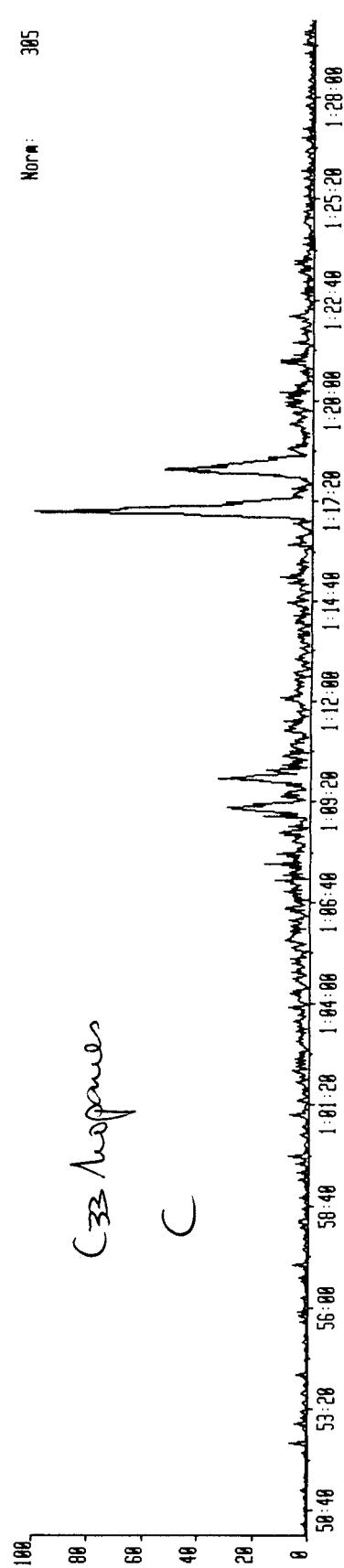
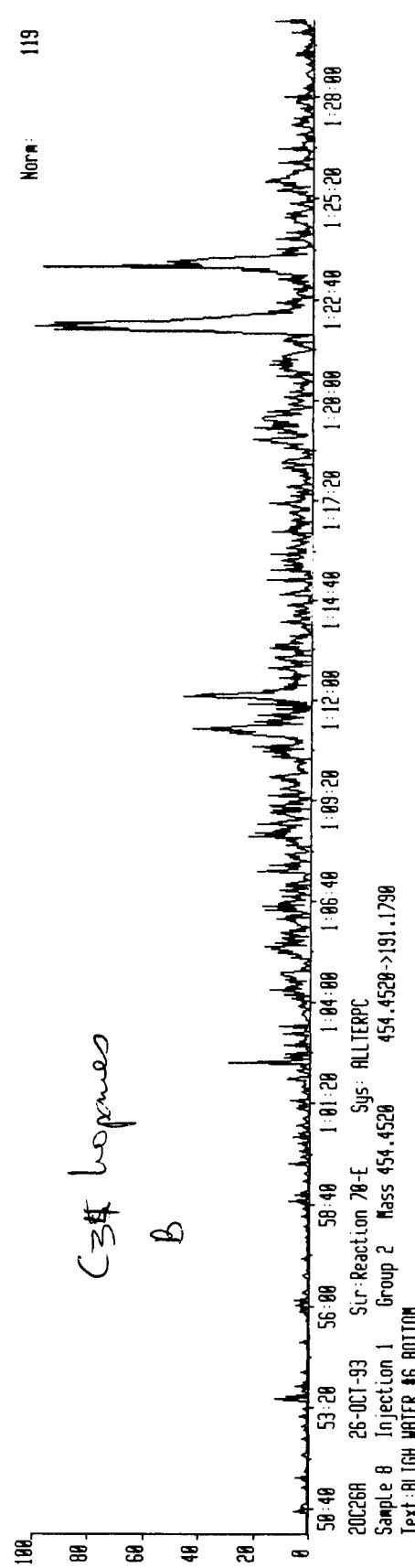
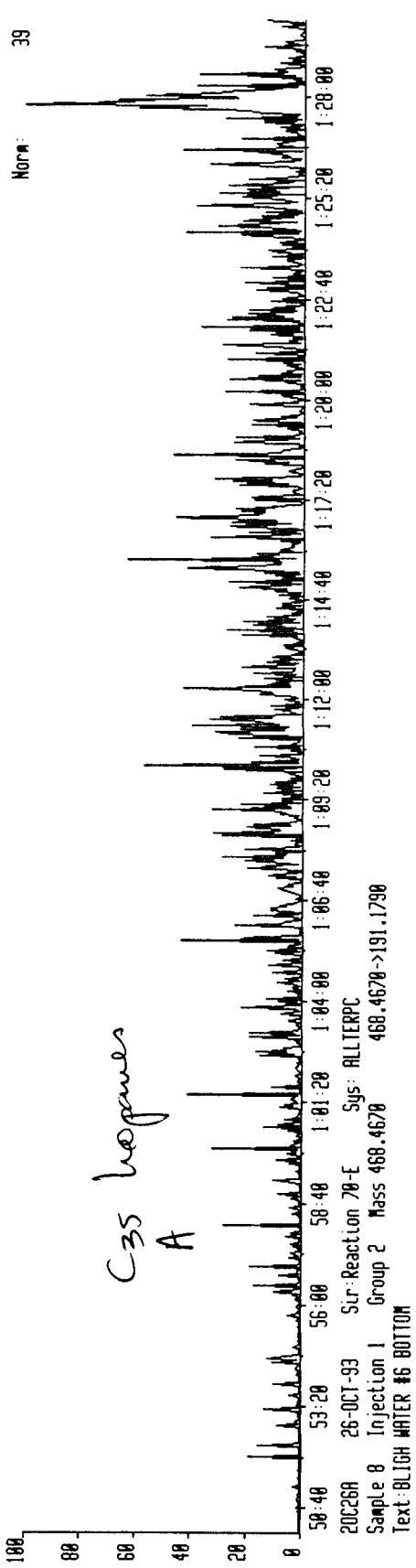


Sample 27 Injection 1 Group 2 Mass 426.4230 426.4230->383.3550
Text:

Norm: 2959

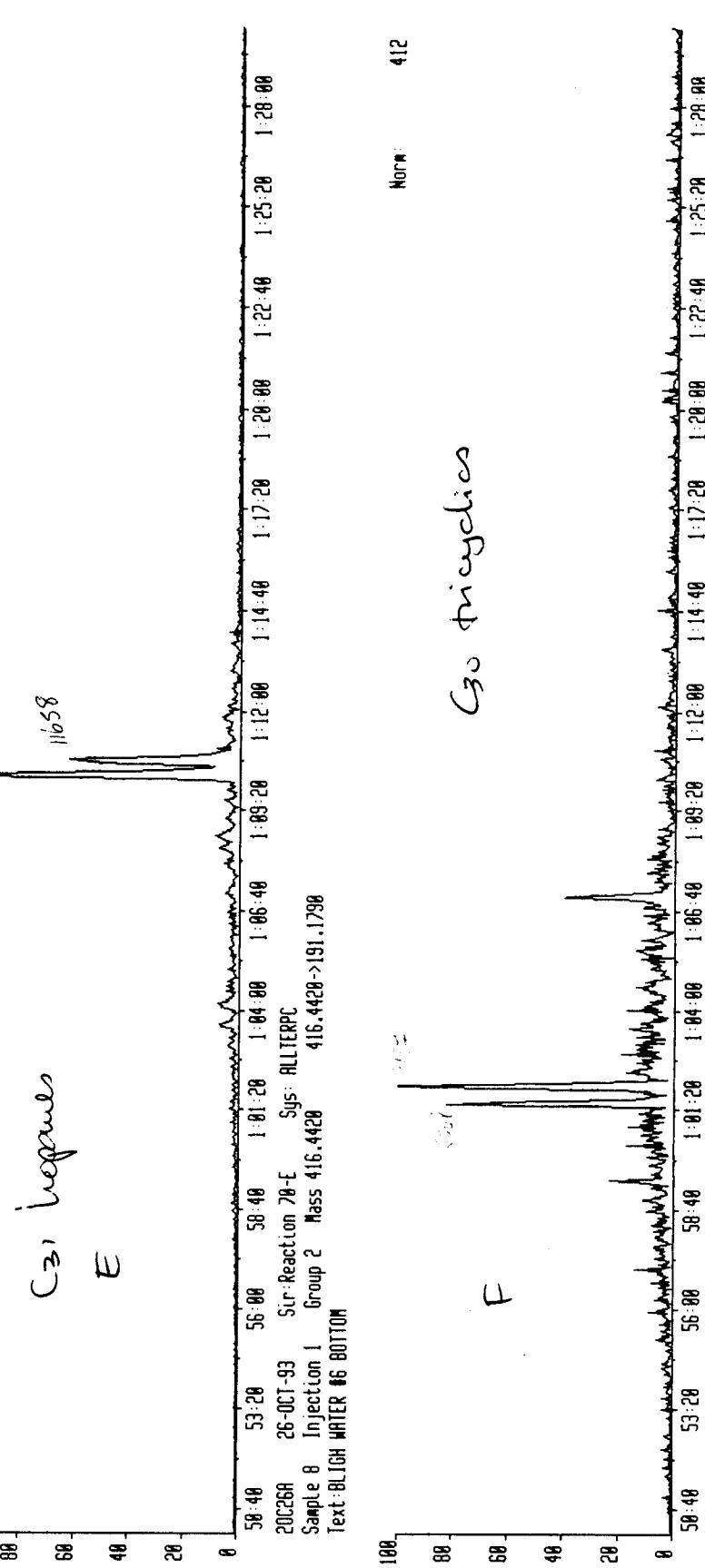
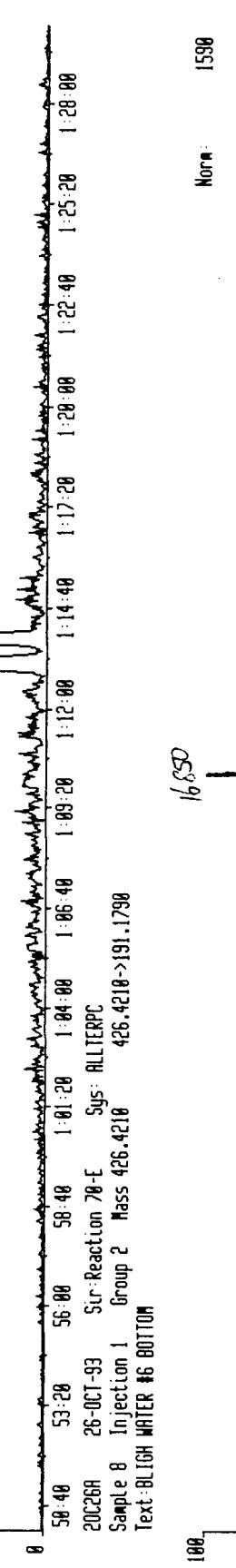
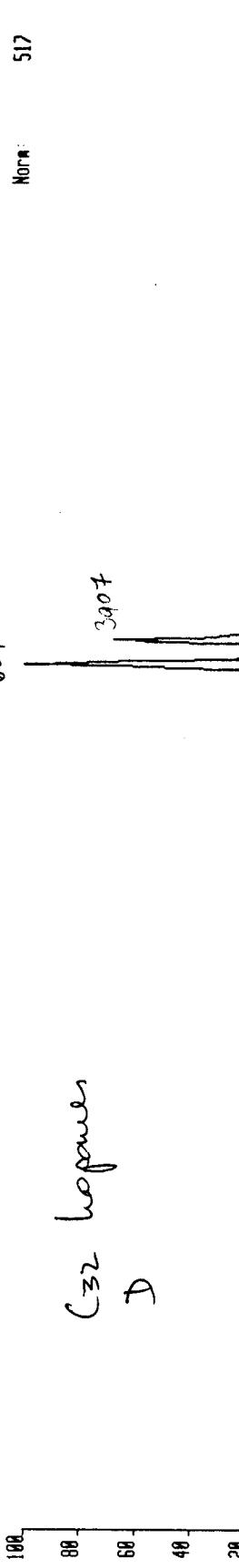


20C28A 26-OCT-93 Sur:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 402.4020 402.4020->191.1798
Text:BLIGH WATER #6 BOTTOM

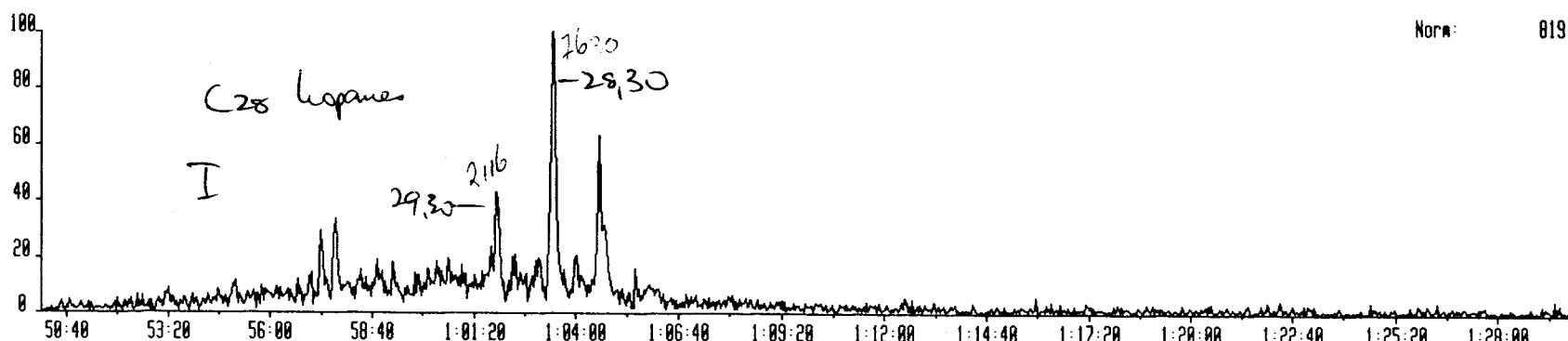
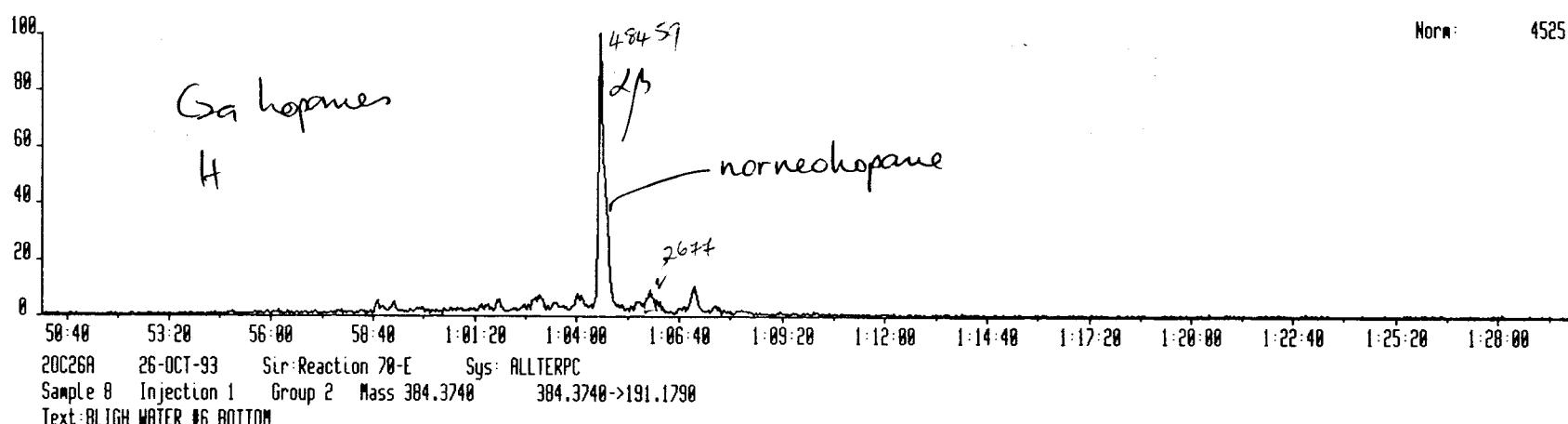
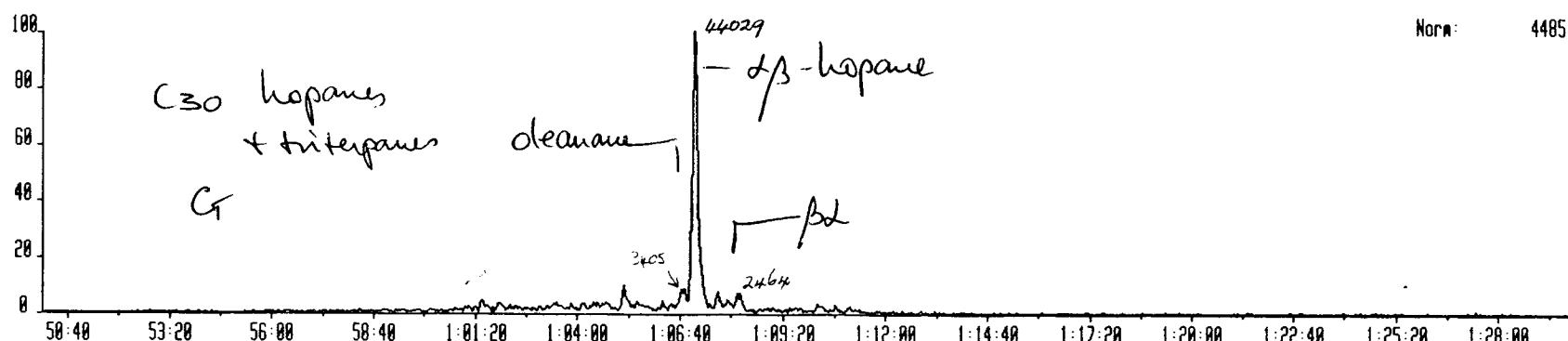


20268 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 400.4370 400.4370->191.1790
Text:BLIGH WATER #6 BOTTOM

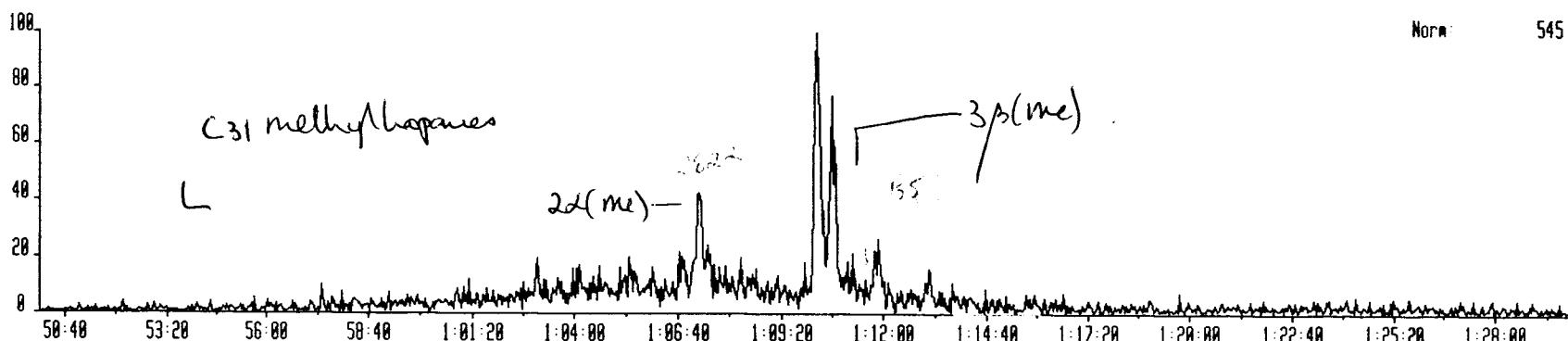
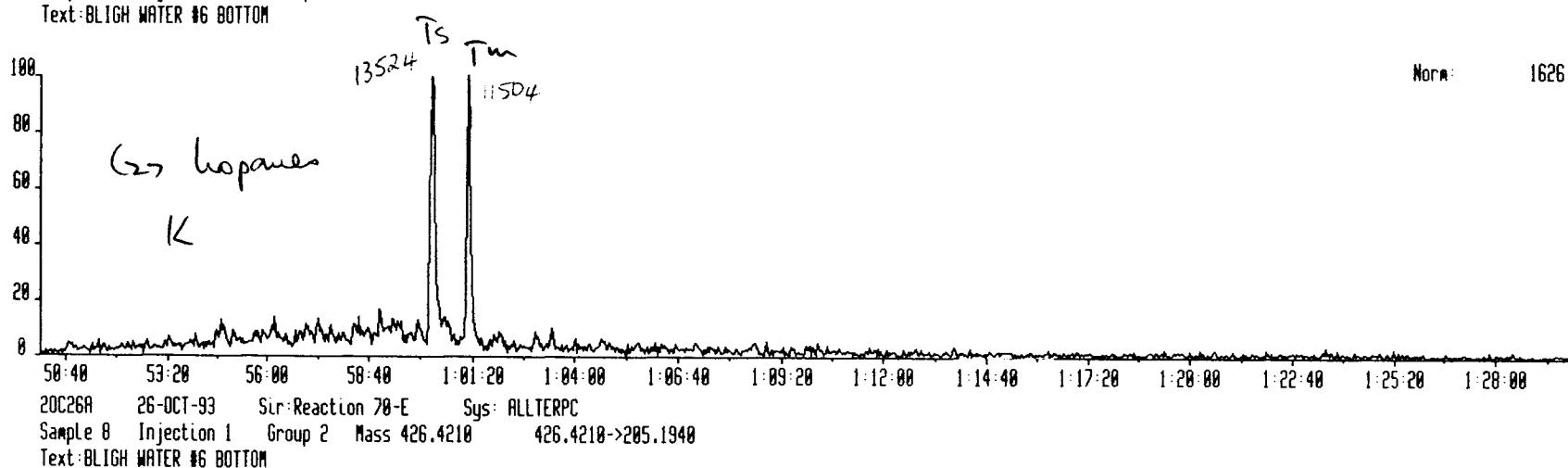
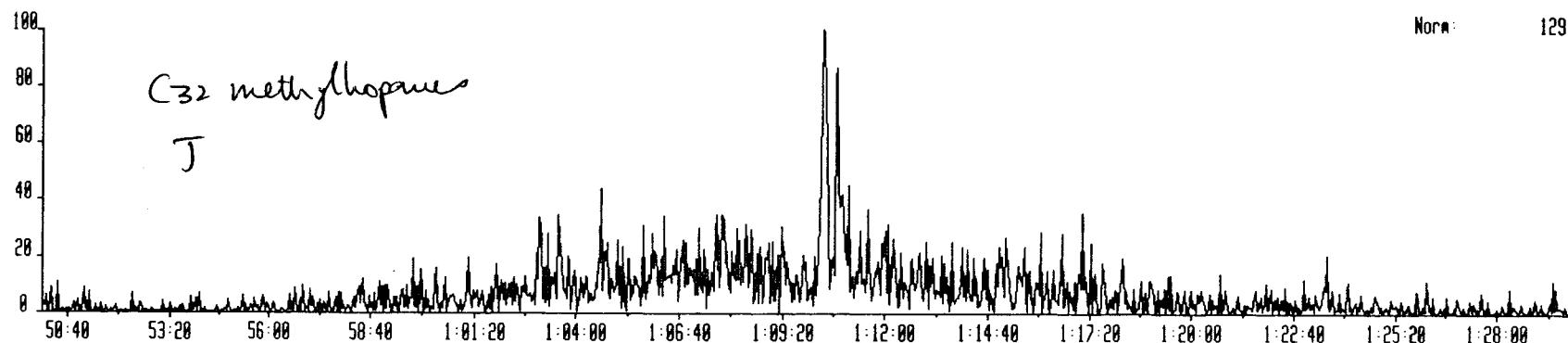
6092



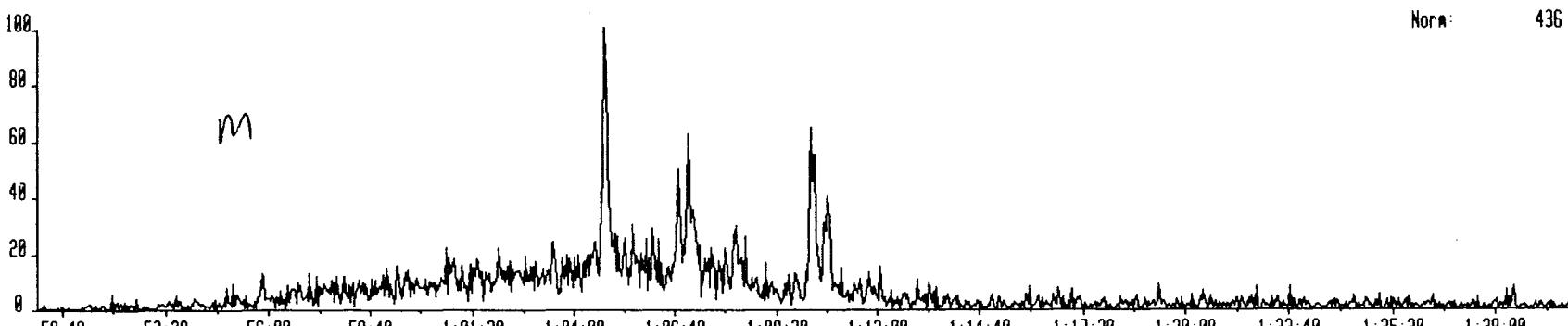
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 412.4060 412.4060->191.1790
Text:BLIGH WATER #6 BOTTOM



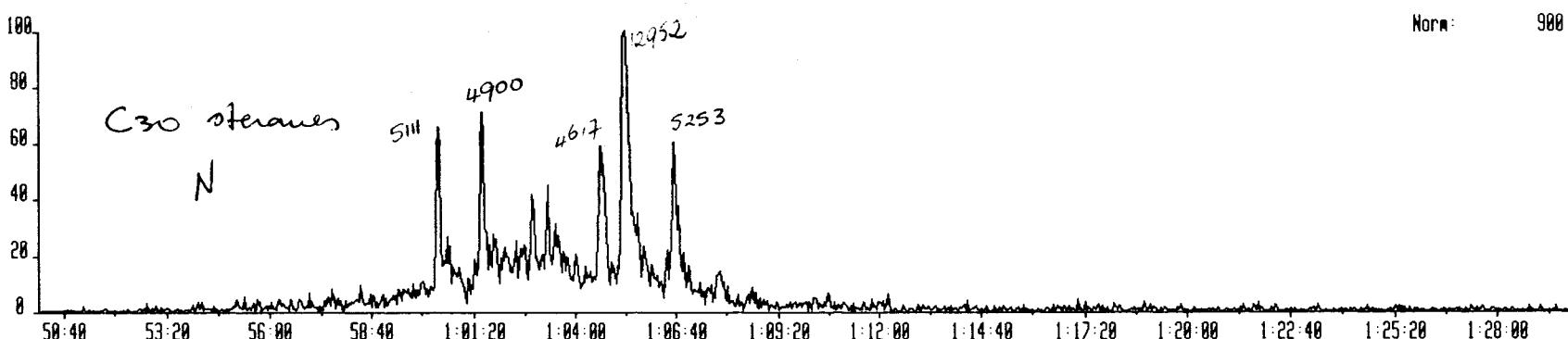
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 440.4378 440.4378->205.1940
Text:BLIGH WATER #6 BOTTOM



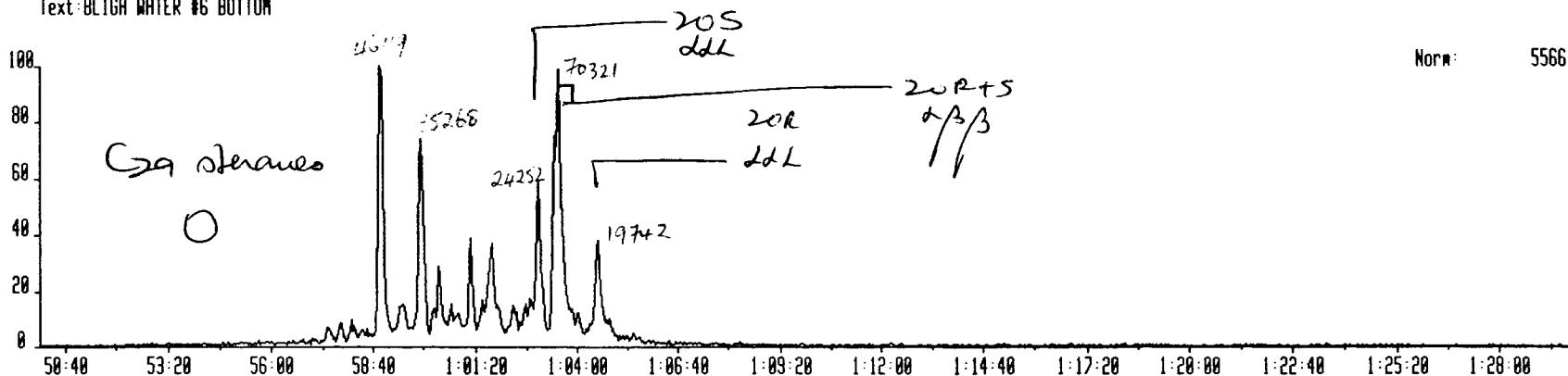
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 412.4060 412.4060->205.1940
Text:BLIGH WATER #6 BOTTOM



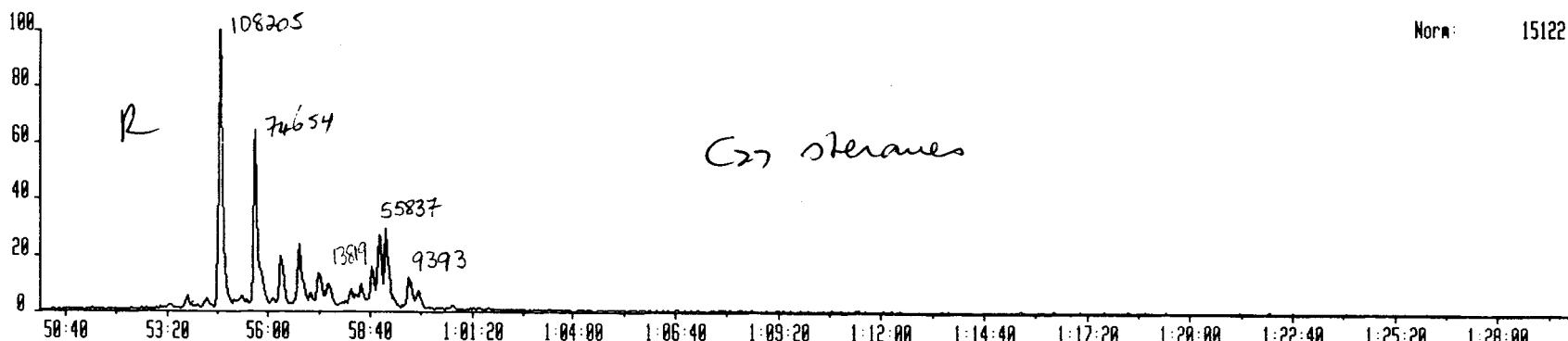
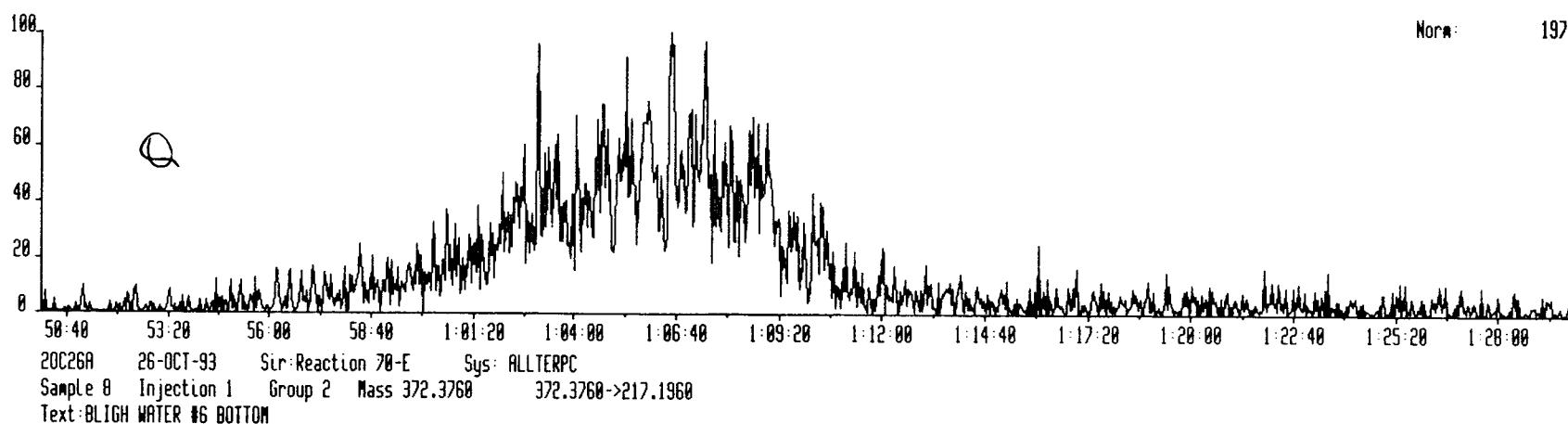
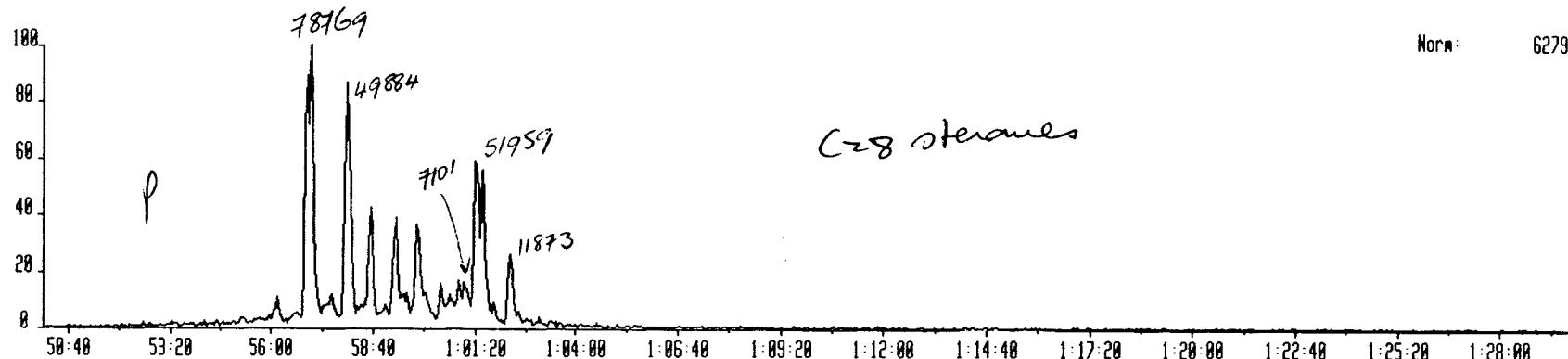
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 414.4230 414.4230->217.1960
Text:BLIGH WATER #6 BOTTOM



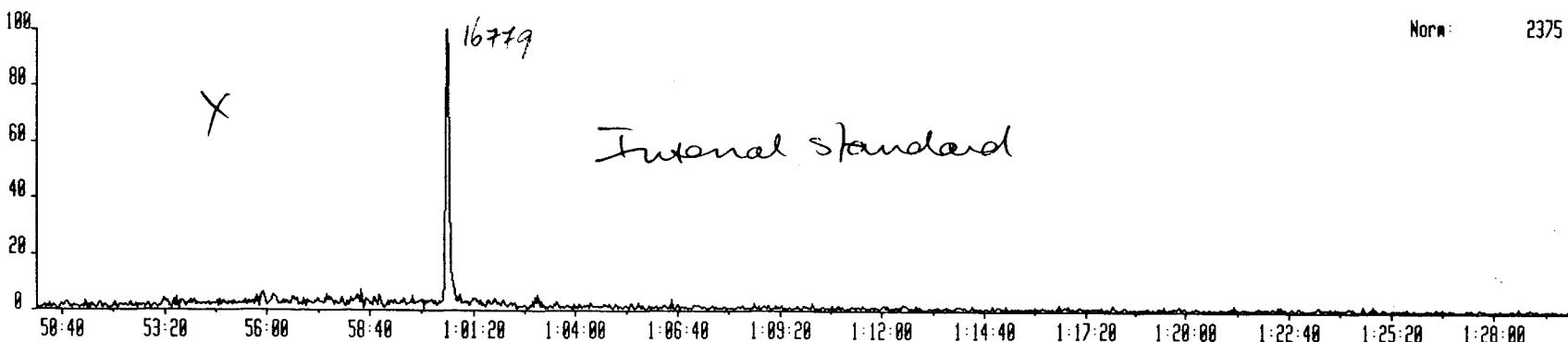
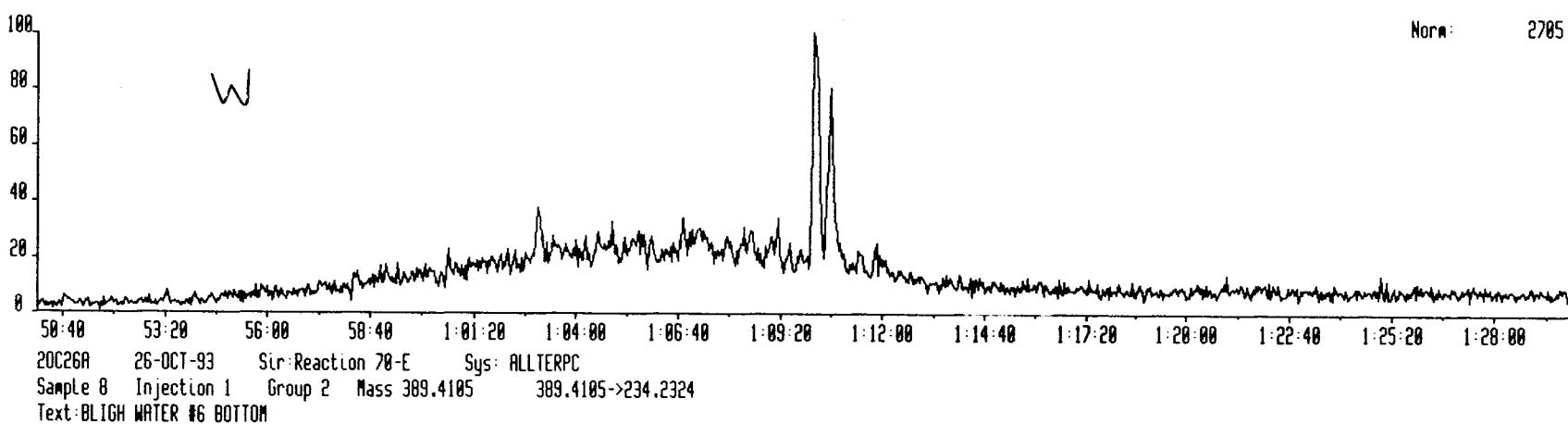
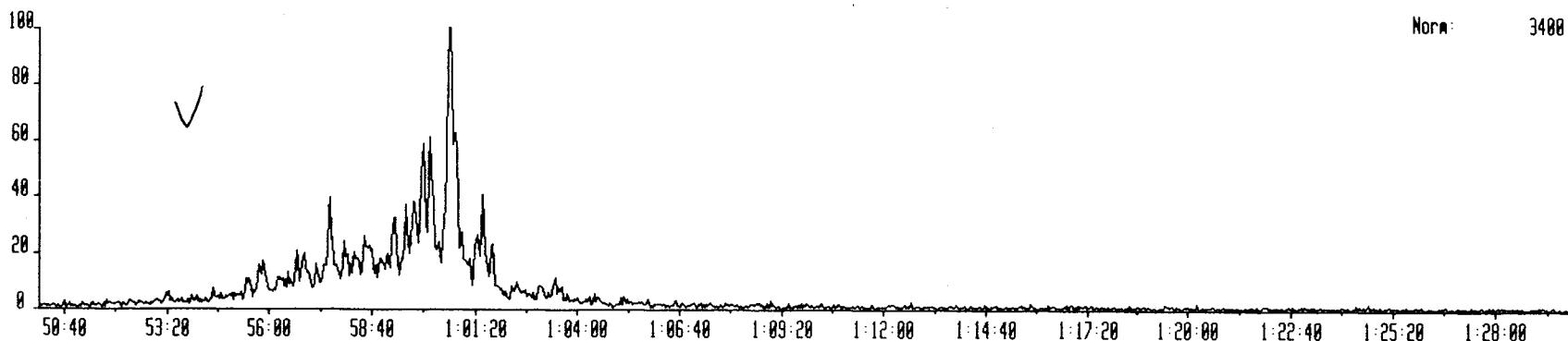
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 400.4070 400.4070->217.1960
Text:BLIGH WATER #6 BOTTOM



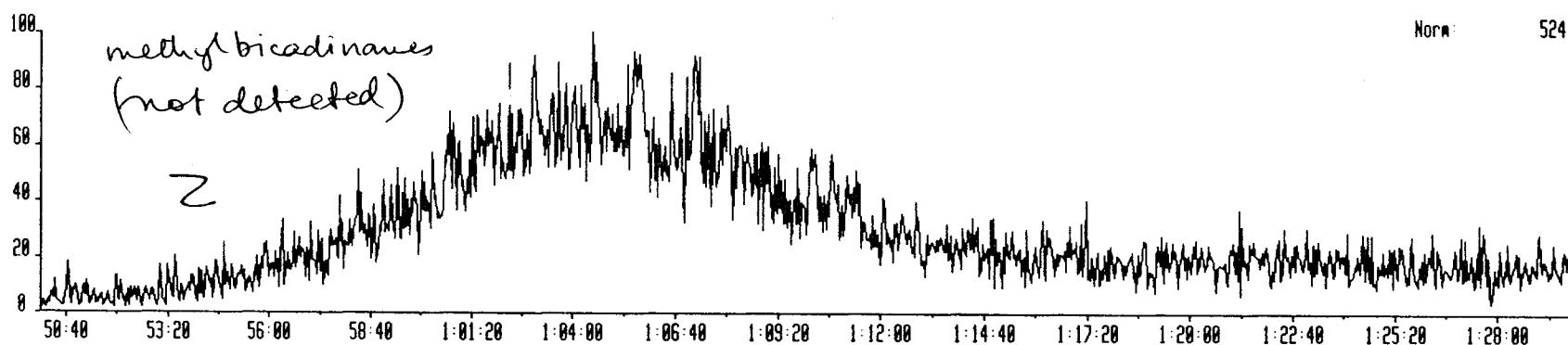
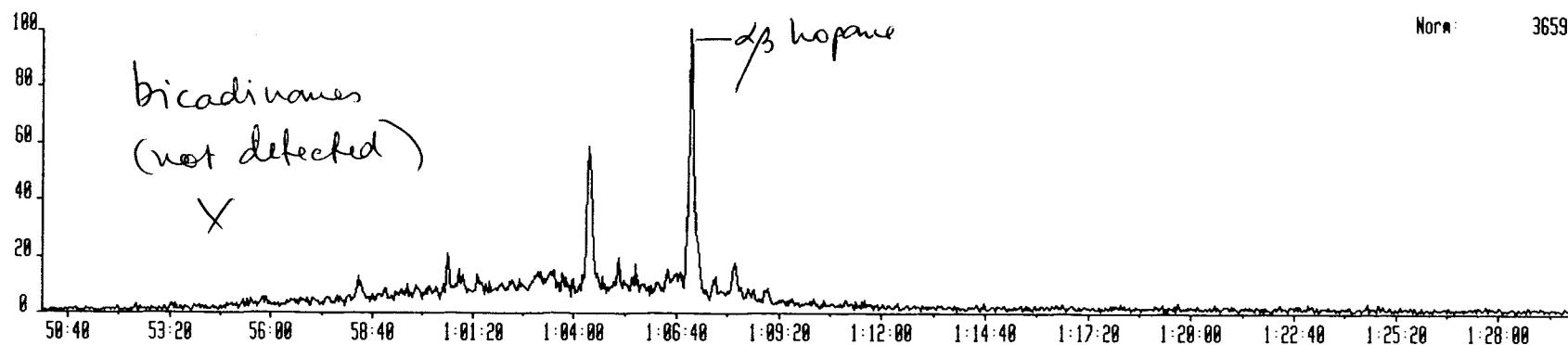
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 386.3910 386.3910->217.1960
Text:BLIGH WATER #6 BOTTOM



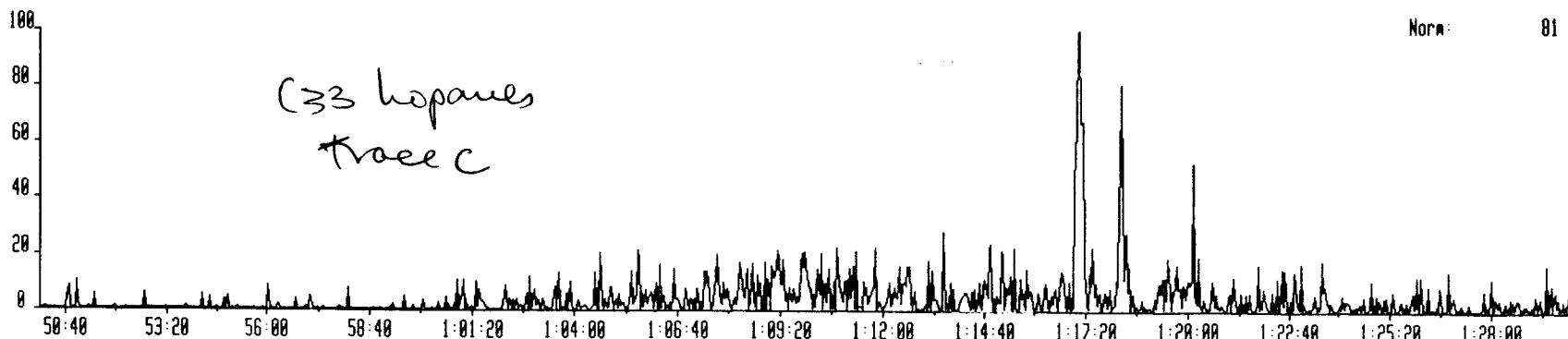
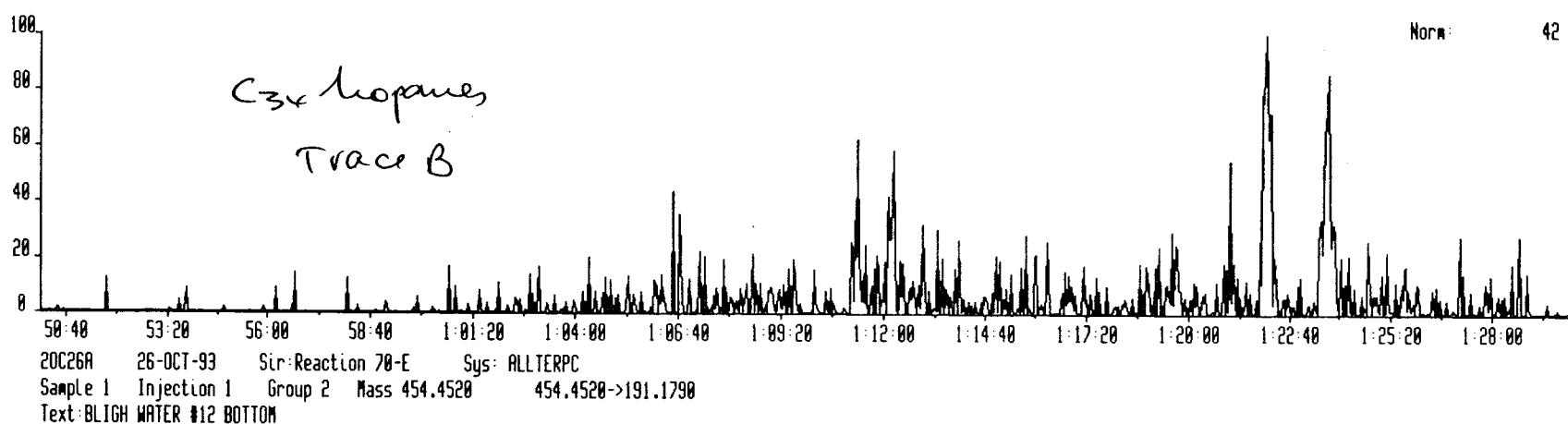
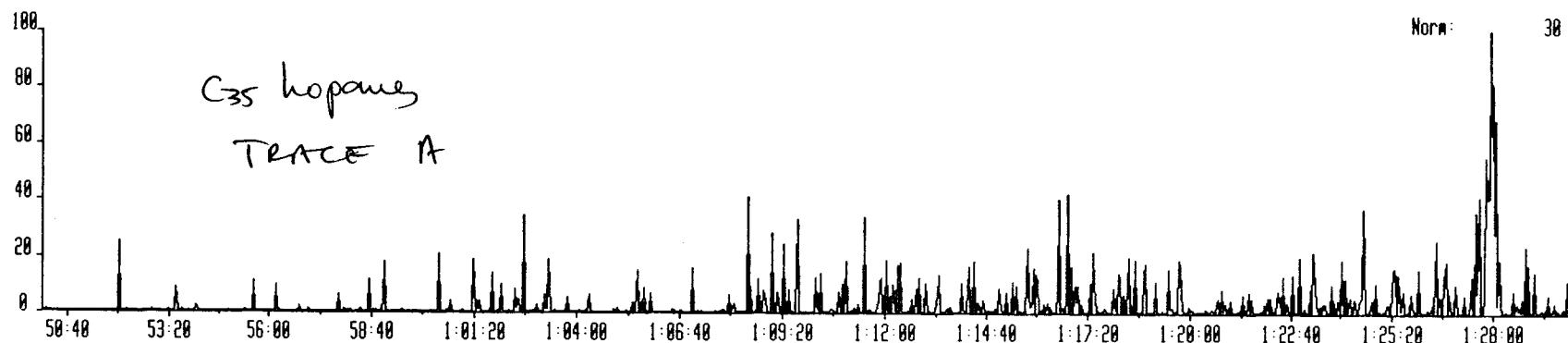
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 386.3910 386.3910->231.2120
Text:BLIGH WATER #6 BOTTOM



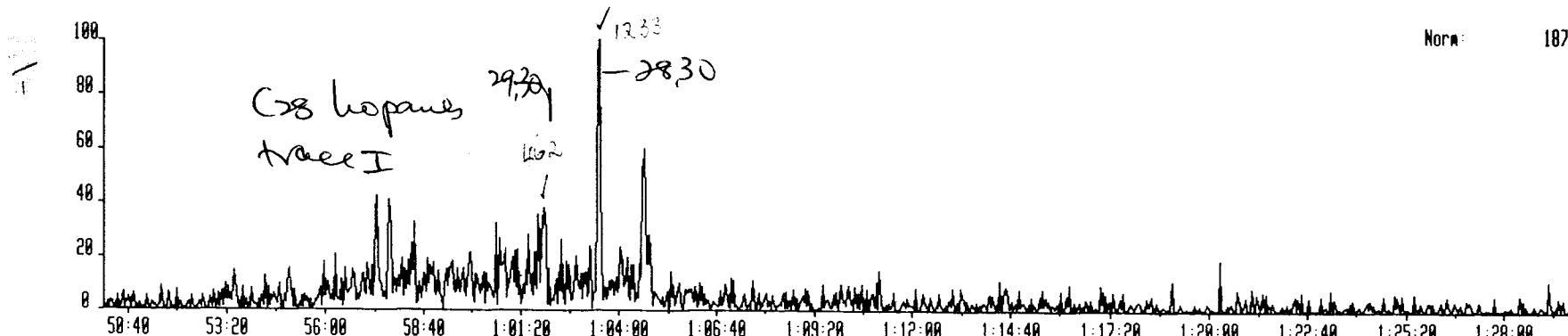
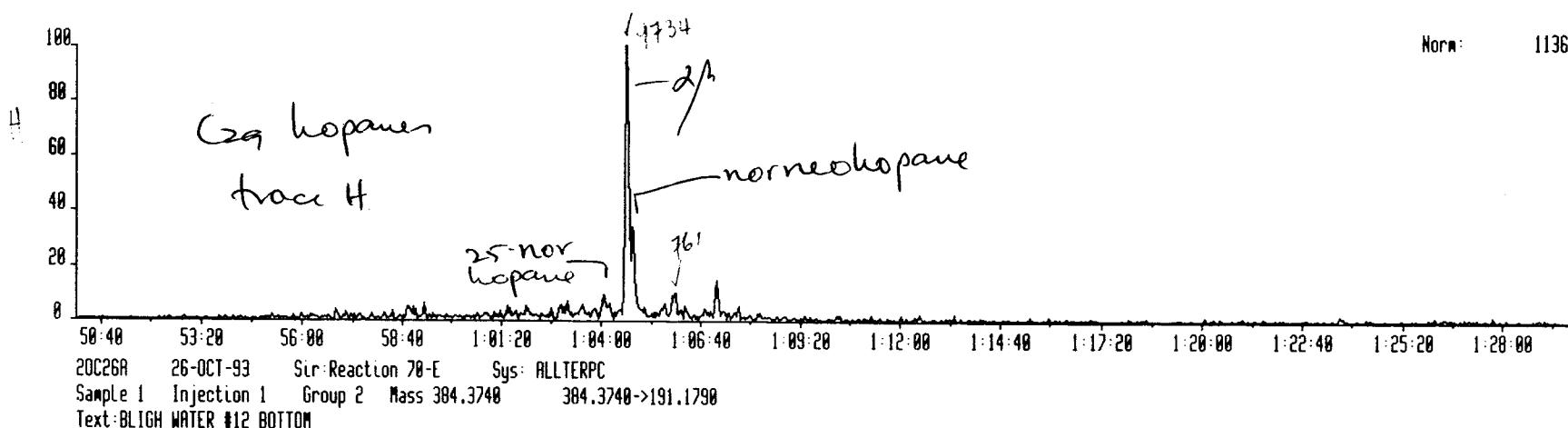
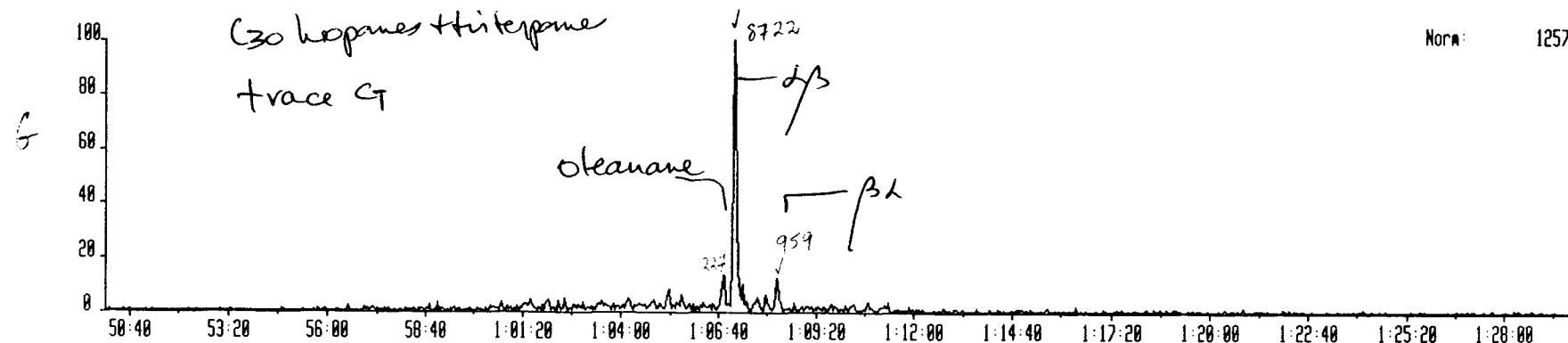
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 8 Injection 1 Group 2 Mass 412.4068 412.4068->369.3408
Text:BLIGH WATER #6 BOTTOM



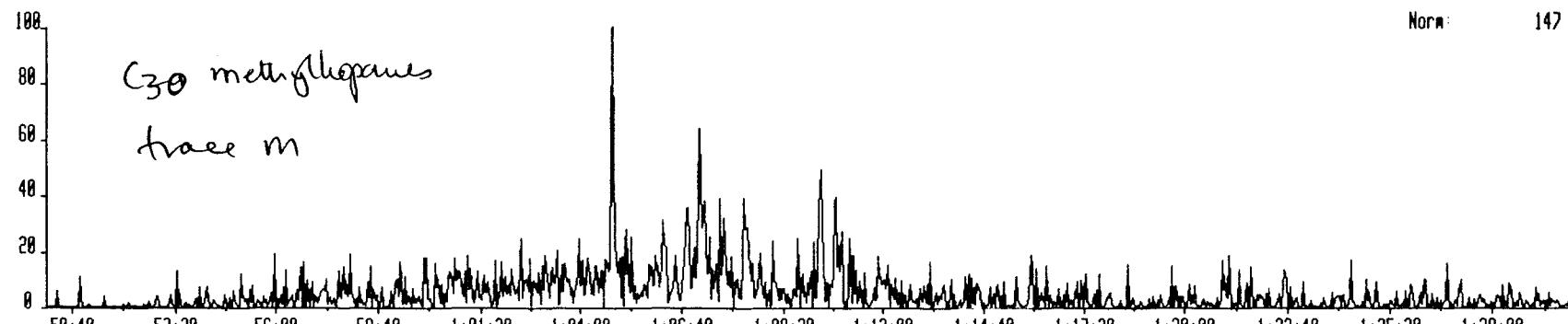
20C6B 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 1 Injection 1 Group 2 Mass 482.4820 482.4820->191.1790
Text:BLIGH WATER #12 BOTTOM



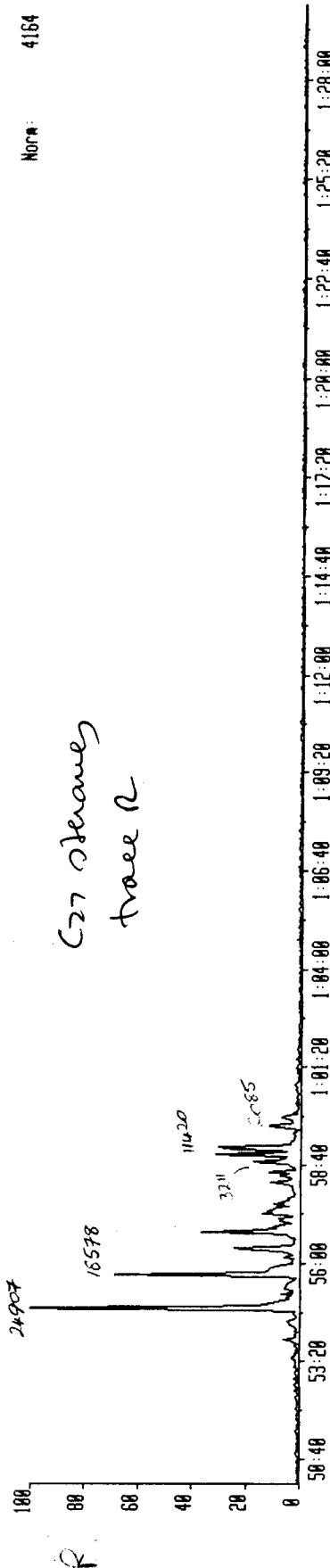
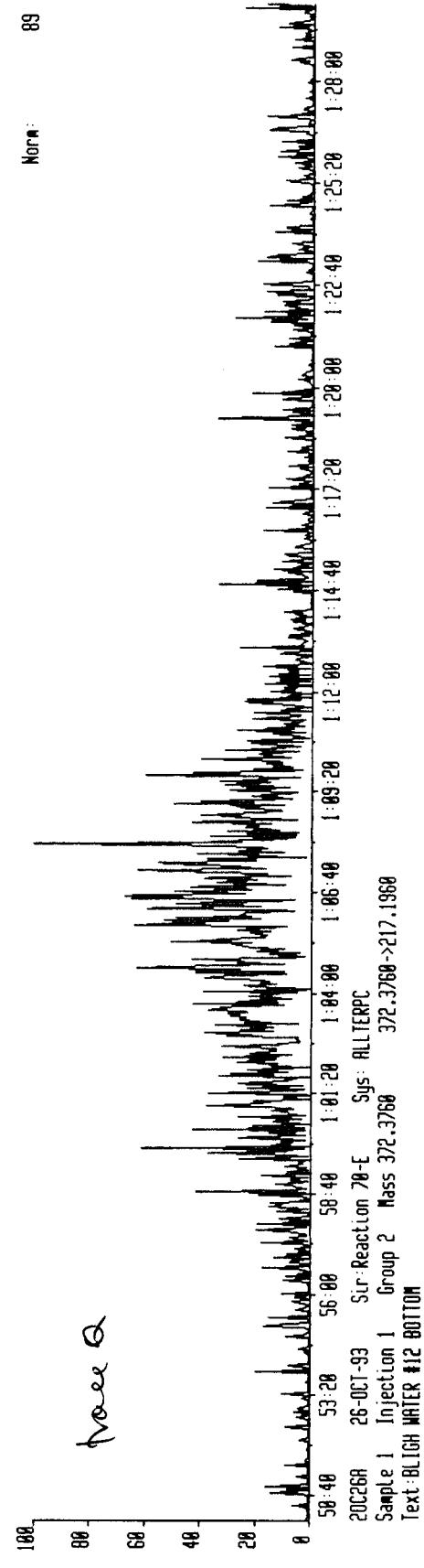
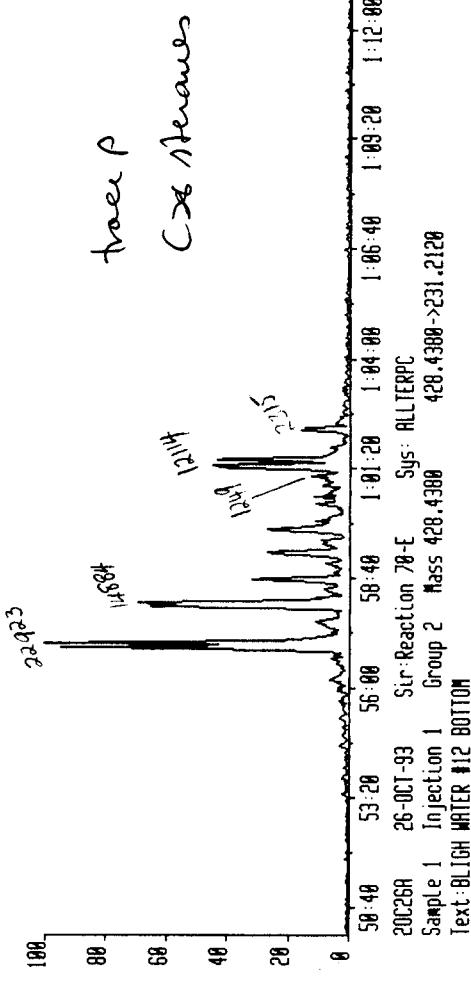
20C26A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 1 Injection 1 Group 2 Mass 412.4060 412.4060->191.1790
Text:BLIGH WATER #12 BOTTOM



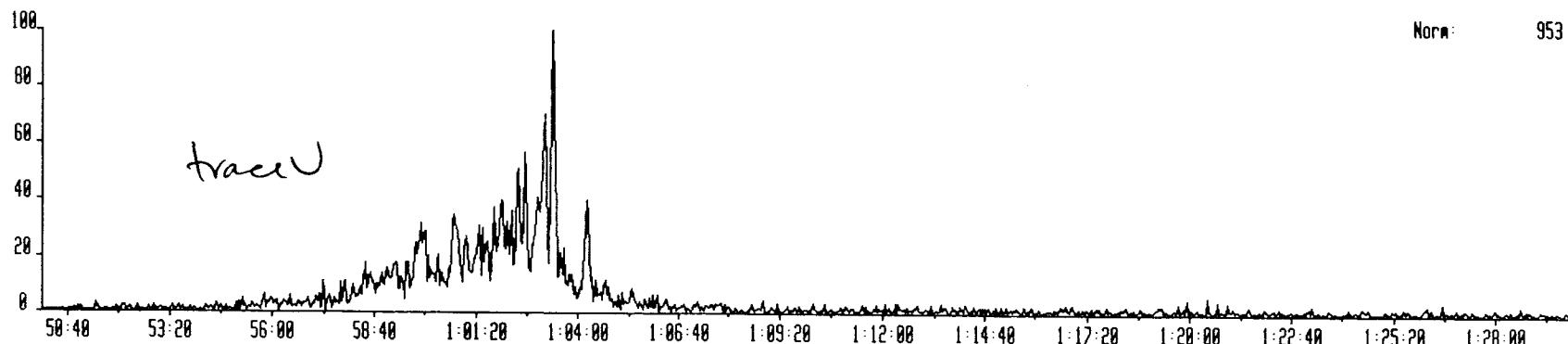
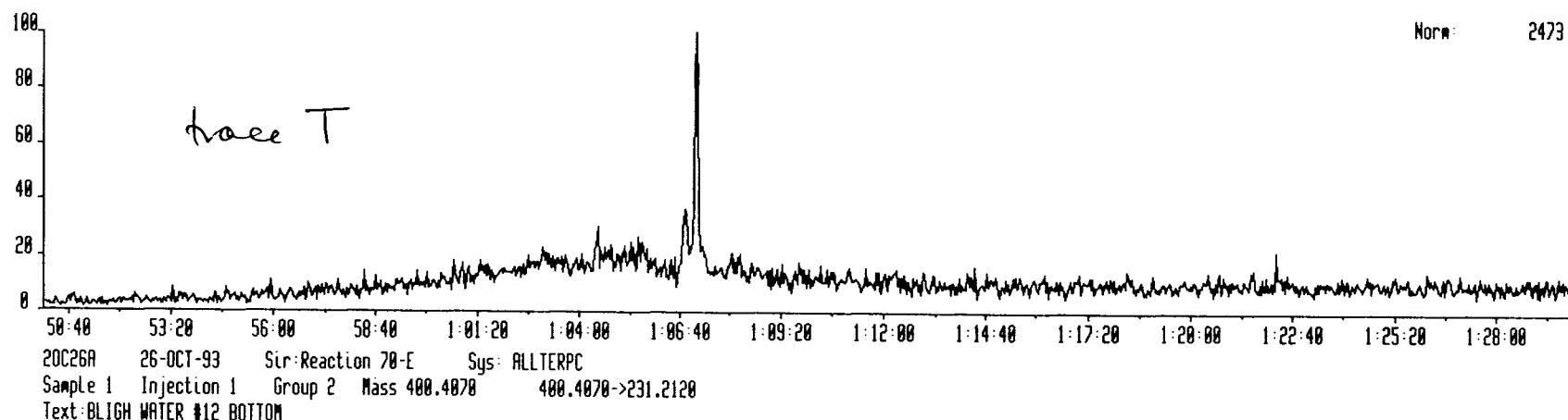
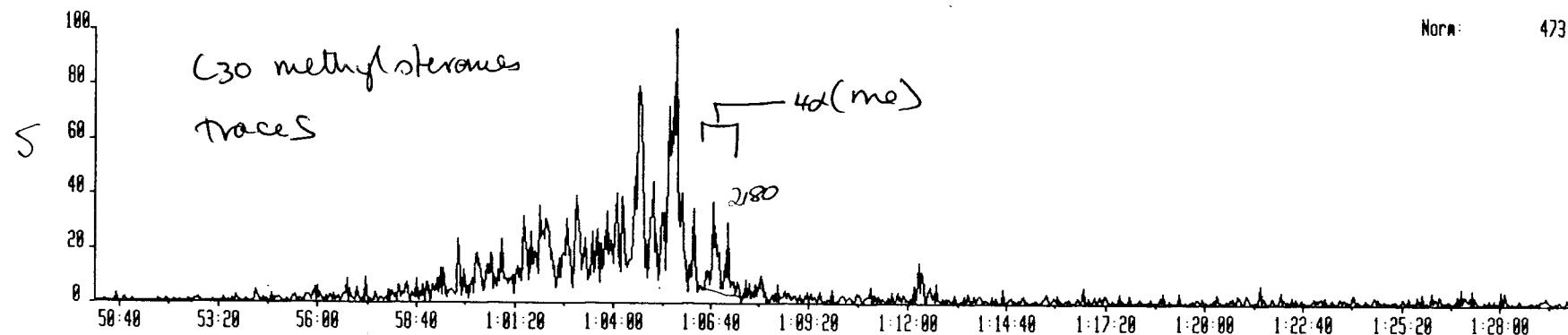
Sample 1 Injection 1 Group 2 Mass 412.4060 412.4060->205.1940
Text:BLIGH WATER #12 BOTTOM



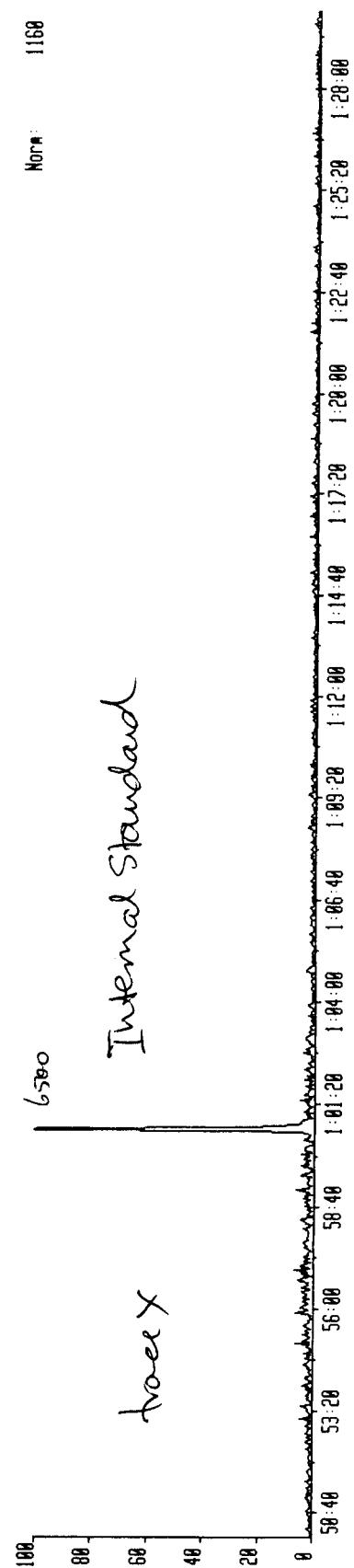
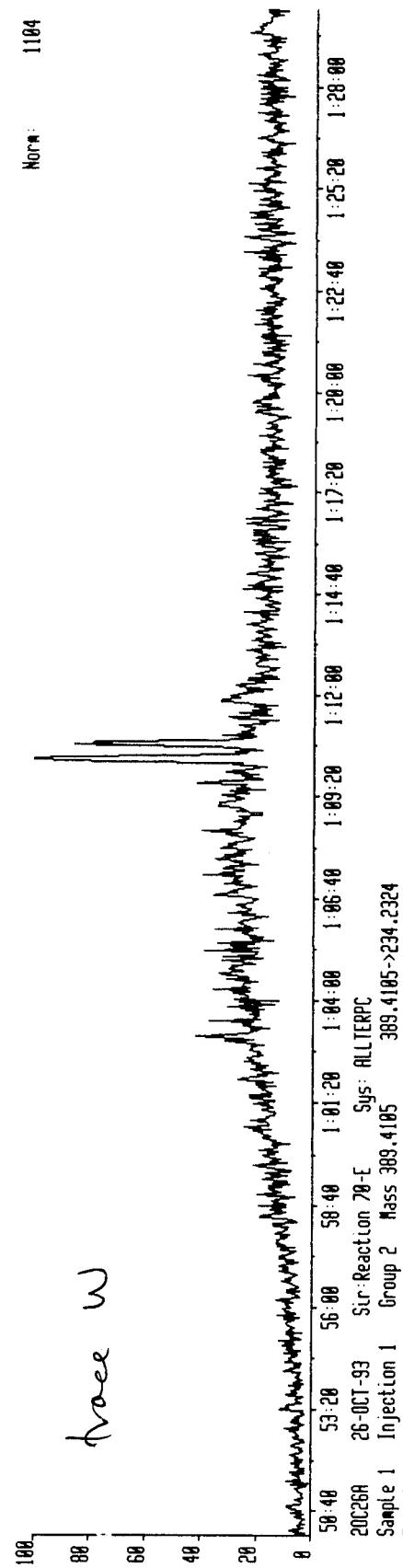
20C28A 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 1 Injection 1 Group 2 Mass 386,3910 386,3910->217,1968
Text:BLIGH WATER #12 BOTTOM



20C26H 26-OCT-93 Sir:Reaction 70-E Sys: ALLTERPC
Sample 1 Injection 1 Group 2 Mass 414.4230 414.4230->231.2120
Text:BLIGH WATER #12 BOTTOM

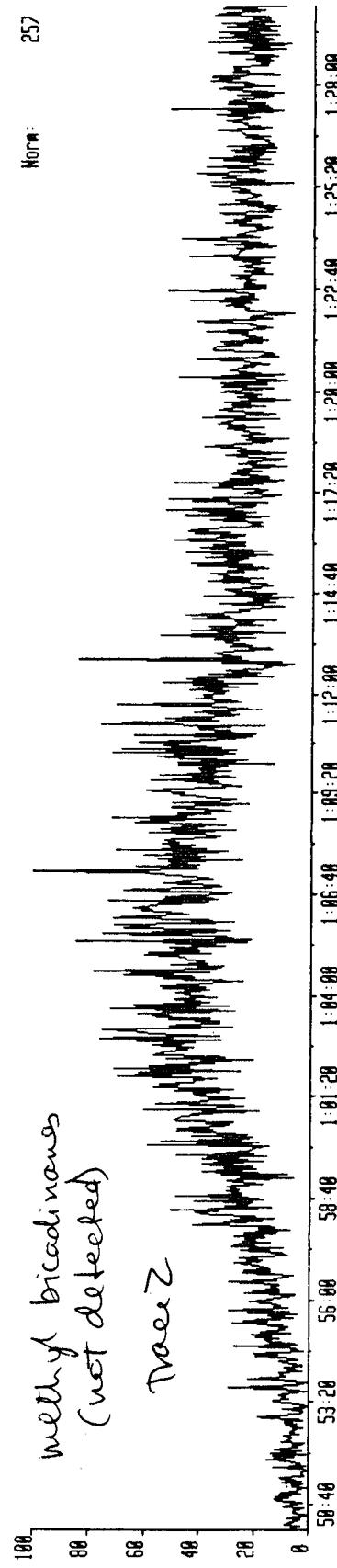
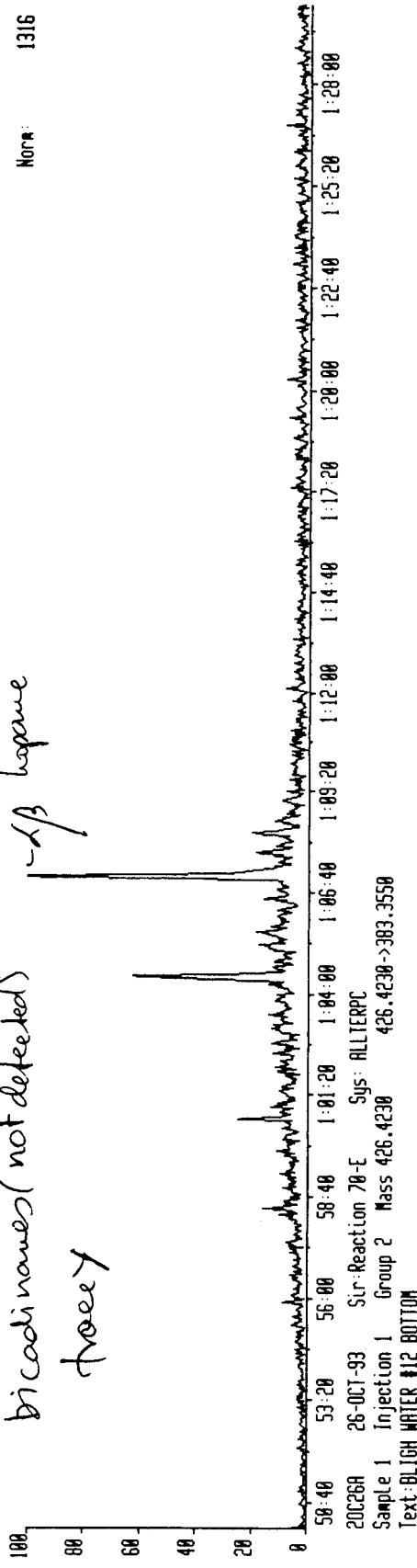


20C26H 26-OCT-93 Str:Reaction 70-E Sys: ALLTEPC
Sample 1 Injection 1 Group 2 Mass 386.3910 386.3910->231.2128
Text:BLIGH WATER #12 BOTTOM



2026B 26-OCT-93 Sir: Reaction A-E Sys: ALLIERPC
Sample 1 Injection 1 Group 2 Mass: 412.4068 412.4060->369.3480
Text:BLIGH WATER #12 BOTTOM

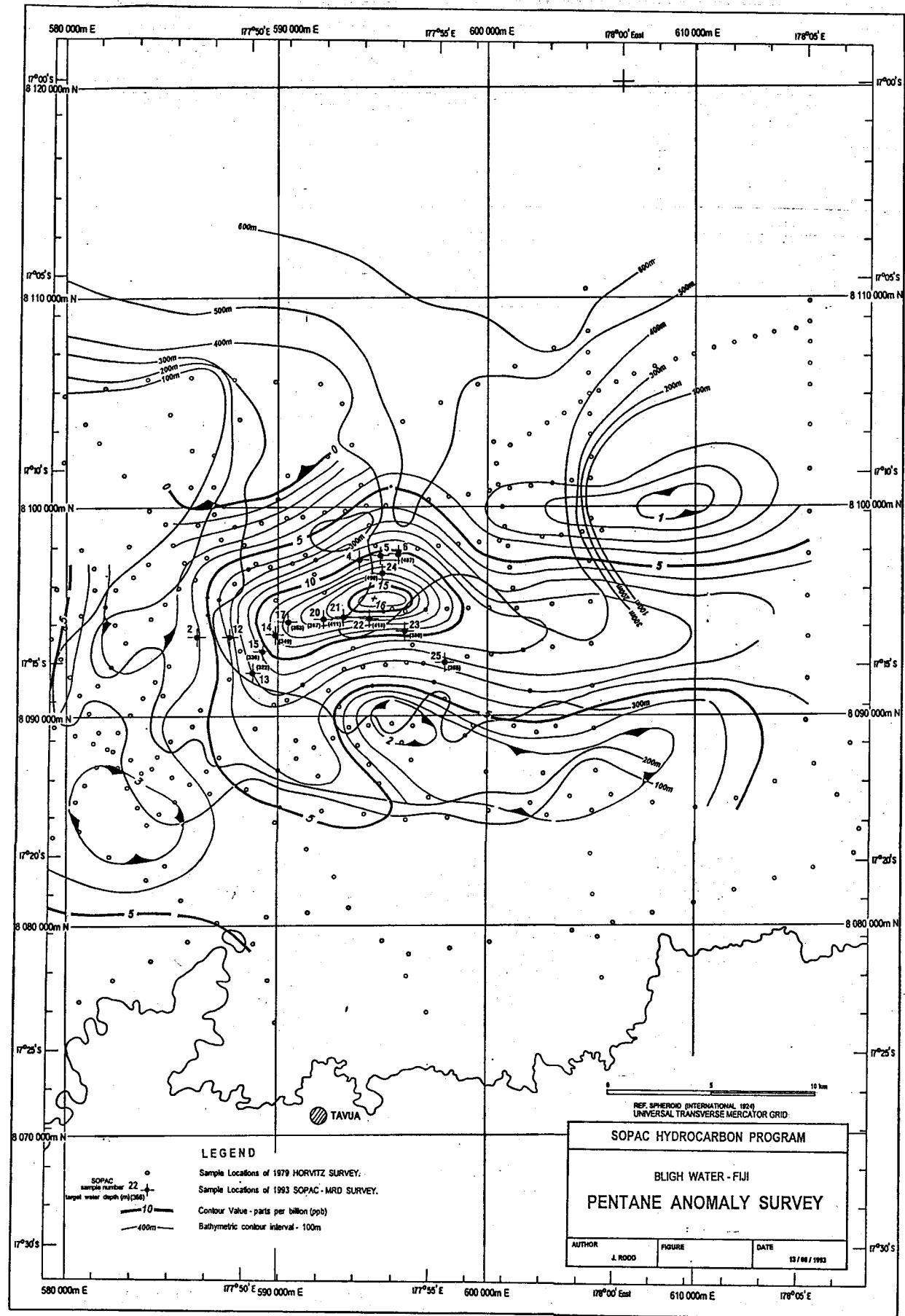
bicadinane (not detected) $\alpha\beta$ hexane
Trace 1



APPENDIX - Summary of Sampling

Sample	Date	Time	Easting (UTM)		Northing (UTM)		Depth (m)		Core Length (m)		Comments
			Target	Actual	Target	Actual	Target	Actual	Target	Actual	
1			585630		8093078		277		1.83		
2	6/9/93	1234	586245	586175	8093864	8093794	283	-	1.83	1.63	olive sandy mud
3			586849		8094705		280		1.83		
4	6/9/93	0857	593776	593868	8097566	8097509	378	-	1.83	1.75	olive fine mud
5	6/9/93	0918	594801	594889	8097759	8097727	439	-	1.22	0.88	
6	6/9/93	0947	595682	595701	8097855	8097825	475	487	0.91	2.15	olive fine mud
7			582868		8092935		219		1.83		
8			583530		8093658		213		1.83		
9			583458		8092550		164		1.83		
10			584067		8091612		256		1.83		
11			587249		8094758		310		1.83		
12	6/9/93	1257	587851	587842	8093853	8093808	315	-	1.83	1.62	olive sandy mud
13	5/9/93	1606	588881	588885	8092153	8092065	320	322	1.83	1.36	olive sandy mud
14	5/9/93	1417	589922	589944	8093879	8093989	329	349	1.83	1.91	olive fine sandy mud
15	5/9/93	1456	589354	589345	8093137	8093110	317	336	1.83	1.86	v. fine sandy mud, top coarser than bottom
16			589543		8096247		335		1.83		
17	5/9/93	1248	590490	590496	8094506	8094535	366	353	1.83	2.03	
18	6/9/93	1122	591549	591519	8092864	8092875	355	-	1.83	2.0	olive sandy mud
19			592053		8091944		330		1.83		
20	5/9/93	1155	592182	592164	8094701	8094683	366	387	1.83	0.90	
21(A)	5/9/93	1024	593190	593278	8094781	8094751	366	411	1.83	0.75	
21(B)	5/9/93	1104	593190	593147	8094781	8094732	366	411	1.83	0.83	
21(C)	6/9/93	1052	593190	593182	8094781	8094640	366	-	1.83	1.93	olive sandy mud
22	5/9/93	0939	594222	594303	8094818	8094678	402	418	1.83	1.71	
23	5/9/93	0840	595953	595972	8094068	8094051	399	380	1.83	1.52	
24	6/9/93	0822	594848	594944	8096899	8096832	470	490	1.83	1.96	very fine olive muddy sand
25	2/9/93	0927	598281	597917	8092734	8092544	350	355	1.83	2.20	olive fine sand with shell frags
Ltka	7/9/93	0945						10			black sand

Coordinates are UTM Zone 60 with WGS-72 datum.



Map of survey area showing location of core samples obtained (coordinates in Appendix A) and the contoured 'pentane anomaly' identified in the 1979 Horvitz survey.