CSIRO
Marine Laboratories

REPORT 90–HC2

AROMATIC HYDROCARBONS IN SOME TASMANIAN BITUMENS

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AUSTRALIA

SAMPLES

This report documents the aromatic hydrocarbon compositions of 13 bitumen samples obtained from the Queen Victoria Museum in Launceston and the Tasmanian Museum in Hobart by Mr P. Baillie of the Tasmanian State Government Department of Resources and Energy. These analyses have been undertaken at the request of Conga Oil Pty Ltd.

Capillary gas chromatograms of the total aromatic hydrocarbons for all samples are shown in the Appendix, together with and gas chromatography–mass spectrometry (GC–MS) data for selected samples. GC–MS data for the Flinders Island bitumen sample, which are generally representative of all the samples, are shown in the body of the text for illustrative purposes.

The samples were all hard, black, shiny asphaltic bitumens. They show a characteristic conchoidal fracture when broken. They contain no inorganic matter and dissolve completely in polar organic solvents such as chloroform. The samples were originally collected from coastal sites around the west coast of Tasmania and from King and Flinders Islands in Bass Strait. Many of these samples are mentioned in a report on petroleum exploration in Tasmania by the Government geologist W. H. Twelvetrees (1917). Most were collected from the turn of the century. A list of samples is provided in Table 1 together with their museum identification codes. Note that the Launceston Museum samples were recatalogued in 1956–57 and thus the "date code" does not refer to their date of collection.

TABLE 1. Hydrocarbon and asphaltene contents of Tasmanian bitumens.

Museum Code	Location	Aliphatics %	Aromatics %	Asphaltenes %	MPI	MPR	$VR_{(CALCa)}$	$VR_{(CALCb)}$
1956-33-357	Flinders Island	14.1	4.4	81.6	0.57	0.74	0.75	0.81
1956-33-359	Deep Creek, Port Davey	14.8	4.6	80.6	0.50	0.59	0.70	0.71
1956-33-360	Rocky Boat Harbour	12.3	6.6	81.1	0.57	0.77	0.74	0.83
1956-33-361	Albina (A)	14.9	5.6	79.5	0.51	0.63	0.70	0.74
1956-33-361	Albina (B)	14.5	4.1	81.4	0.55	0.73	0.73	0.81
1956-33-362	Mouth of Deep Creek, Port Davey	15.0	5.4	79.6	0.60	0.79	0.75	0.84
1956-33-364	Surprise River Beach	13.8	5.2	81.0	0.55	0.72	0.73	0.80
1957-33-944	Point Hibbs	14.1	6.4	79.4	0.60	0.83	0.76	0.86
1957-33-946	Beach at Port Davey	13.2	3.9	82.9	0.56	0.68	0.74	0.77
1957-33-948	King Island	14.8	6.2	79.0	0.58	0.79	0.75	0.84
1957-33-949	New River	13.2	6.7	80.0	0.51	0.58	0.71	0.71
X2283*	Marrawah	14.4	4.9	80.7	0.61	0.73	0.77	0.81
X3256*	Flinders Island	13.6	7.4	78.9	0.56	0.71	0.74	0.79

* Samples from Tasmanian Museum, Hobart. All other samples from Queen Victoria Museum, Launceston.

METHODS AND RESULTS

Total Hydrocarbon Content

A portion of the chloroform extract was analysed by Iatroscan thin–layer chromatography–flame ionisation detection (Volkman *et al.*, 1986) to determine the total hydrocarbon concentration in each bitumen. Typical TLC–FID chromatograms are shown in Volkman and O'Leary (1990). The samples were also re–analysed by TLC–FID using hexane as the developing solvent (Figure 1) to separate aliphatic from aromatic hydrocarbons: quantitative data are shown in Table 1. These values were also compared with the amounts (determined by TLC–FID) of the aliphatic and aromatic hydrocarbon fractions isolated by column chromatography. Aliphatic hydrocarbons represented 13.2–15.0 % of the total extracts, whereas aromatic hydrocarbons were less abundant and represented 3.9–6.7 % of the extracts. About 80 % of the extractable material consisted of polar resins and asphaltenes. Within the accuracy of the method (± 6 % of value quoted), most of the values obtained are not significantly different from one sample to another.

Saturated and aromatic hydrocarbons were isolated by applying 30 mg of extract to a column of 3 g of silicic acid (100–200 mesh) capped with 1 g of activated alumina (BDH). Aliphatics were eluted with hexane (20 mL), and a second fraction containing aromatic hydrocarbons was obtained by eluting with toluene:hexane (1:1; 20 mL). Resins and asphaltenes were eluted with chloroform (20 mL) and methanol (10 mL).

Aliphatic and aromatic hydrocarbon fractions were analysed by capillary gas chromatography on a 50 metre non–polar methyl silicone fused silica capillary column.

The aliphatic hydrocarbon distributions give an overall impression of a non–waxy, weathered heavy crude oil (Volkman and O'Leary, 1990). There is little to indicate that the samples have been water washed to any great extent and certainly they have been subjected to little biodegradation. It seems unlikely that they could have been floating in the sea for more than a few days (if at all), and that it is probable that they were derived from a source close to where they were collected.

Details of the aromatic hydrocarbons are provided below.

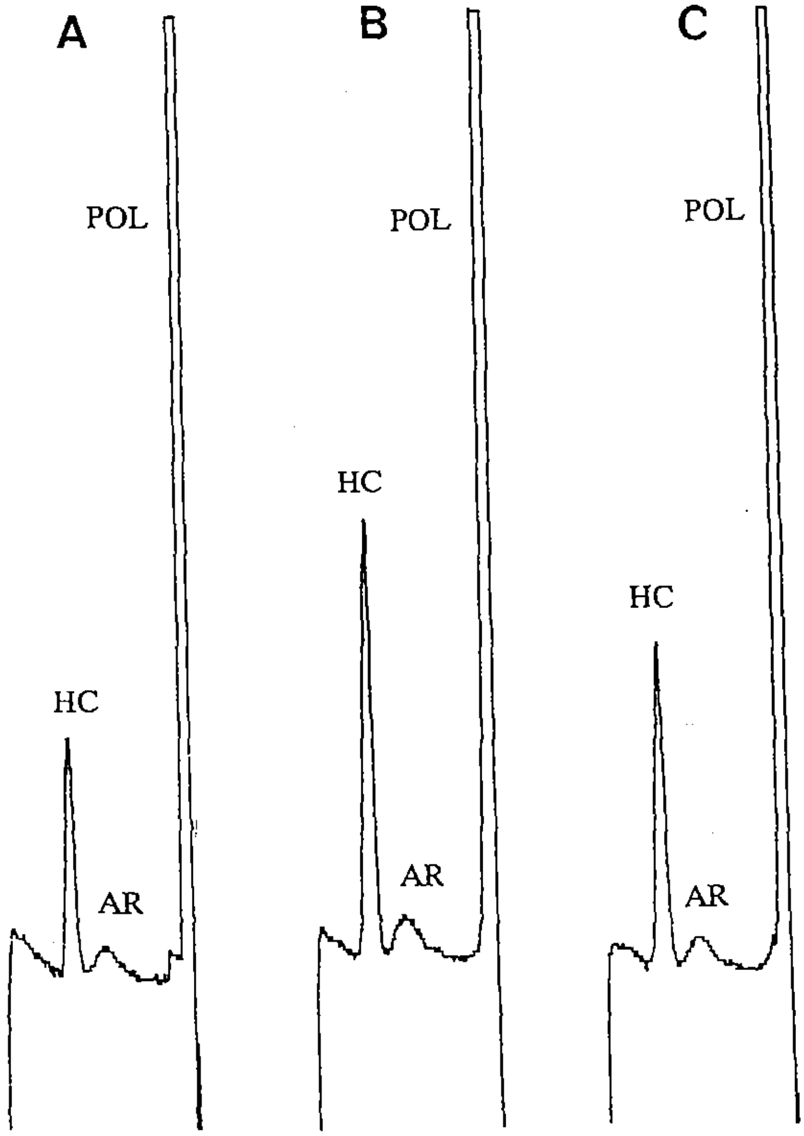


Figure 1. TLC–FID chromatograms showing composition of solvent–extractable material from bitumen samples (A) Flinders Island 33–357, (B) Marrawah X2283 and (C) King Island 33–948. Peak identifications: HC–aliphatic hydrocarbons; AR–aromatic hydrocarbons; POL–resins and asphaltenes. The developing solvent was hexane.

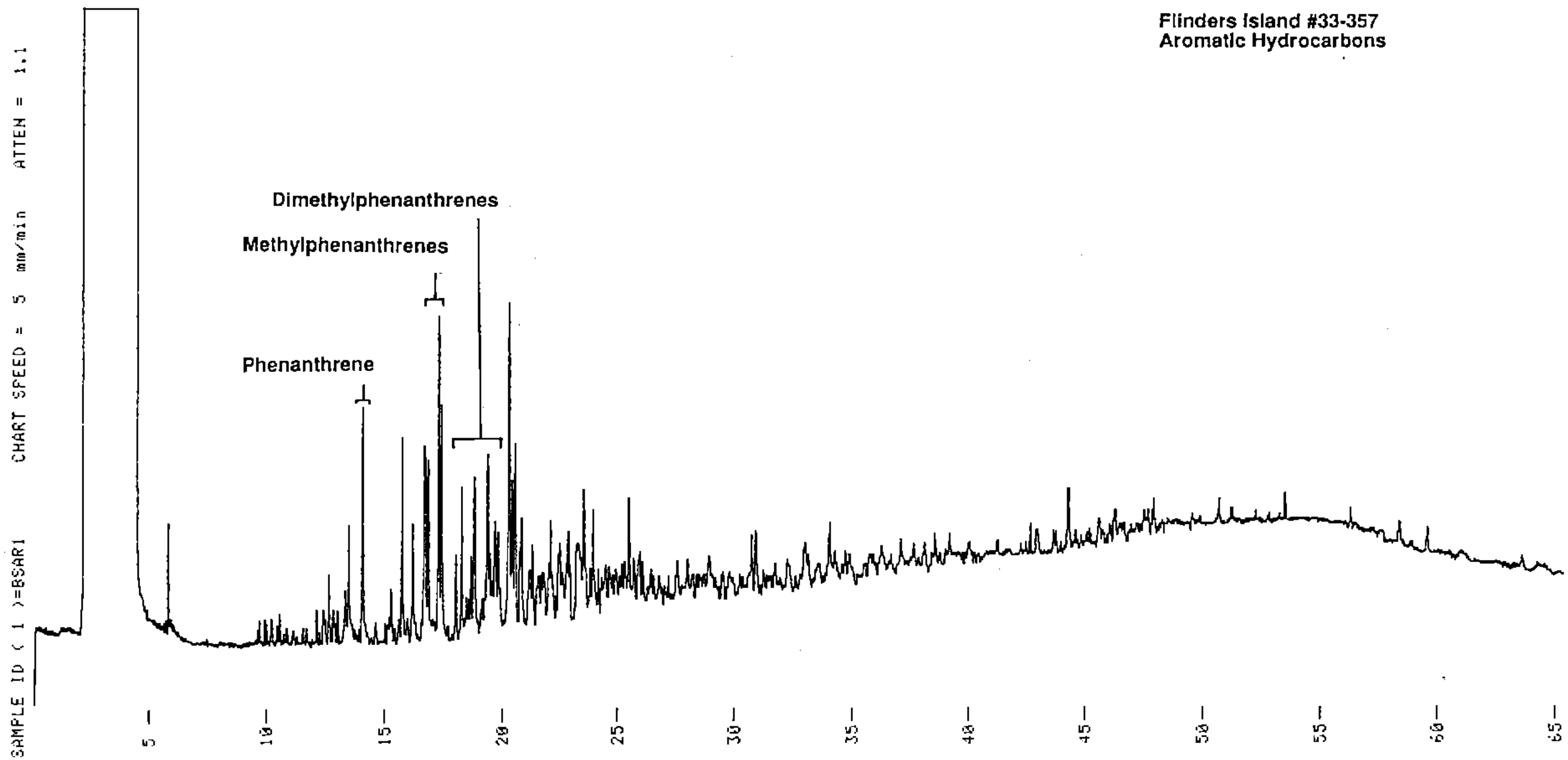


Figure 2. Capillary gas chromatogram showing the distribution of total aromatic hydrocarbons in the Flinders Island bitumen.

Aromatic Hydrocarbons

The bitumens have a distinctly "aromatic" smell, not unlike naphthalene. A gas chromatogram of the aromatic hydrocarbons in the Flinders Island bitumen is shown in Figure 2. The GC temperature program was 45 ^{o}C for 1 minute followed by a ramp of 30 ^{o}C/min. to 120 ^{o}C, then a ramp of 4 ^{o}C/min. to 320 ^{o}C. The oven was maintained isothermally at 320 ^{o}C for 15 minutes. Peak areas were measured using DAPA software. Note that some chromatograms shown in the Appendix contain a regular series of peaks after about 40 minutes due to some tailing of aliphatic hydrocarbons into the aromatic hydrocarbon fractions during column chromatography.

Most of the chromatograms show a broad hump representing an unresolved complex mixture of components. This type of chromatogram is typical of weathered or biodegraded bitumens. Had these samples been extensively water washed, we would not have expected to see compounds such as phenanthrene in such abundance, although clearly there has been substantial removal of alkylated benzenes, naphthalene and some alkylated naphthalenes which are more volatile and water-soluble (e.g. Volkman *et al.*, 1984). The hump is less obvious in the chromatogram of the Flinders Island sample (Figure 2) than in many of the tars from the west coast (Appendix), and the relative amount of compounds more volatile than phenanthrene is also less. This appears to be due to differences between the separations of aliphatic and aromatic fractions achieved during column chromatography, rather than due to major differences between samples.

Phenanthrene and alkylated phenanthrenes

Superimposed on the hump, are a number of discrete peaks due mainly to alkylated (C_1–C_4) phenanthrenes. Representative mass fragmentograms for m/z 178 (phenanthrene) and 192 (methylphenanthrenes) are shown in Figure 3. Mass fragmentograms for selected samples are given in the Appendix. The methylphenanthrene distributions in each sample are remarkably similar, and quite unusual compared with many other geological materials that we have studied. In mature oils, the 2– and 3–methylphenanthrene isomers usually predominate over the 1– and 9–methylphenanthrene isomers (Radke and Welte, 1983), but the reverse is found in the bitumens.

Methyl phenanthrene parameters (MPI; Radke and Welte 1983) were calculated directly from peak areas in the capillary gas chromatograms (Table 1); values are given in Table 1. The equivalent vitrinite reflectances calculated from the MPI values according to the equation of Radke and Welte (1983) covers the range 0.55–0.61 (i.e. early oil window), which is in good agreement with the biomarker maturity parameters. If the alternative equation proposed by Boreham *et al.* (1988) is used, the equivalent vitrinite reflectances are calculated to be about 0.5. Note that this latter equation refers strictly to coals and carbonaceous shales, and so may not be appropriate here. The abundance of the 1-methylphenanthrene is quite low which argues against a source containing coniferous higher plant organic matter (Alexander *et al.*, 1988).

Equations used to calculate methylphenanthrene ratios and equivalent vitrinite reflectance values:

$$MPI = \frac{1.5\,(2\text{-}MP + 3\text{-}MP)}{P + 1\text{-}MP + 9\text{-}MP}$$

$$MPR = \frac{2\text{-}MP}{1\text{-}MP}$$

VR_{CALCa} = 0.6 MPI + 0.4 (Radke and Welte, 1983)

VR_{CALCb} = 0.99 $\log_{10}$ MPR + 0.94 (Radke *et al.*, 1984)

VR_{CALCc} = 0.7 MPI + 0.22 (Boreham *et al.*, 1988).

Methylphenanthrene distributions had earlier been determined for hydrocarbons isolated from several Tasmanian limestones (AMDEL, 1987). Mass fragmentograms are shown in Figure 4. None of these distributions match very well with the bitumen profiles. The Ordovician limestones from Surprise Bay and Ida Bay appear to much more mature, but those from the Kamburg and Chicken Island limestones are only slightly more mature. Further information about the aromatic hydrocarbons in these samples is not available.

Values for another maturity ratio MPR which is based on the abundances of 1- and 2-methylphenanthrenes (Radke *et al.*, 1984) are also shown in Table 1. These show slightly greater variation than the MPI ratios and suggest a slightly higher equivalent vitrinite reflectance of 0.71-0.86 (Table 1). As with the MPI values, these interpretations should be treated as a guide only since they are not calibrated against actual sediments of known maturity from this region.

The distributions of dimethylphenanthrenes and trimethylphenanthrenes determined from mass fragmentograms for m/z 206 and 220 respectively (Figure 5 and Appendix) were also remarkably similar for each sample. Individual isomers were not determined.

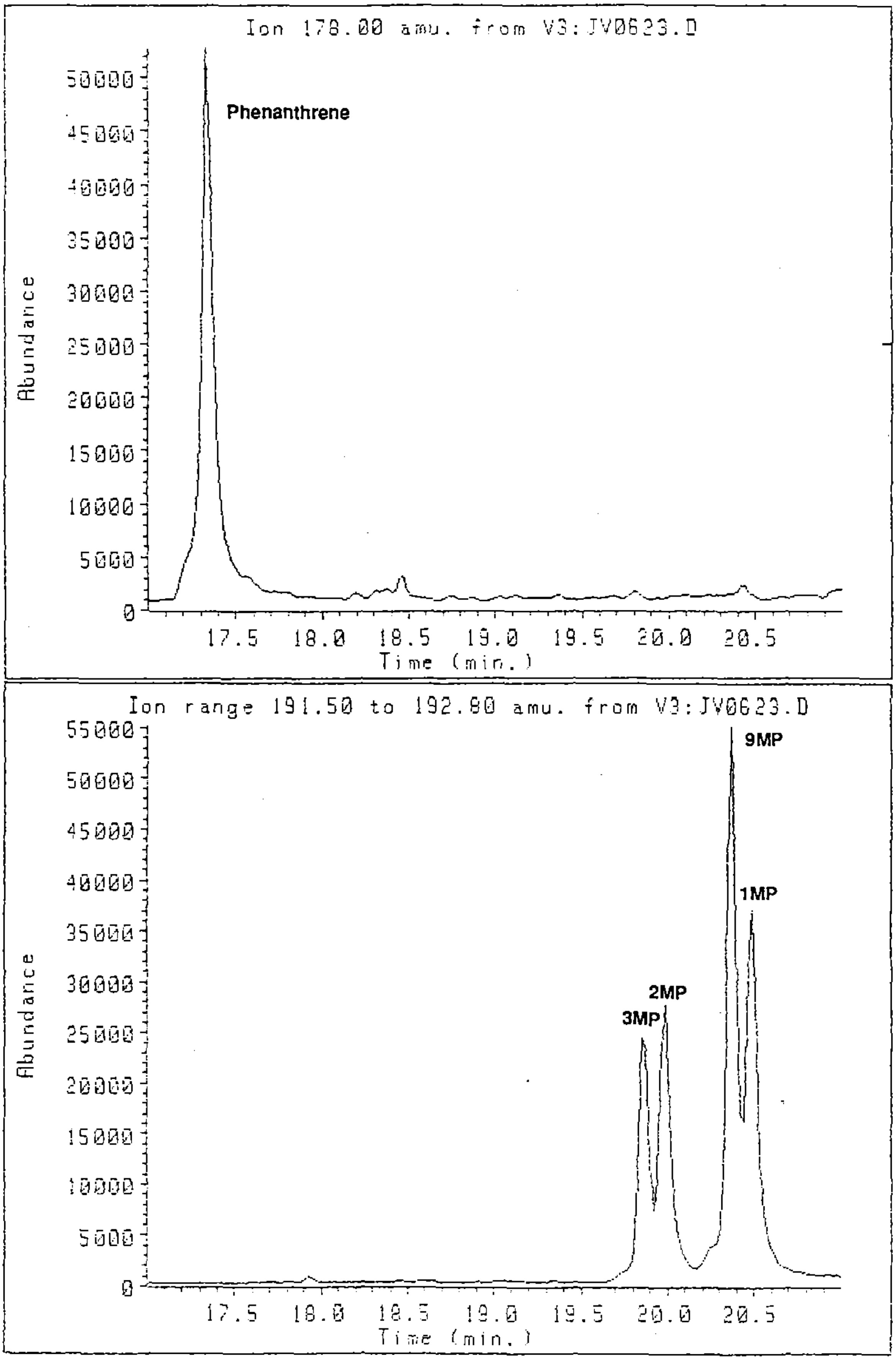


Figure 3. Mass fragmentograms for m/z 178 and 192 showing the presence of phenanthrene and distribution of methylphenanthrenes respectively in the Flinders Island bitumen.

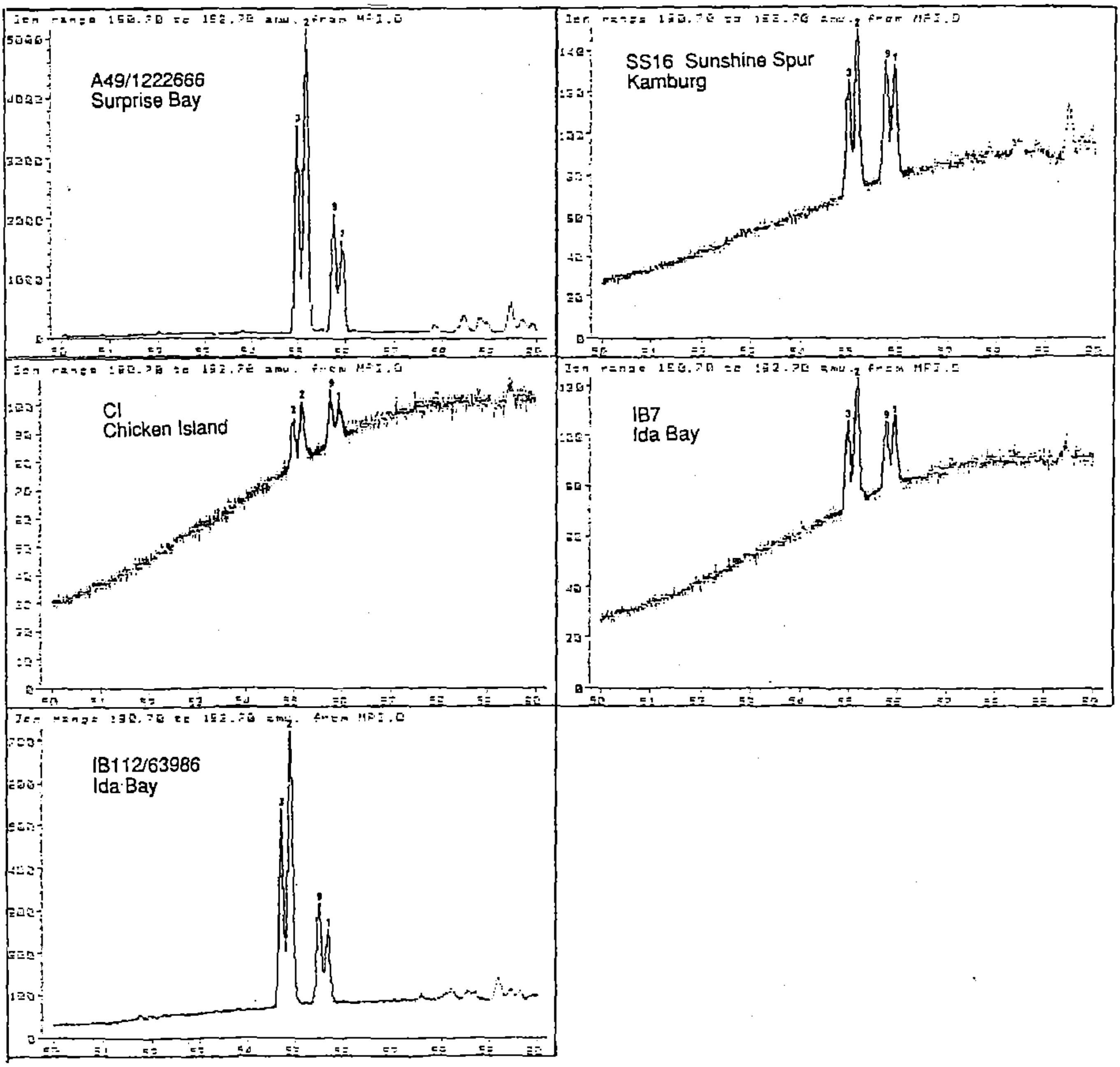
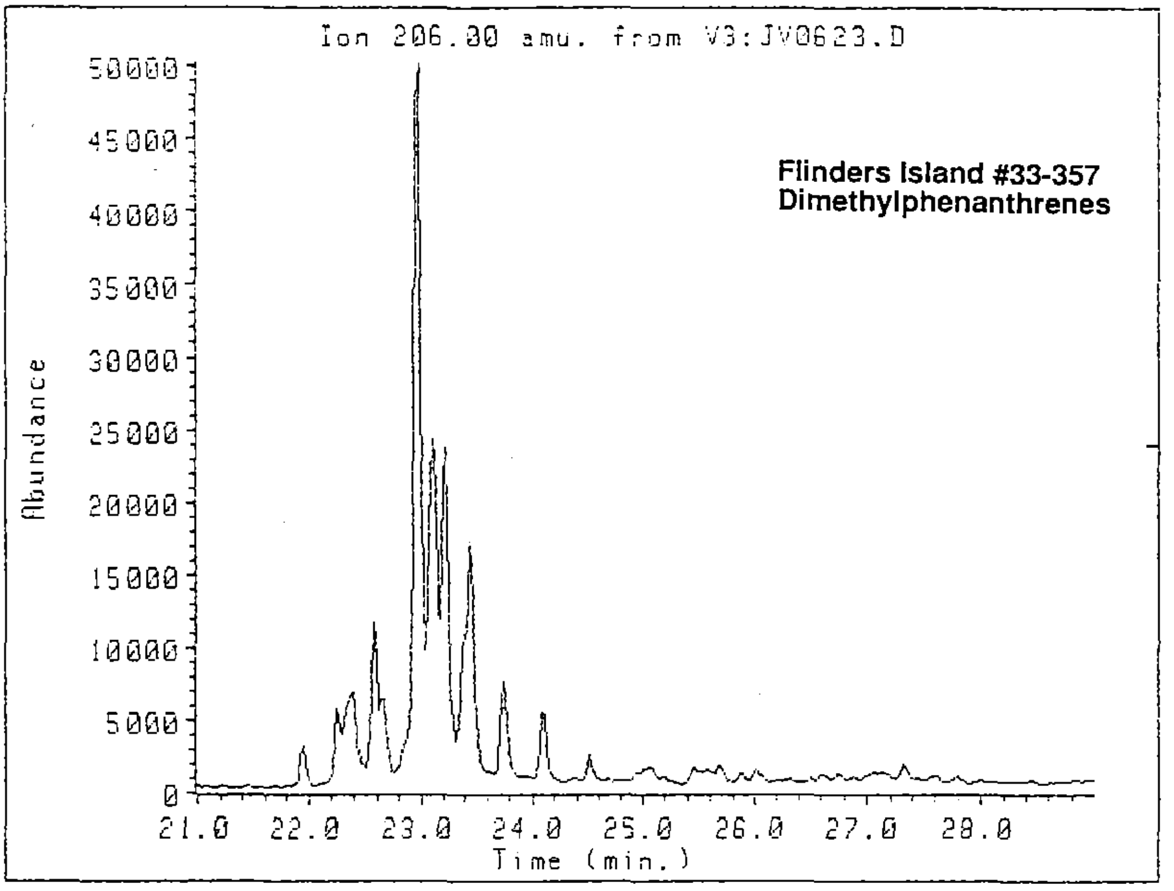


Figure 4. Mass fragmentograms for m/z 191 + 192 showing the distribution of methylphenanthrenes in some Tasmanian limestones (AMDEL, 1987).



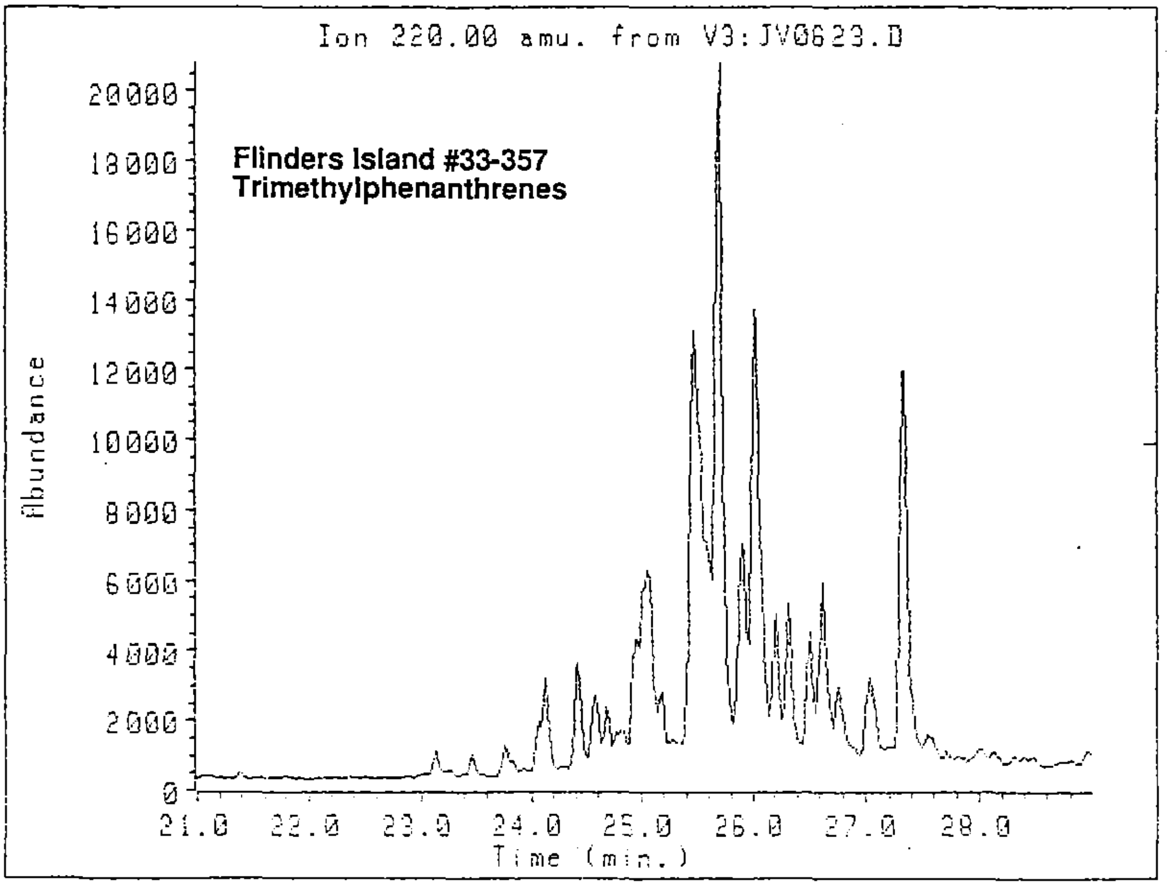


Figure 5. Mass fragmentograms for m/z 206 and 220 showing the distribution of di- and trimethylphenanthrenes in the Flinders Island sample.

Aromatic Steroid Biomarkers

The aromatic hydrocarbon fractions were analysed by gas chromatography–mass spectrometry in selected ion monitoring mode (SIM) to determine the distribution of mono– and tri–aromatic hydrocarbons. These compounds mainly provide information about the maturity of a crude oil, although some information on source affinities can sometimes be obtained from the distribution of C_{26}–C_{29} components.

At least 7 types of aromatic steroid hydrocarbons are commonly found in oils as summarised in Figure 6 (Mackenzie *et al.*, 1981). Monoaromatic components may have one, two or three methyl groups distributed on the A, B and C rings. These give characteristic cleavage ions at m/z 239, 253 and 267 respectively. A GC–MS search for monoaromatic steroids was made using these characteristic ions, but only trace amounts could be detected in the samples (Figure 7). Such low abundances are quite unusual, but nonetheless the distributions observed are typical of moderately mature crude oils.

Triaromatic steroid hydrocarbons, however, were readily detected. Ion chromatograms for ions m/z 231 and 245 (triaromatic steroids with one and two methyl groups in the ABC ring system respectively) in the Flinders Island sample are shown in Figure 8. Minor amounts of compounds having an m/z 217 base peak were also detected (data not shown). Mass fragmentograms for the other samples (Appendix) are very similar and confirm that all the bitumens were generated from the same or similar source rock at a very similar level of thermal maturity.

The m/z 231 mass fragmentogram is typical of sediments within the early oil window. With increasing maturation, one generally sees a decrease of the C_{26}–C_{28} components relative to the C_{20} and C_{21} components, such that at high maturities the latter predominate. This is not seen in the distributions in the bitumens, providing further evidence that the bitumens were derived from oils generated at low to moderate maturity. For comparison, similar distributions are found in sediments from the Paris Basin at depths greater than 2000 m.

The m/z 245 mass fragmentogram is also typical of those found in moderately mature petroleums. The distribution of C_{27}–C_{30} compounds is quite complex, and further study is needed to assign individual components. In common with other studies, few of the compounds gave molecular ions so it was not possible to assign the carbon number of most of the major components. However, the distribution should be very useful for comparing with those in potential source rocks.

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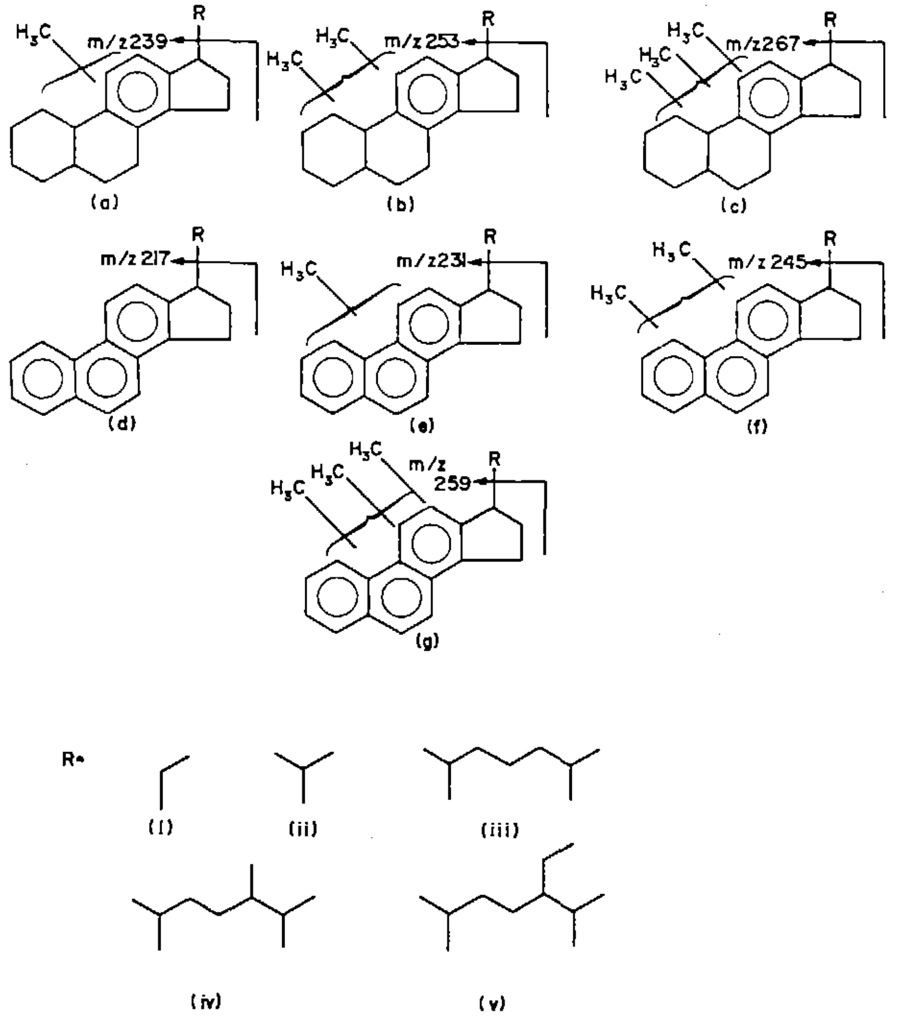


Figure 6. Structures of aromatic steroid hydrocarbons commonly found in crude oils. The major fragment ions are show, plus structures of the common side–chains (R).

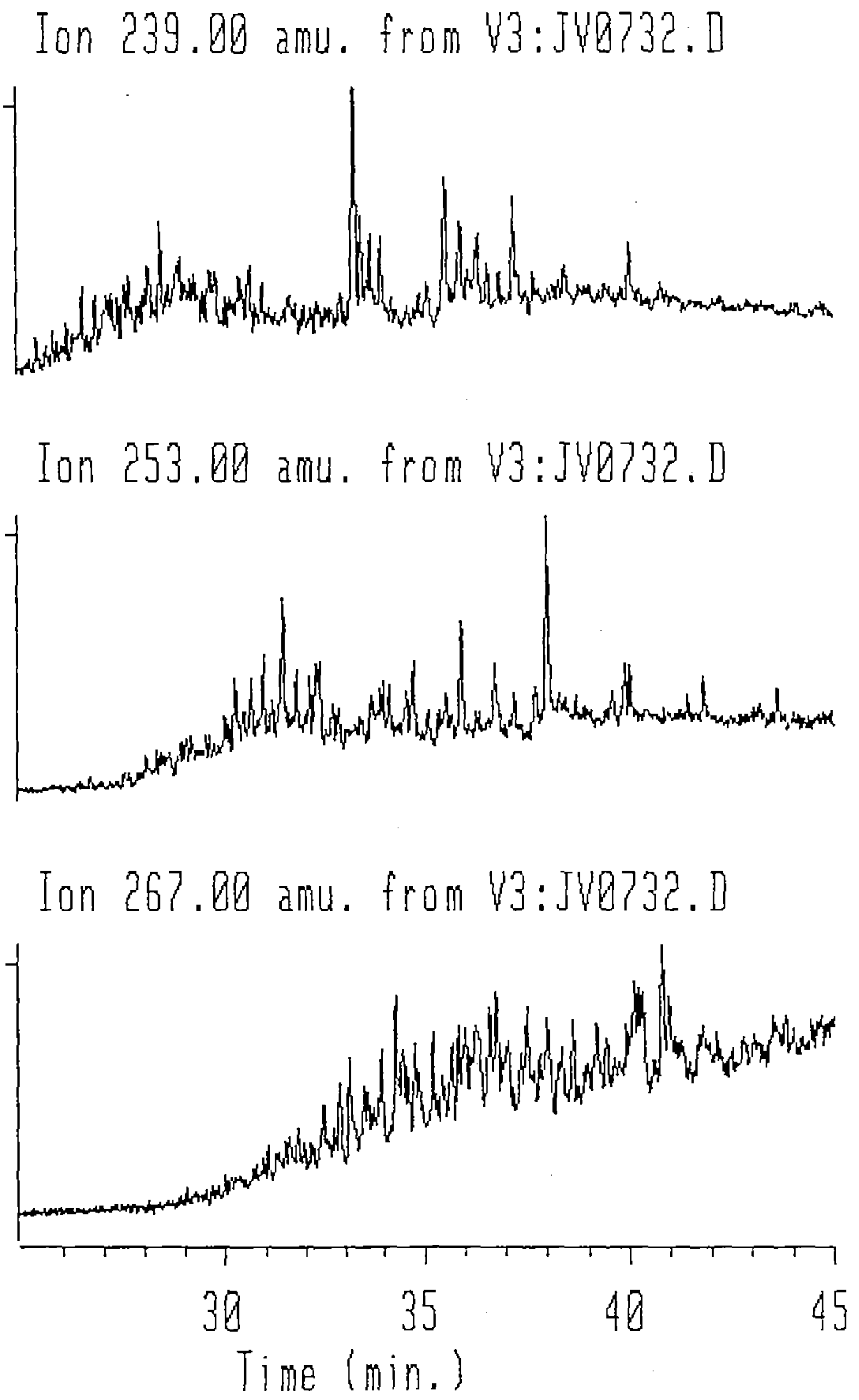
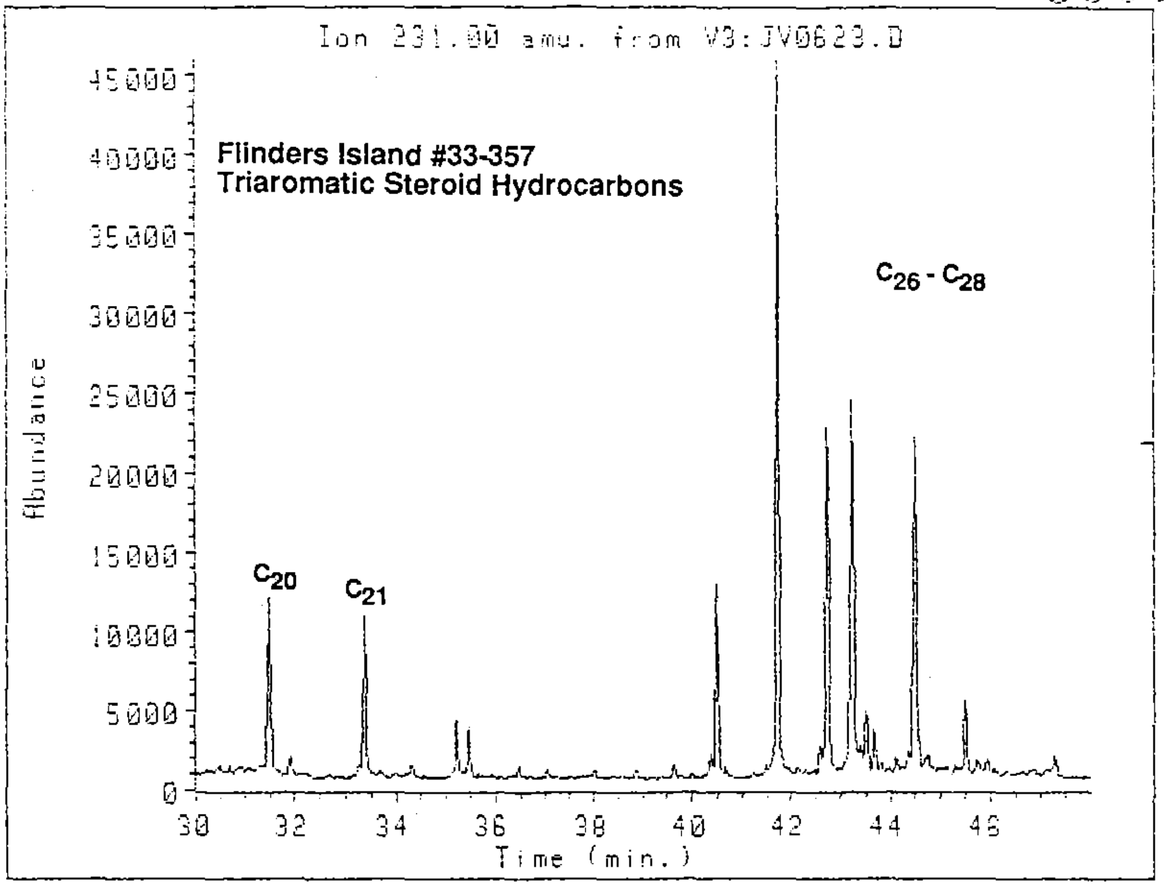


Figure 7. Mass fragmentograms for m/z 239, 253 and 267 showing the distribution of monoaromatic steroidal hydrocarbons (with one, two and three methyl groups in the ABC ring system respectively) in Flinders Island bitumen.



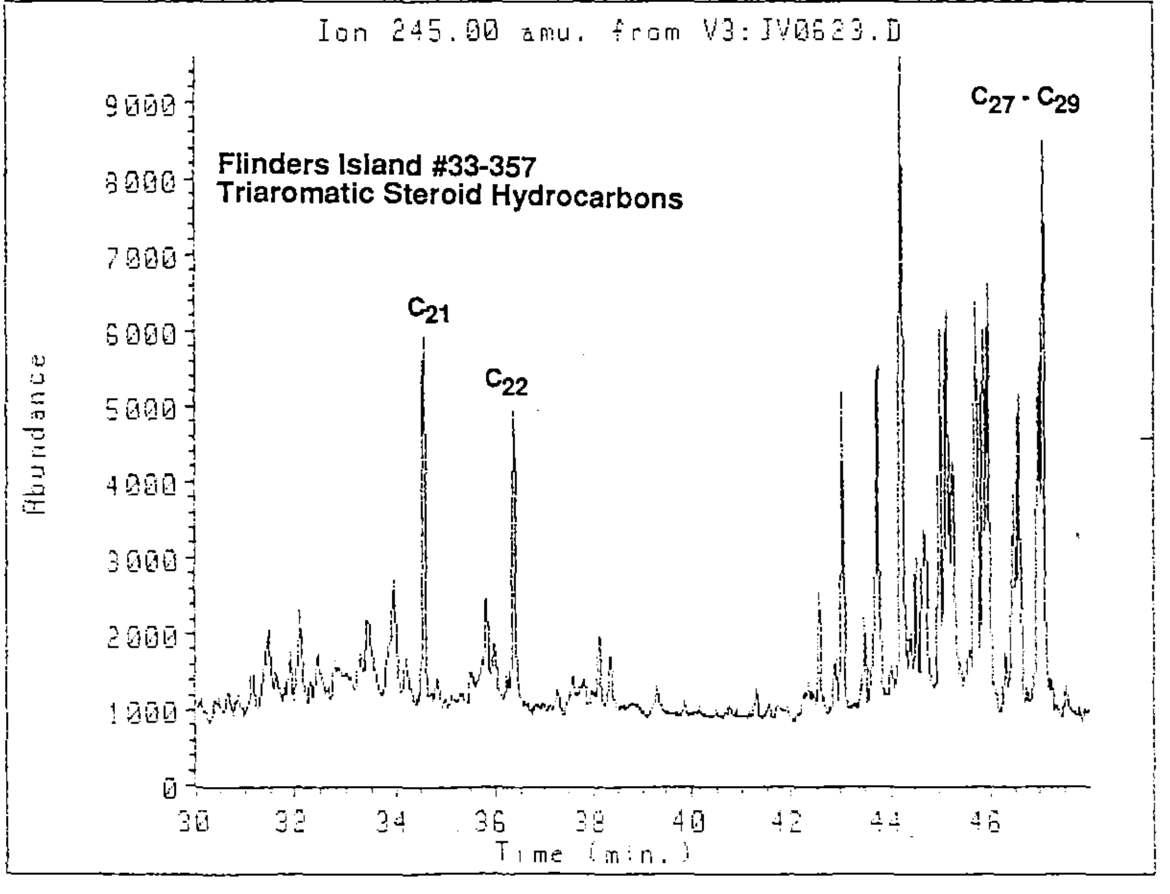


Figure 8. Mass fragmentograms for m/z 231 and 245 showing the distribution of triaromatic steroidal hydrocarbons in Flinders Island bitumen. Compounds giving a major m/z 231 ion have a single methyl group in the ABC ring system; those giving rise to a major ion at m/z 245 have two methyl groups in the ABC ring system.

CONCLUSIONS

The bitumens contain appreciable amounts of hydrocarbons with a ratio of aliphatic to aromatic hydrocarbons of about 3:1. The distributions of aromatic hydrocarbons are typical of those found in moderately mature crude oils, but the isomer distributions are distinctive and should prove valuable for correlating with presumed source rocks. The lack of shorter chain n-alkanes and low molecular weight aromatic hydrocarbons in all samples is typical of weathered bitumens, but there is no evidence that the tars have been significantly biodegraded.

These new data support our previous conclusions: (i) The bitumens were probably derived from a heavy non-waxy crude oil having a high content of asphaltenes. (ii) The aliphatic and aromatic biomarker distributions are all remarkably similar and indicate a common source and generation at very similar levels of thermal maturity. (iii) Some aliphatic biomarker parameters are similar to those found in hydrocarbons from Ordovician limestones from Ida Bay and Queenstown except for the absence of methyl hopanes and 29-norhopanes. The remarkable similarity between the sterane distributions implies that the hydrocarbons in both are probably derived from the same type of (algal/bacterial) organic matter.

It is apparent that the bitumens are unrelated to the tasmanite oil shales. They appear to have been generated from an organic-rich shale. Sterane parameters rule out higher plants as the major contributors of organic matter in the source rocks. The bitumens also appear to be unrelated to oils presently recovered from the Bass and Gippsland Basins.

The presence of abundant diasteranes implies a depositional environment in which the sediments contain a high content of silt or clay. The absence of methyl hopanes argues against a shallow carbonate depositional environment represented by the limestones previously analysed from Ida Bay and Queenstown. Aliphatic and aromatic biomarker parameters suggest that the oils were generated at an equivalent vitrinite reflectance of about 0.6–0.7, i.e. within the early oil window. A detailed geochemical study of possible source rocks is now required to determine the source of the bitumens.

REFERENCES

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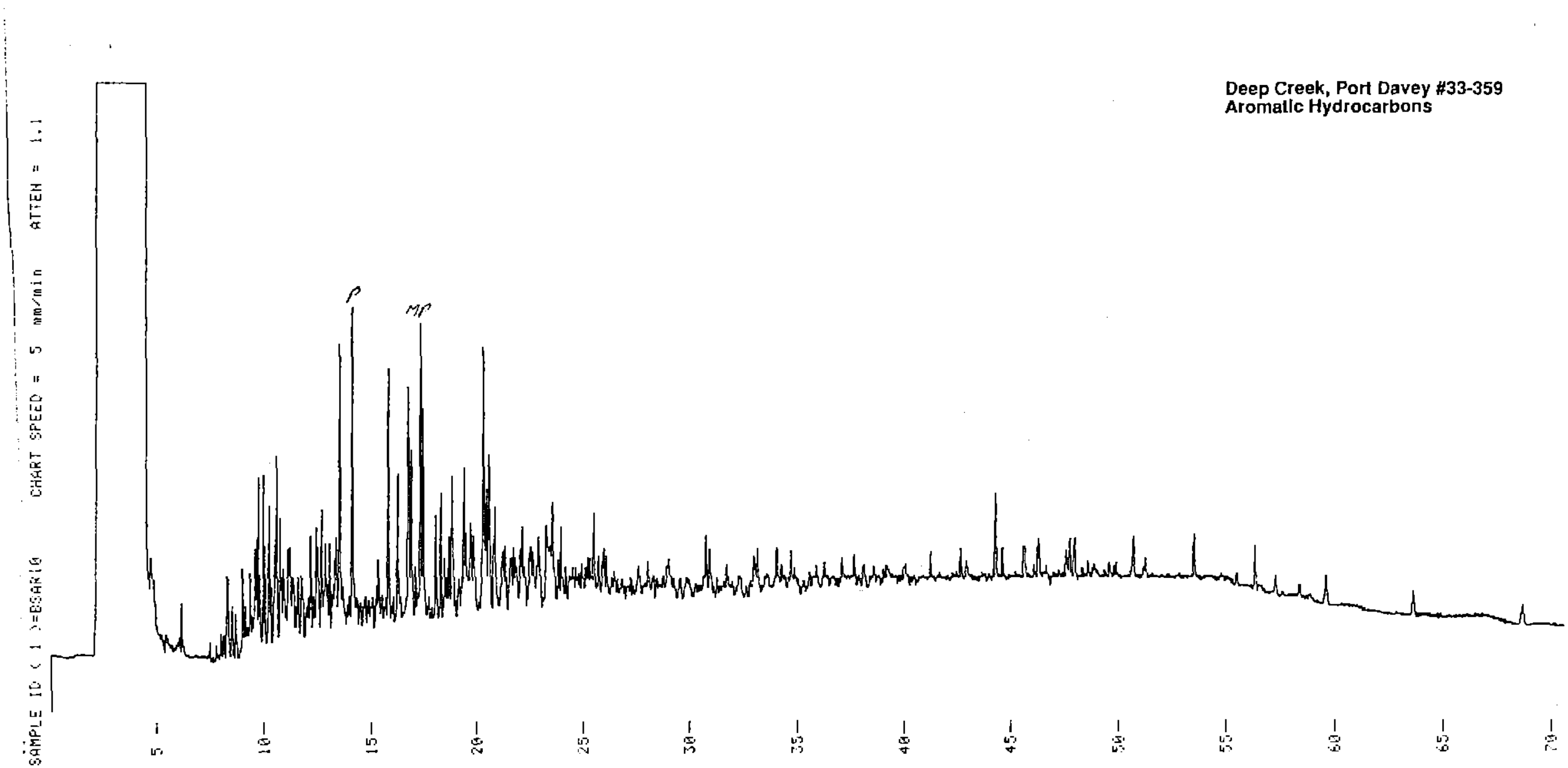
Volkman, J. K., Alexander, R., Kagi, R., Rowland, S. J. and Sheppard, P. N. (1984) Biodegradation of aromatic hydrocarbons in crude oils from the Barrow sub-basin of Western Australia. *Org. Geochem.* **6**, 619–632.

APPENDIX

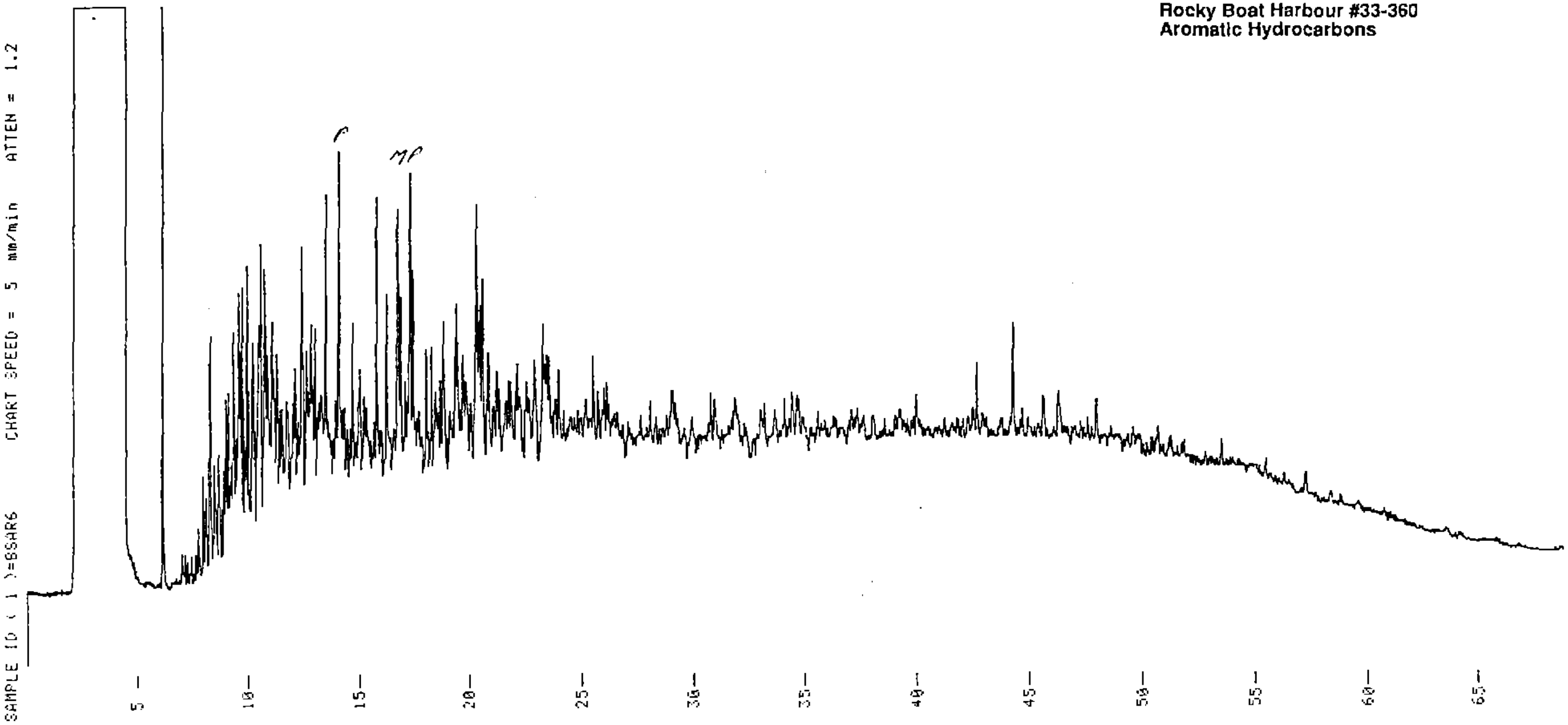
Capillary gas chromatograms of total aromatic hydrocarbons

in some Tasmanian bitumens.

Deep Creek, Port Davey #33-359
Aromatic Hydrocarbons



Rocky Boat Harbour #33-360
Aromatic Hydrocarbons



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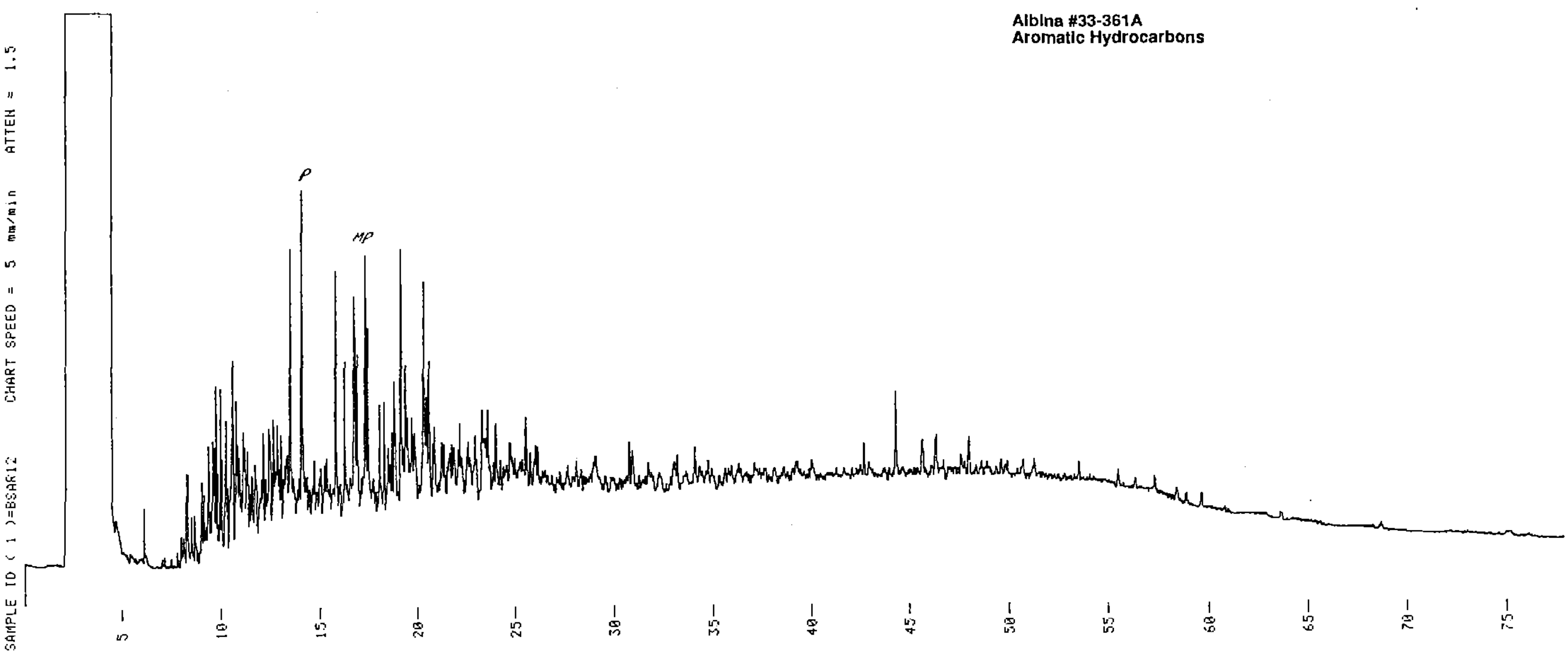
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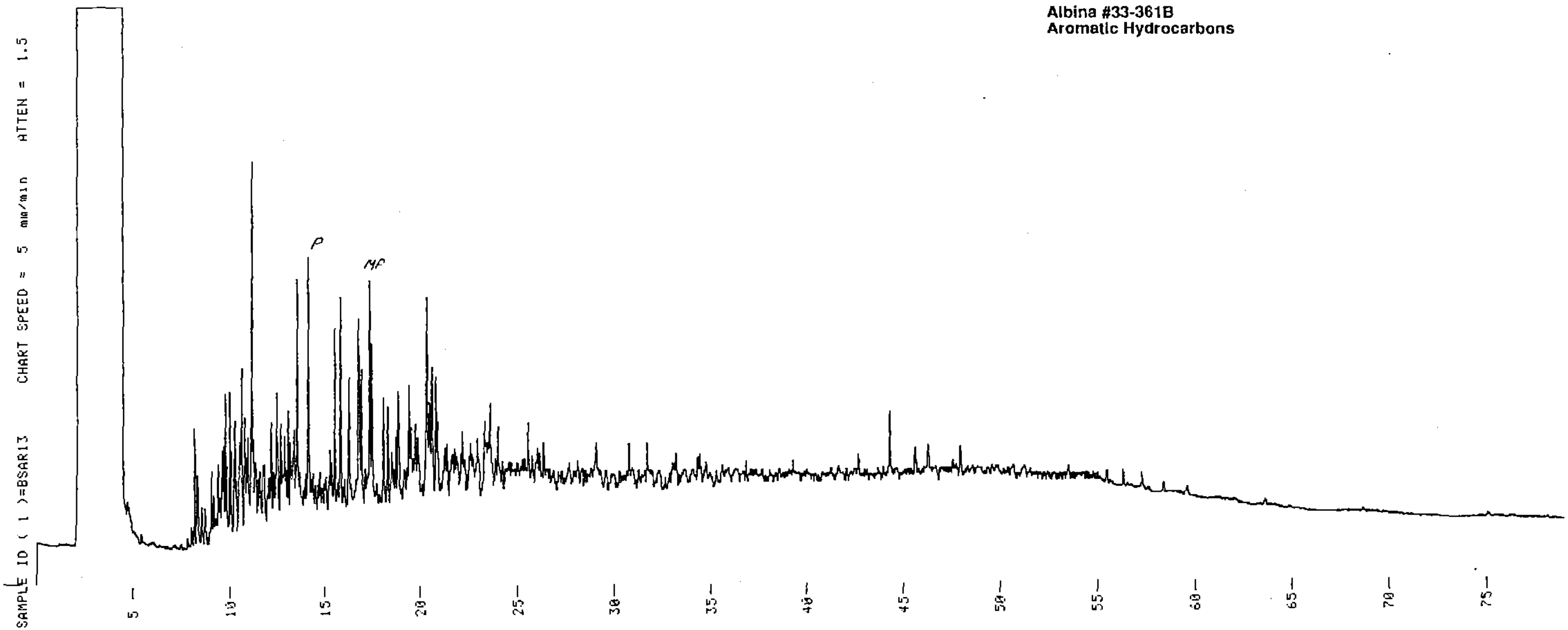
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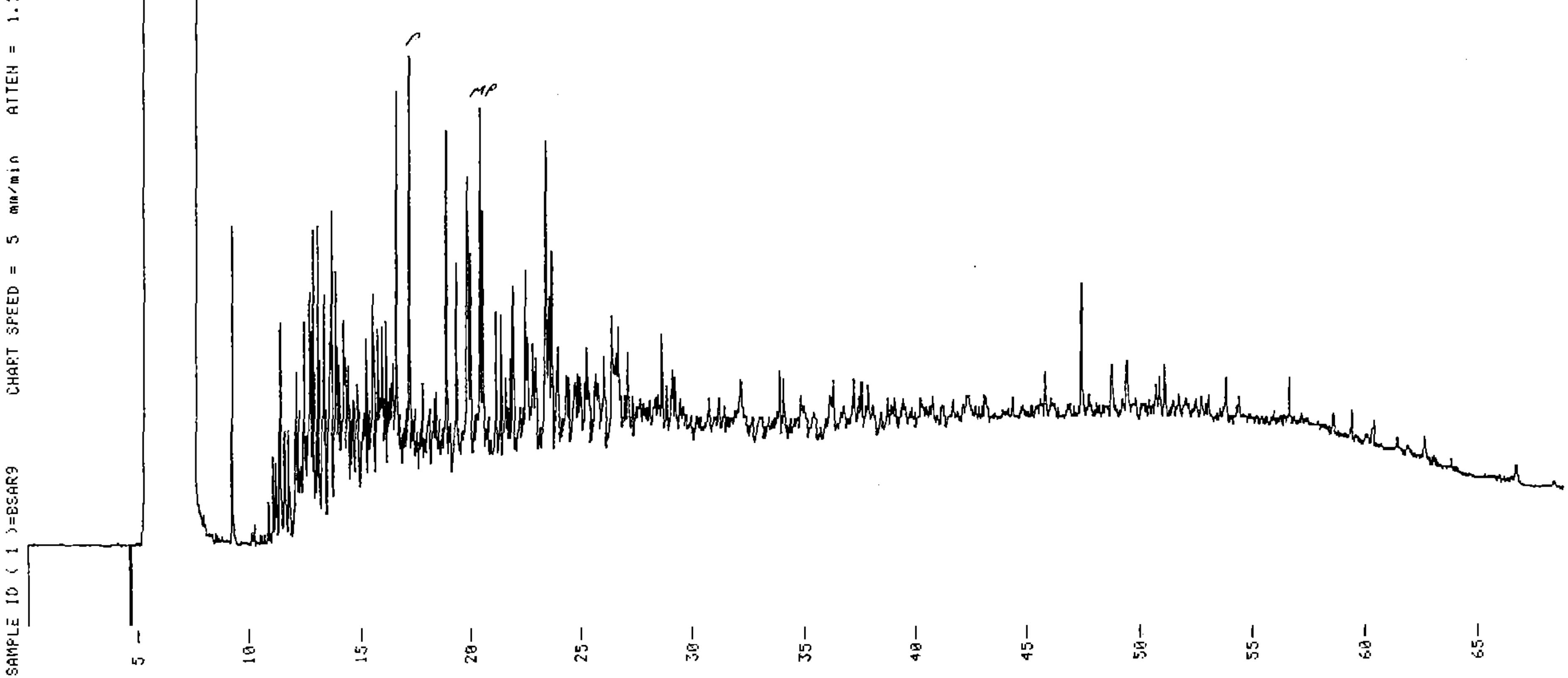
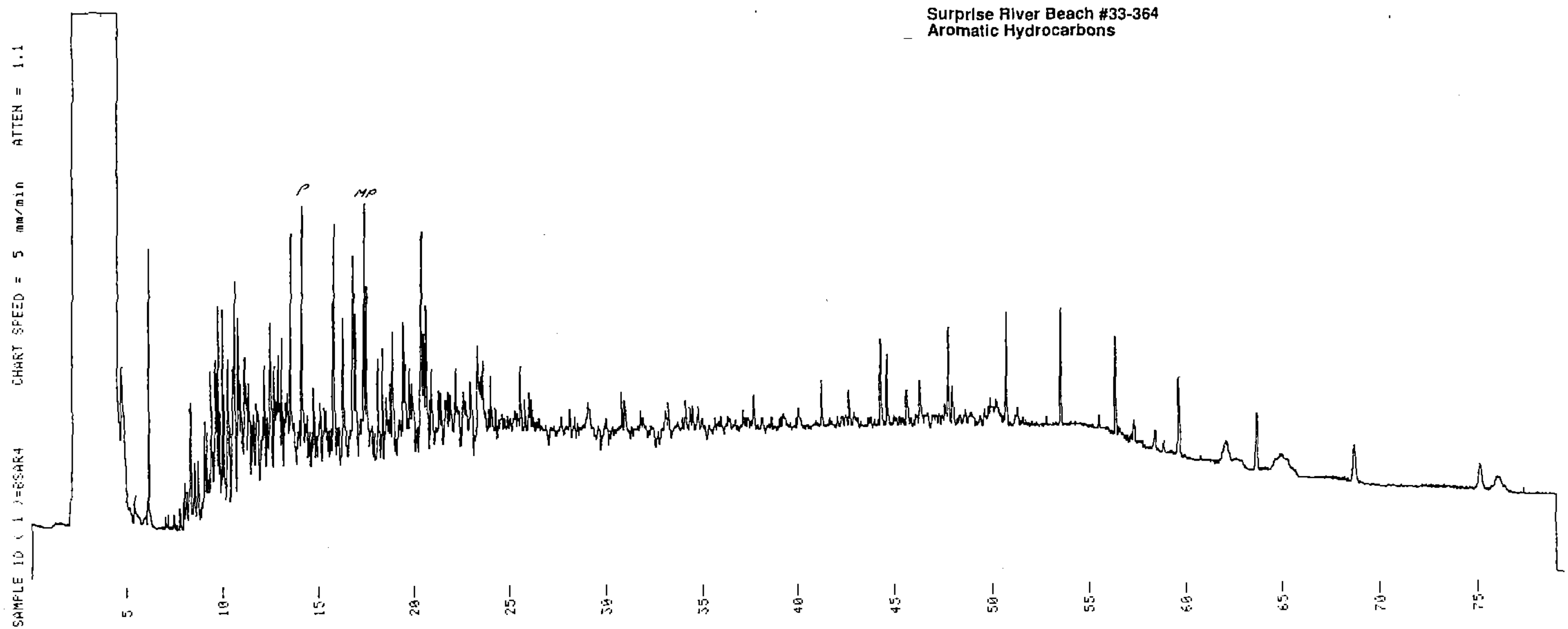
3860022

Mouth of Deep Creek,
Port Davey #33-362
Aromatic Hydrocarbons



386023

Surprise River Beach #33-364
Aromatic Hydrocarbons



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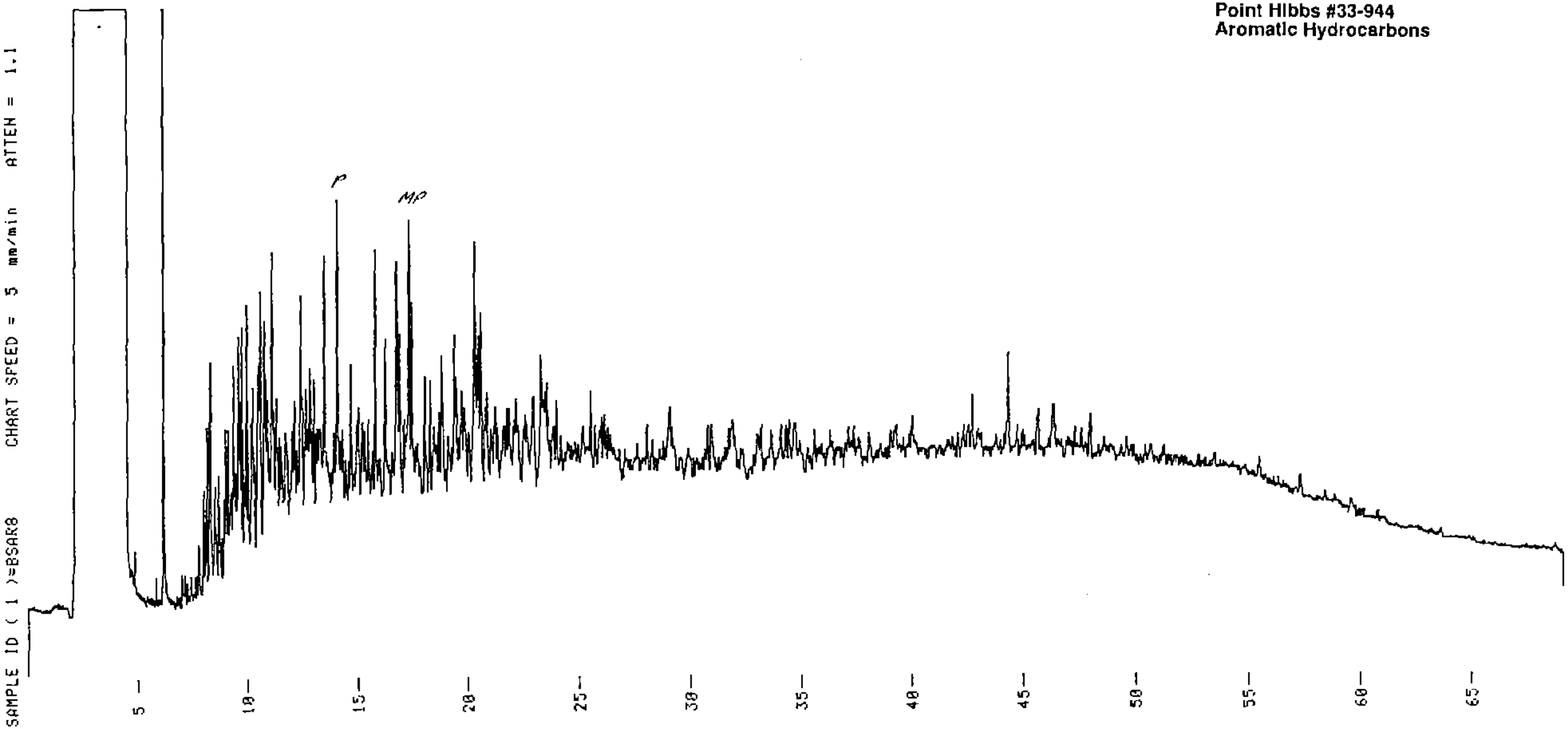
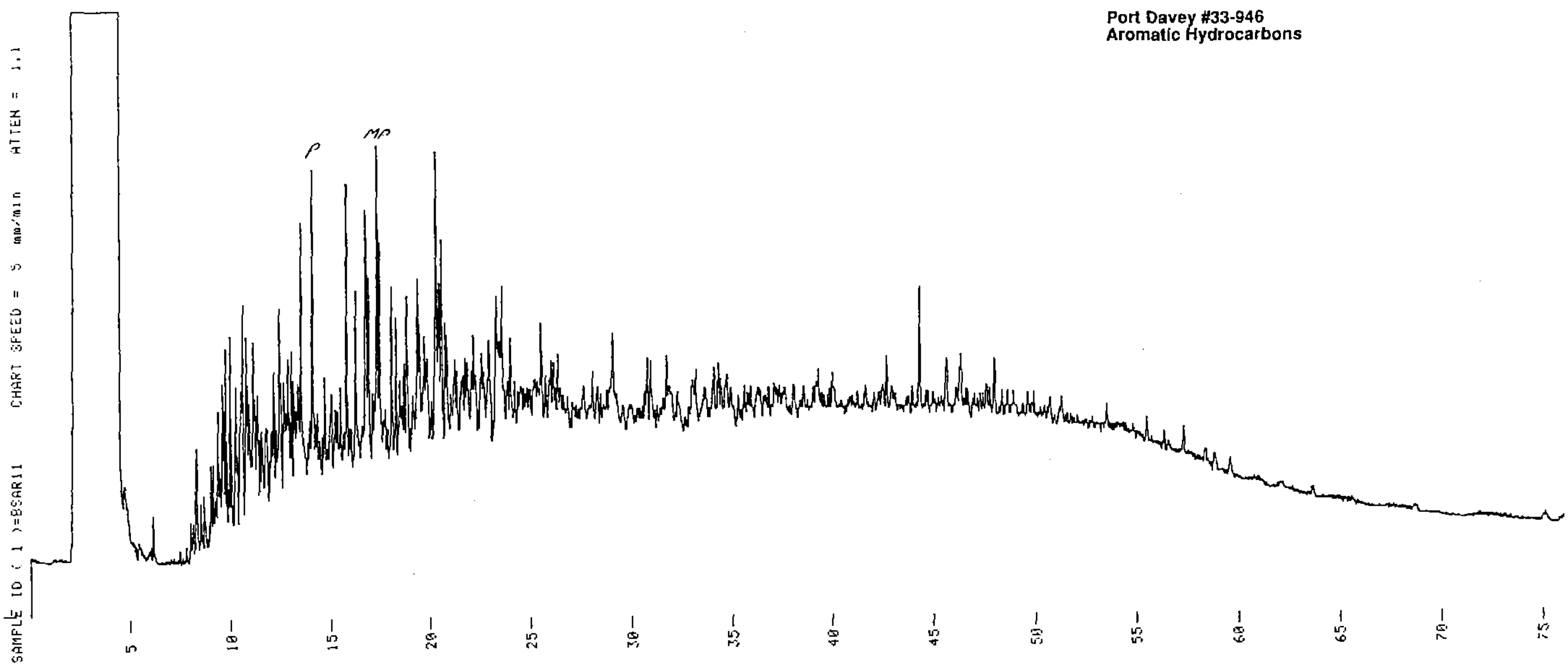
Aromatic Hydrocarbons

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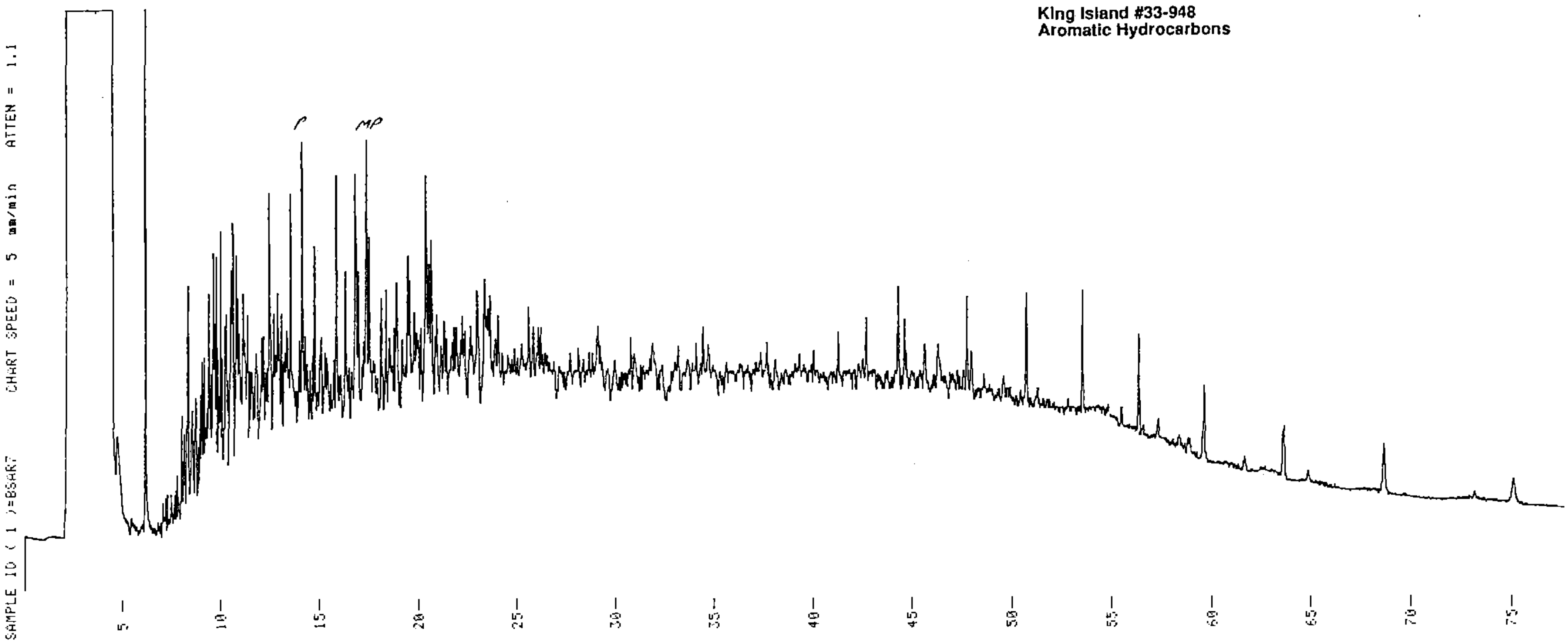
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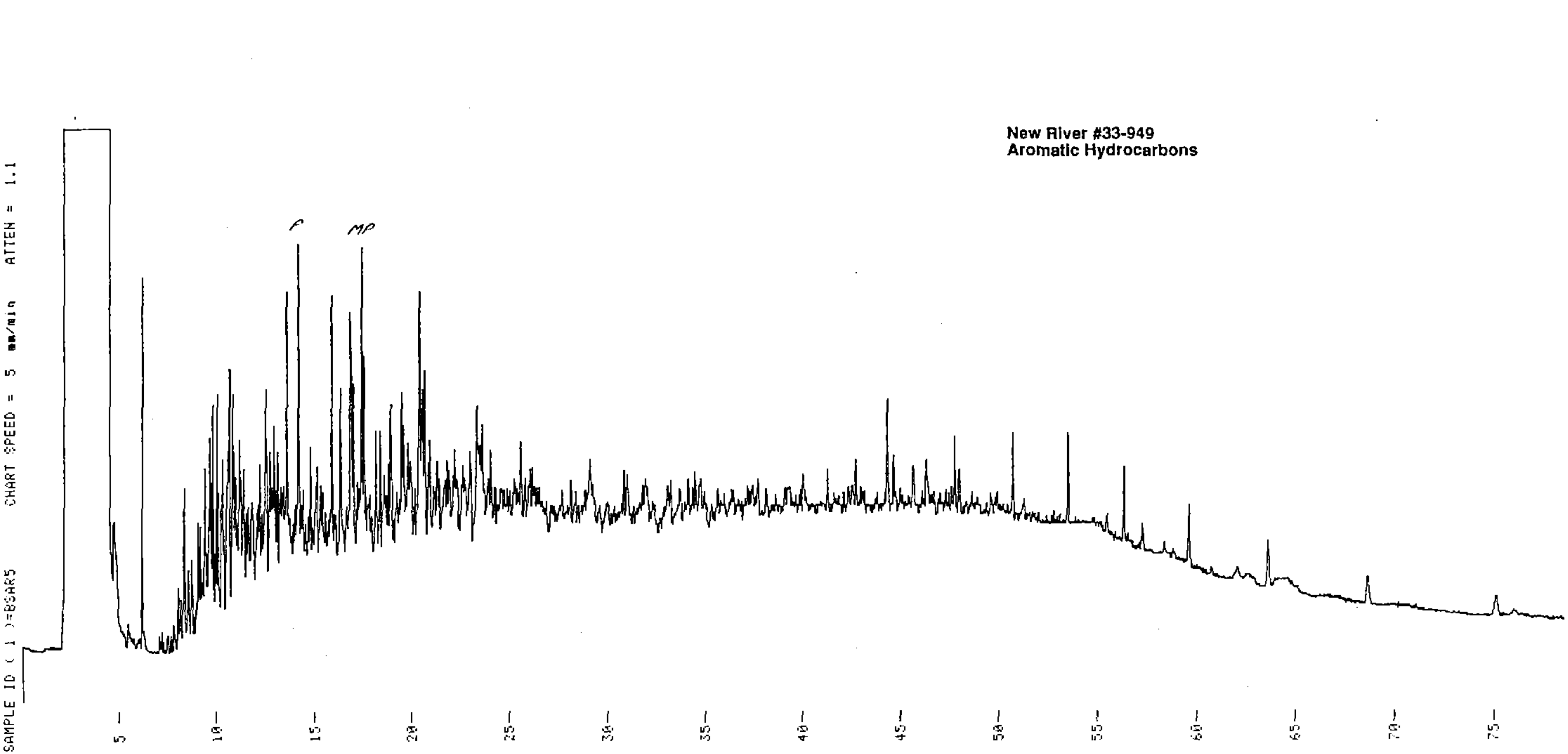
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Aromatic Hydrocarbons
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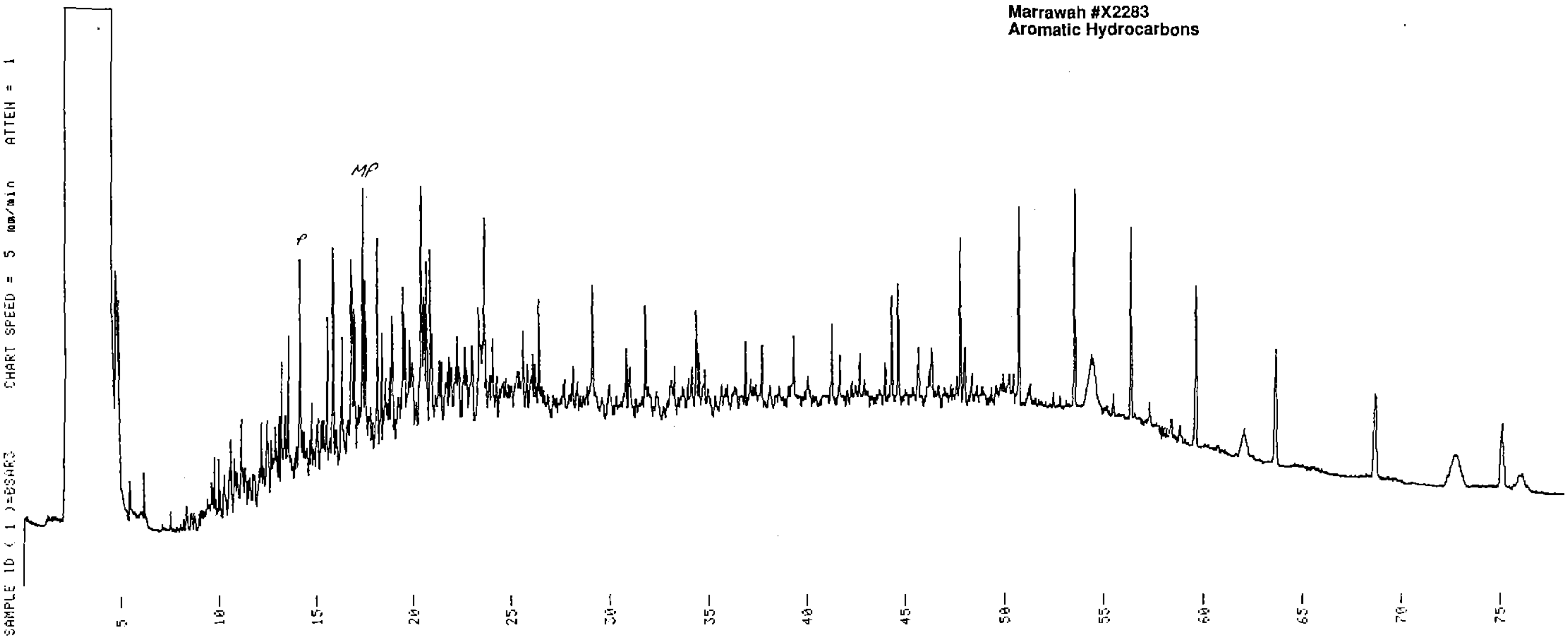
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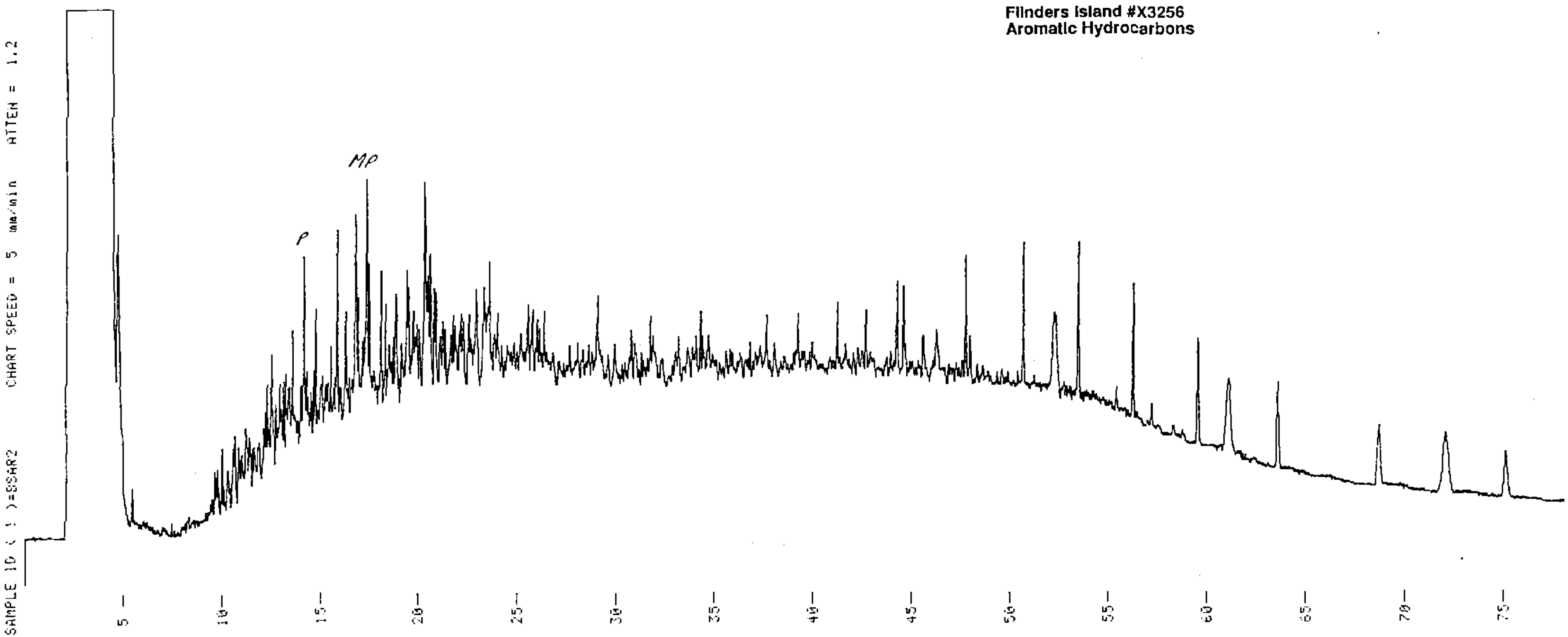
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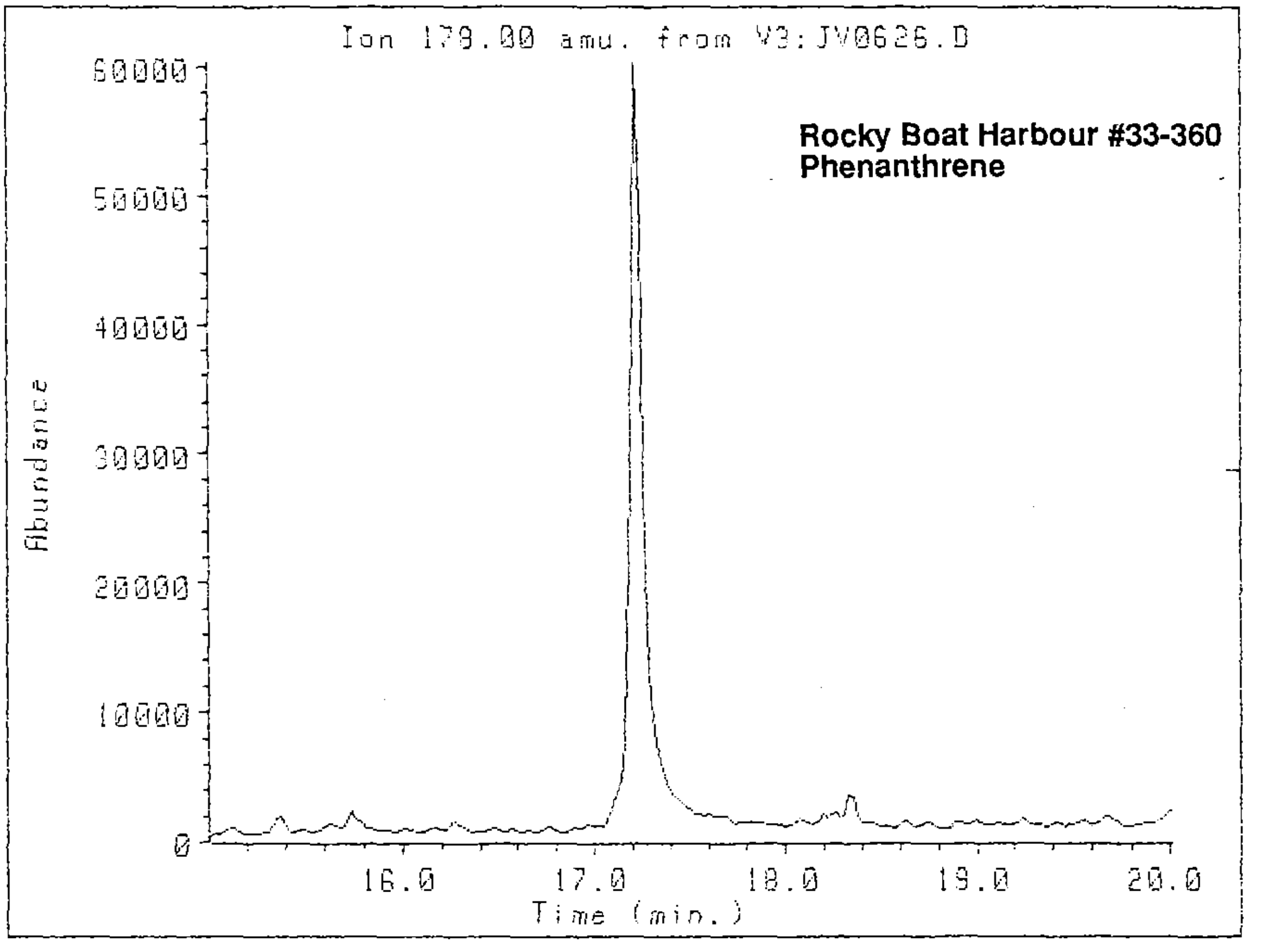
APPENDIX

GC–MS data for alkyl phenanthrenes and triaromatic steroids in some Tasmanian bitumens


Ion 178.00 amu. from V3:JV0626.D
Rocky Boat Harbour #33-360
Phenanthrene
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40000
30000
20000
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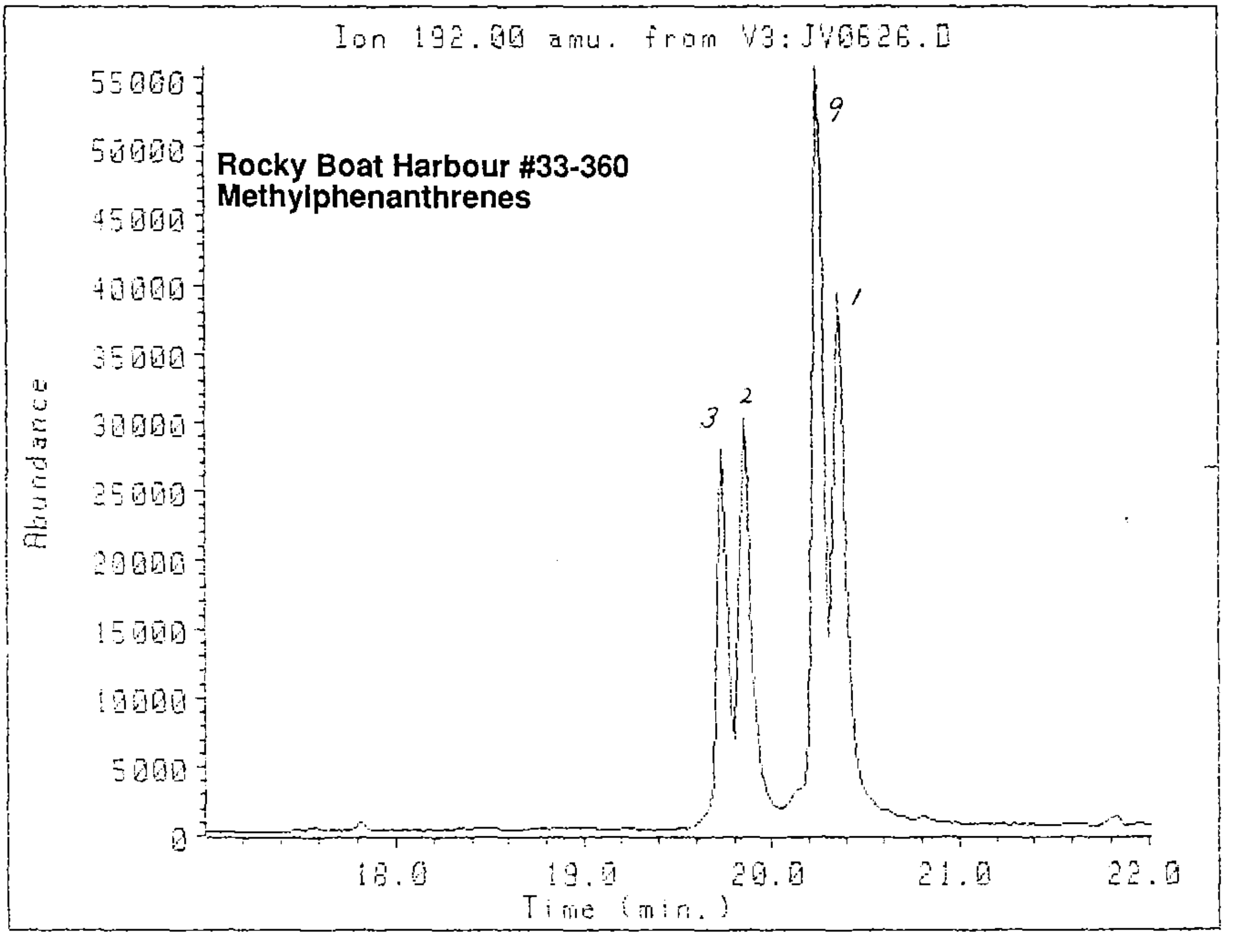
18.0
19.0
20.0
Time (min.)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Rocky Boat Harbour #33-360
Methylphenanthrenes
Abundance
55000
50000
45000
40000
35000
30000
25000
20000
15000
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2
9
1
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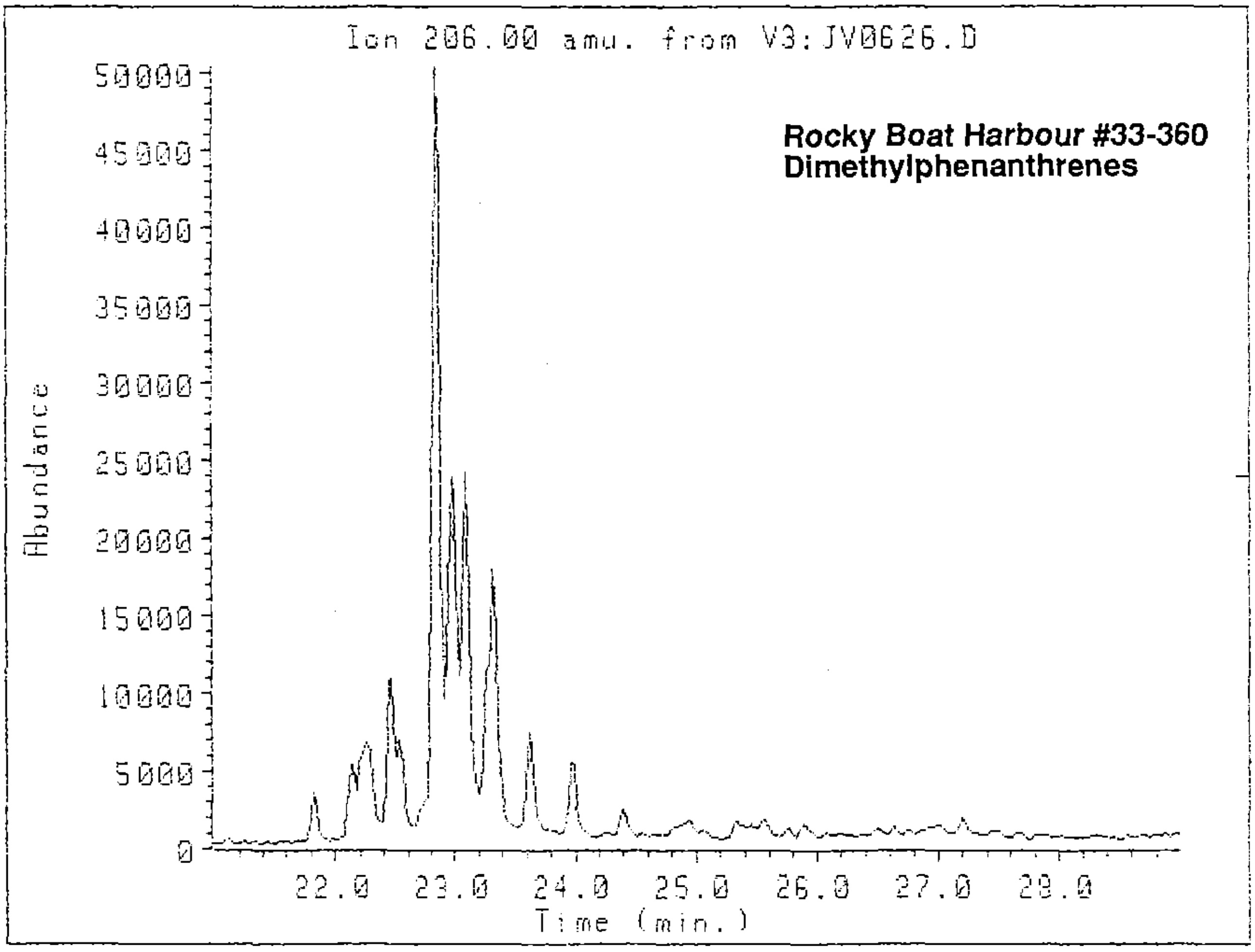
19.0
20.0
21.0
22.0
Time (min.)


Ion 206.00 amu. from V3:JV0626.D
Rocky Boat Harbour #33-360
Dimethylphenanthrenes
Abundance
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
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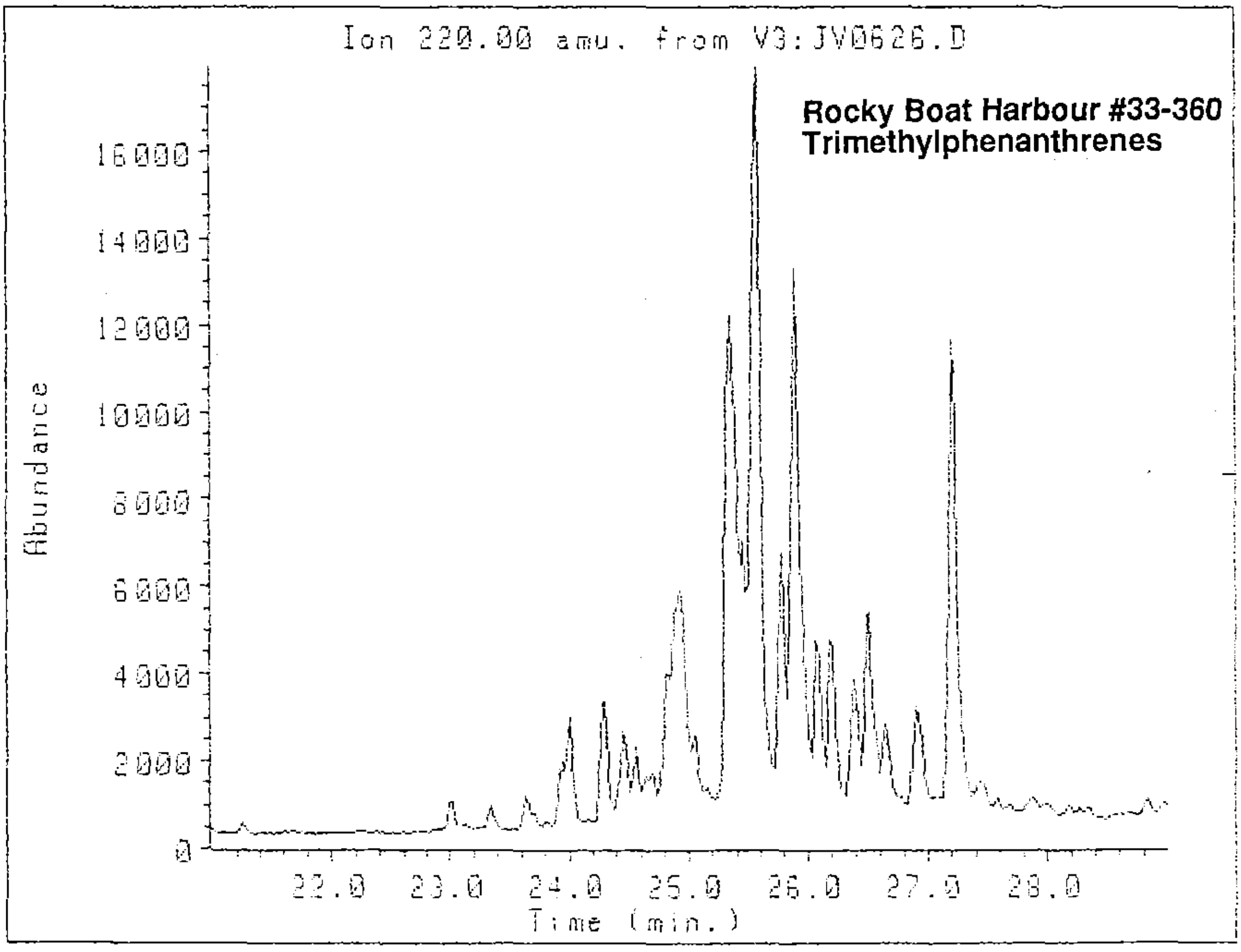
22.0
23.0
24.0
25.0
26.0
27.0
28.0
Time (min.)


Ion 220.00 amu. from V3:JV0626.D
Rocky Boat Harbour #33-360
Trimethylphenanthrenes
Abundance
16000
14000
12000
10000
8000
6000
4000
2000
0
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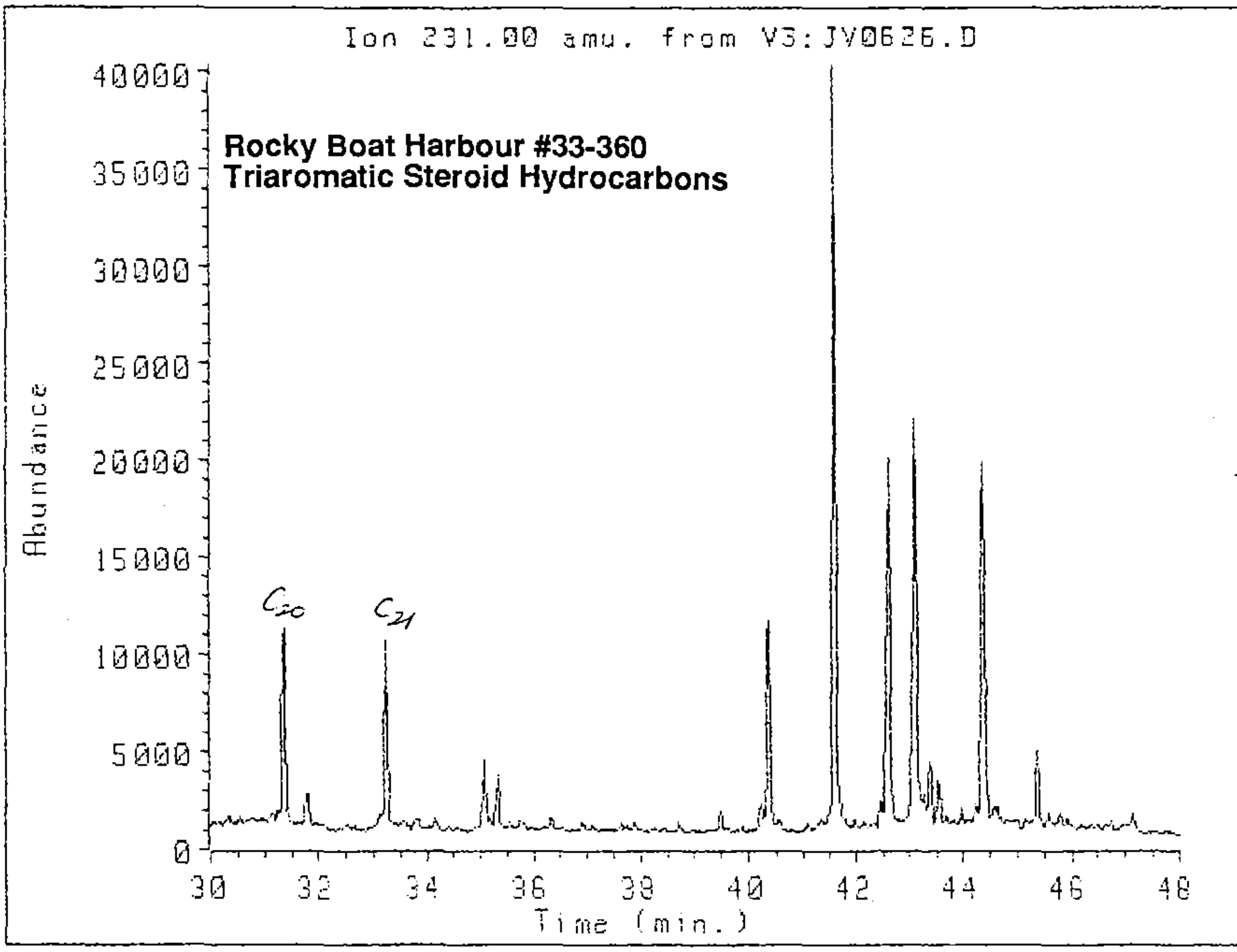
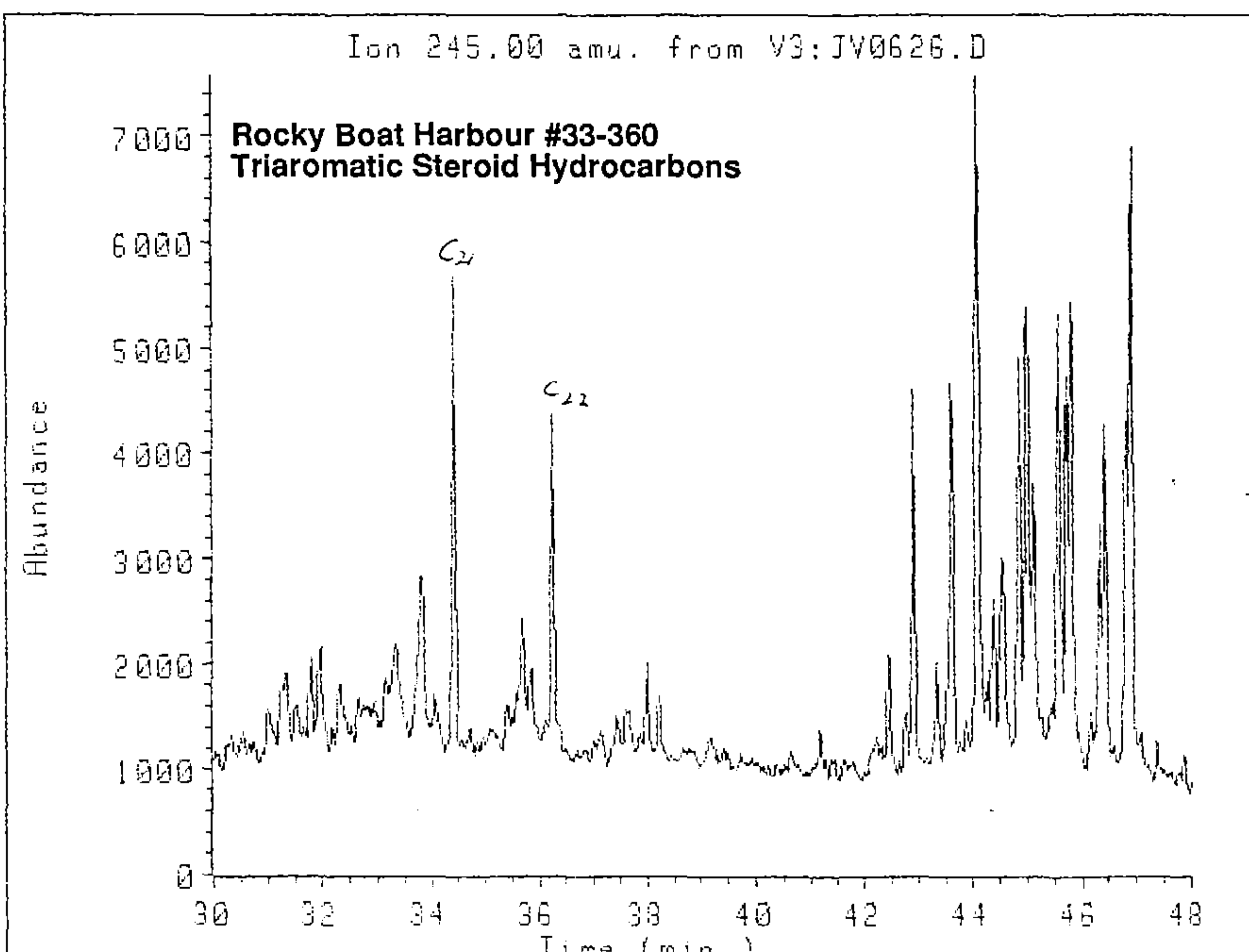
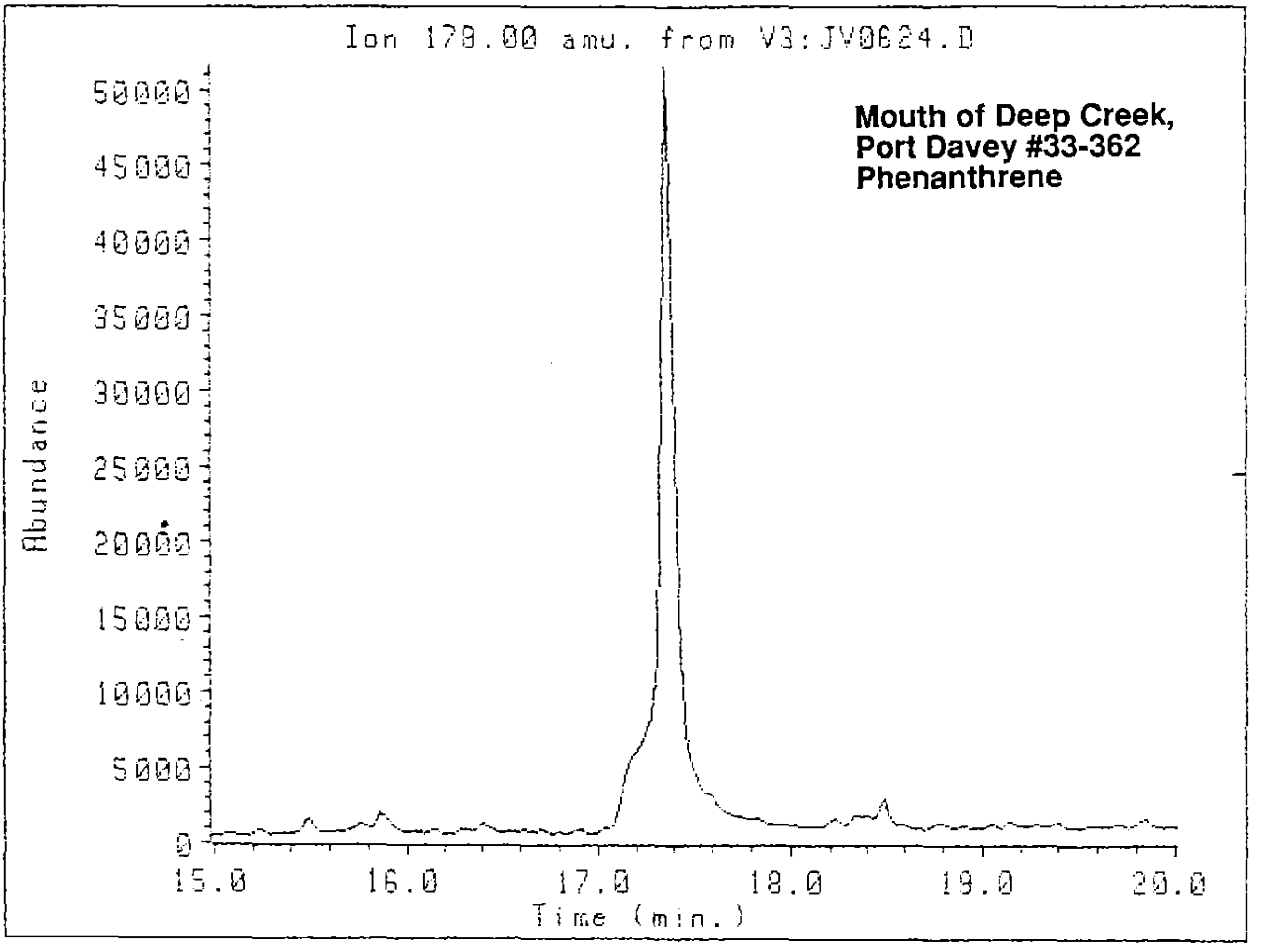
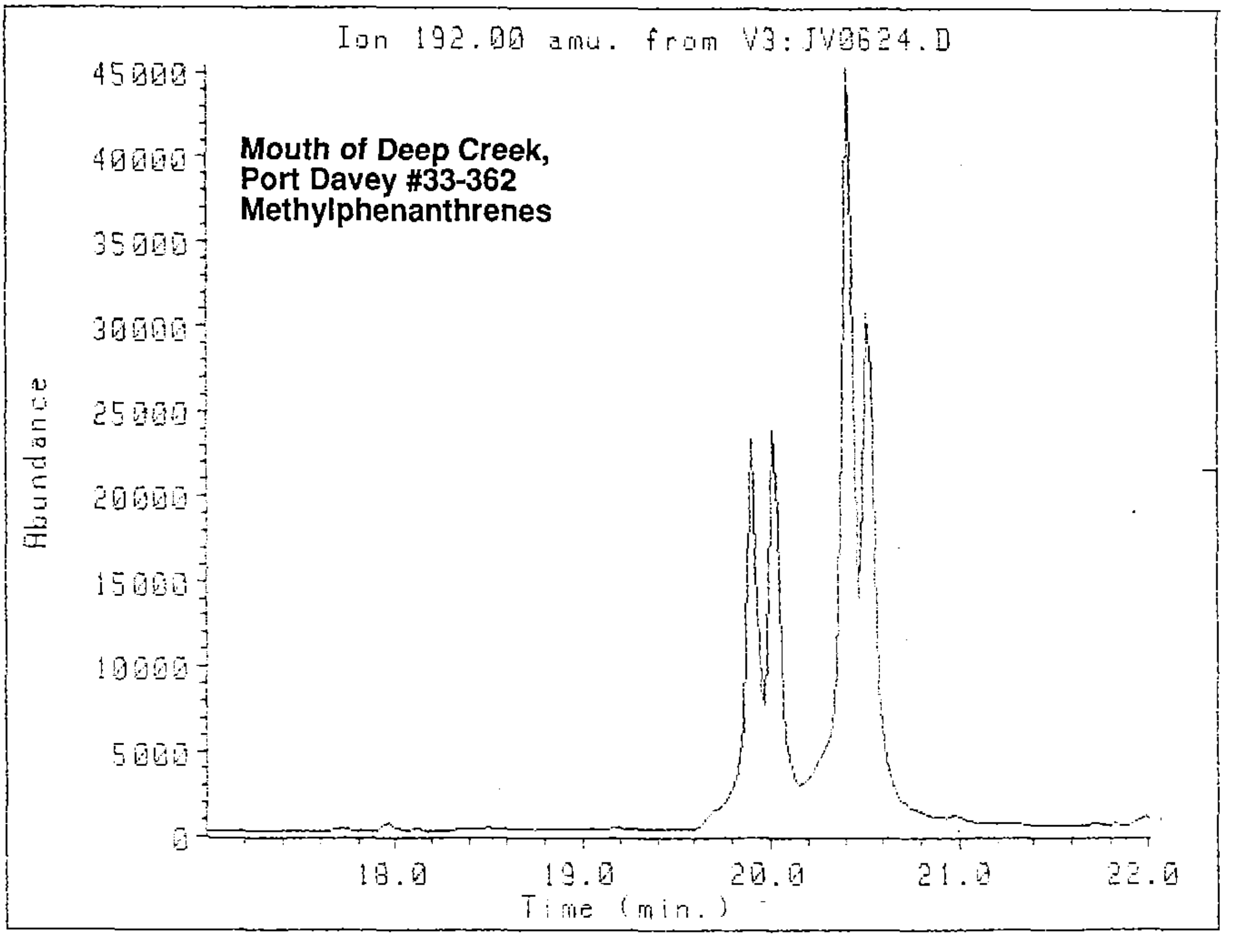
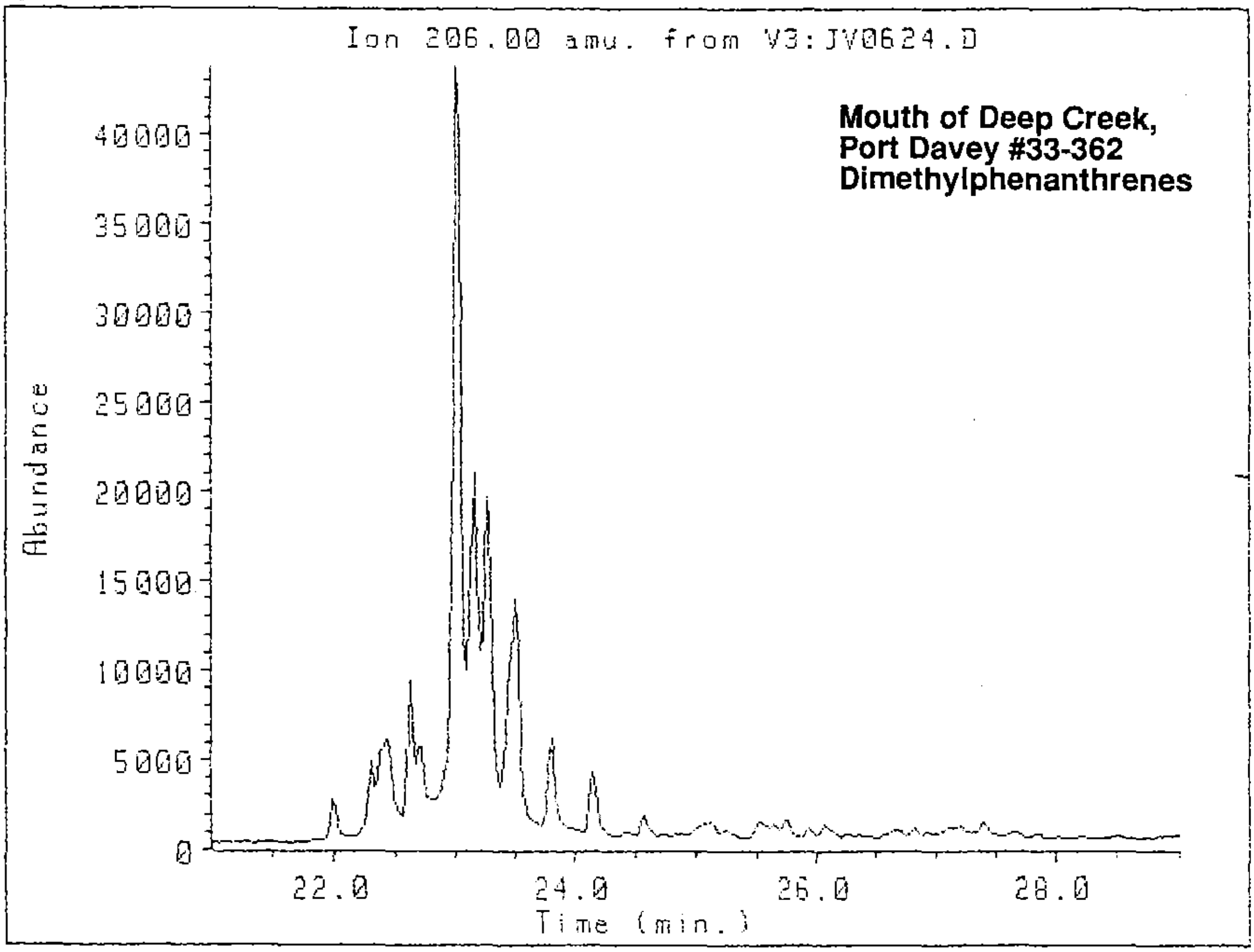
23.0
24.0
25.0
26.0
27.0
28.0
Time (min.)

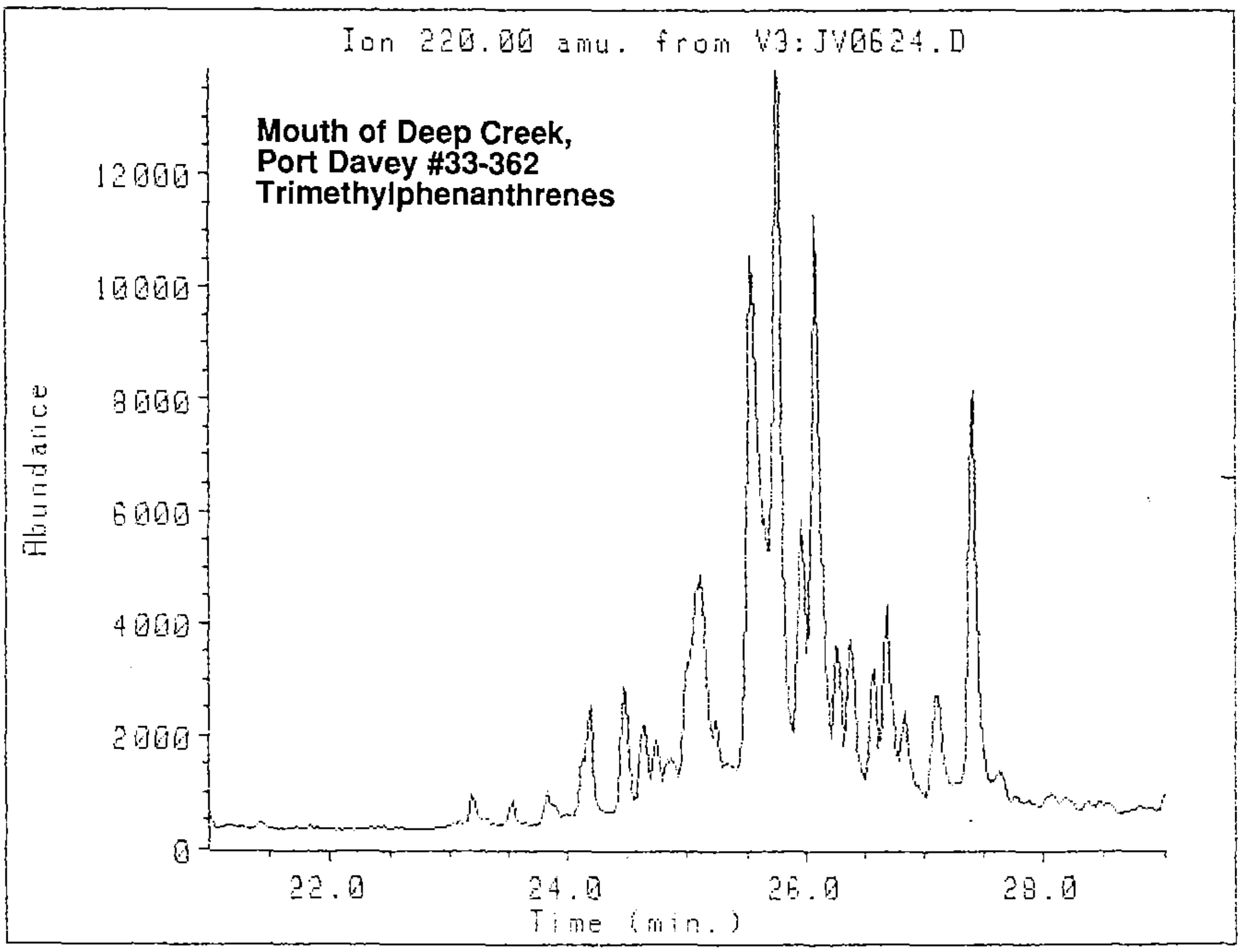

Ion 231.00 amu. from V3:JV0626.D
Rocky Boat Harbour #33-360
Triaromatic Steroid Hydrocarbons
Abundance
40000
35000
30000
25000
20000
15000
10000
5000
0
C20
C21
30
32
34
36
38
40
42
44
46
48
Time (min.)

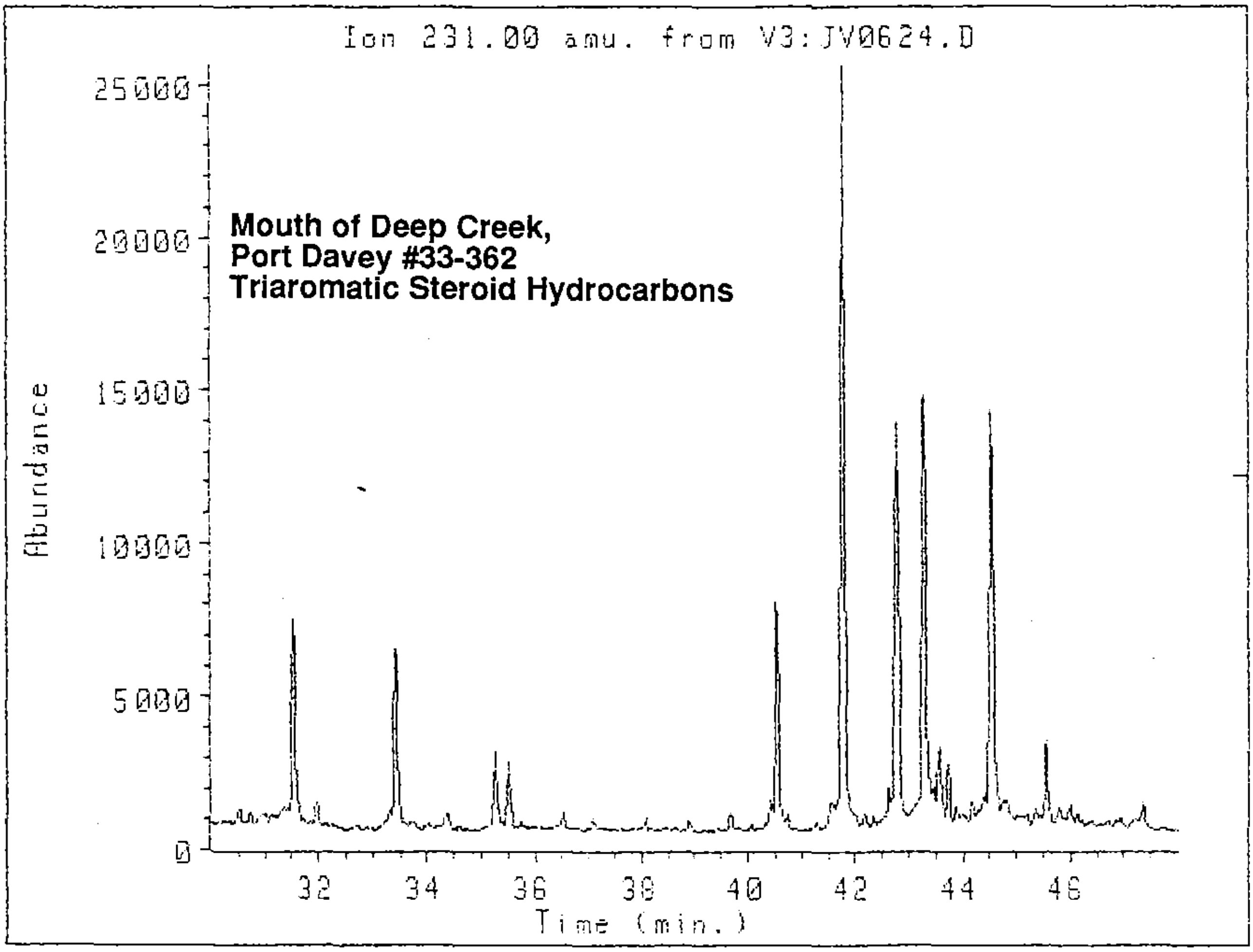

Ion 245.00 amu. from V3:JV0626.D
Rocky Boat Harbour #33-360
Triaromatic Steroid Hydrocarbons
Abundance
7000
6000
5000
4000
3000
2000
1000
0
C21
C22
30
32
34
36
38
40
42
44
46
48
Time (min.)

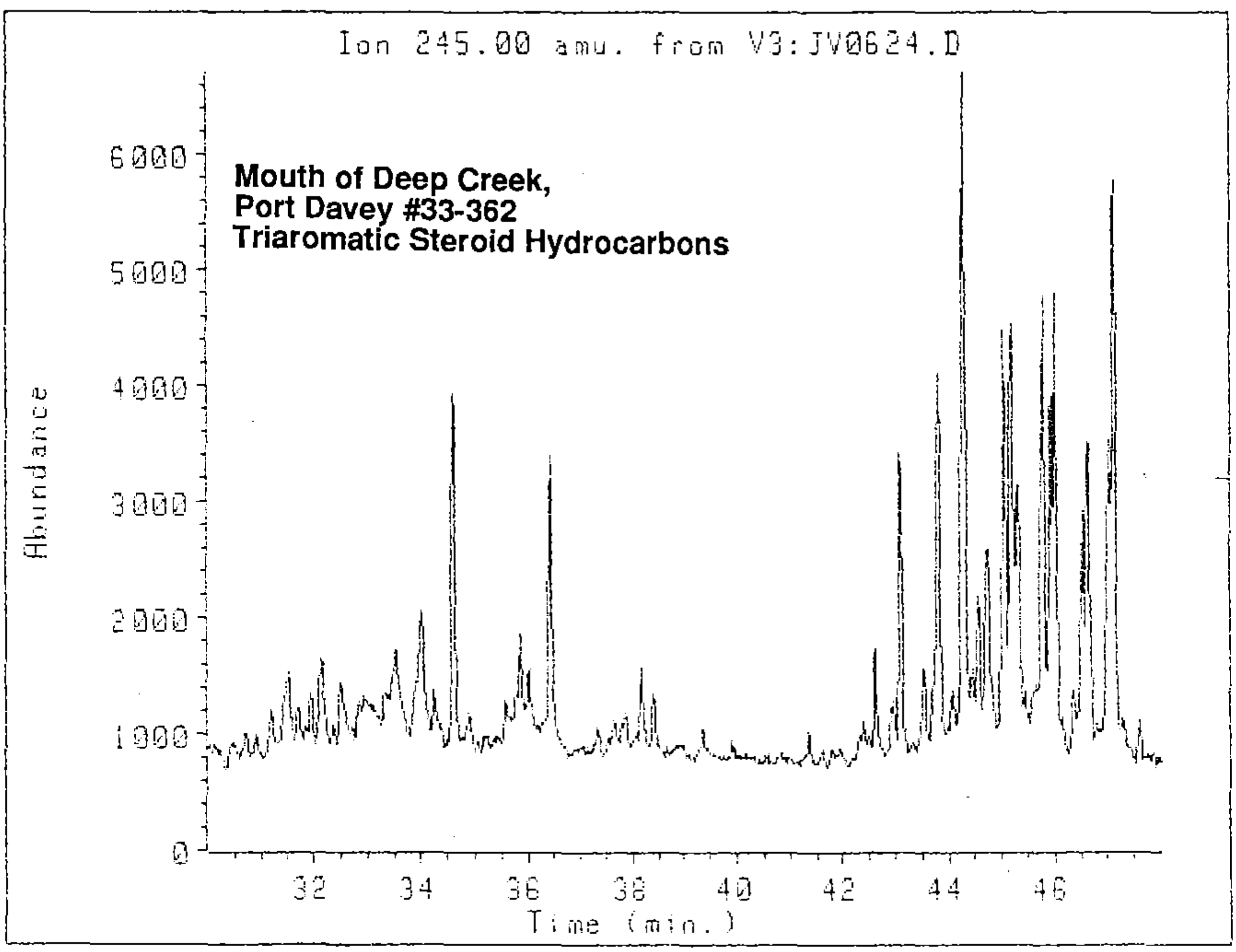

Ion 178.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Phenanthrene
Abundance
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
15.0
16.0
17.0
18.0
19.0
20.0
Time (min.)

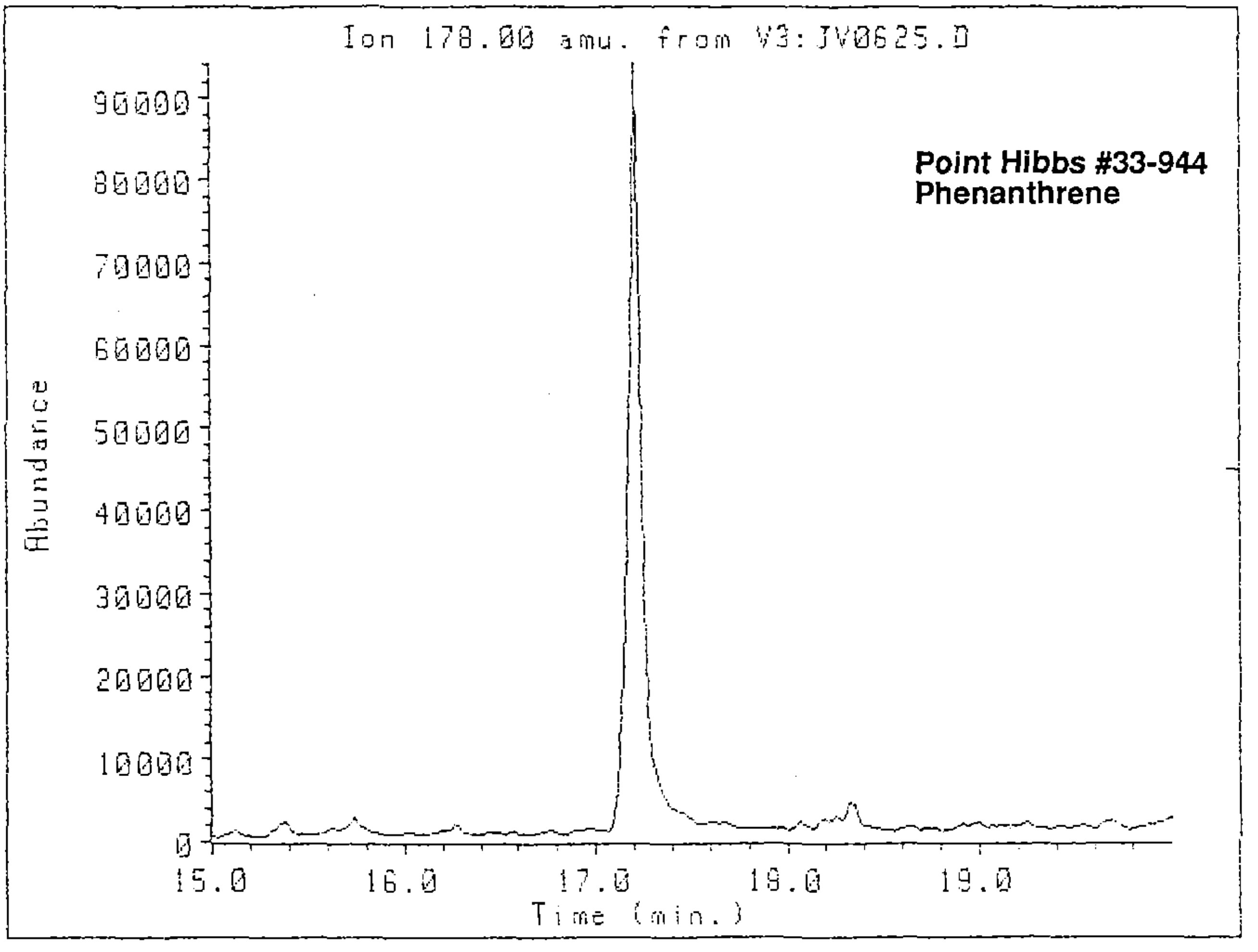

Ion 192.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Methylphenanthrenes
Abundance
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
18.0
19.0
20.0
21.0
22.0
Time (min.)

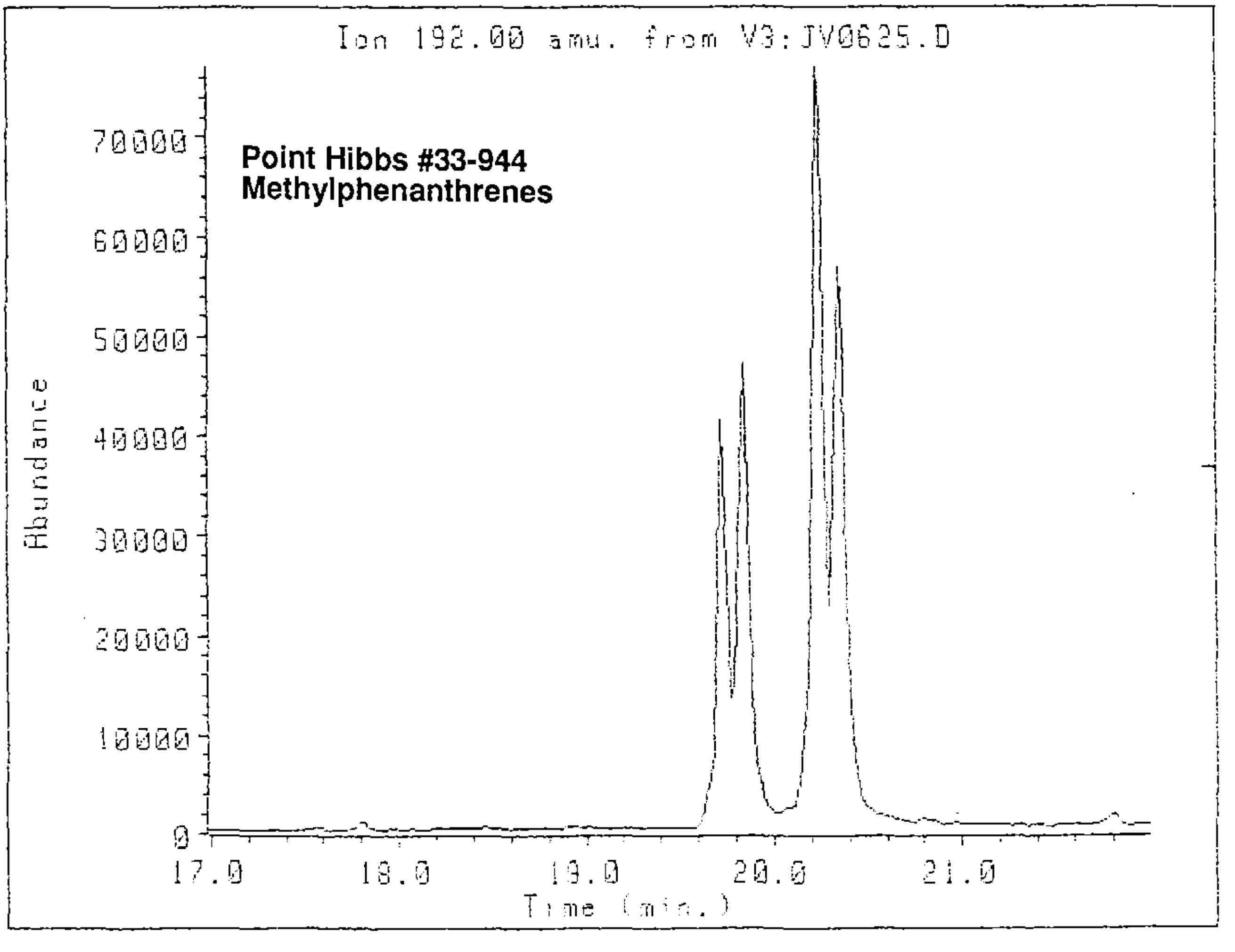

Ion 206.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Dimethylphenanthrenes
Abundance
40000
35000
30000
25000
20000
15000
10000
5000
0
22.0
24.0
26.0
28.0
Time (min.)

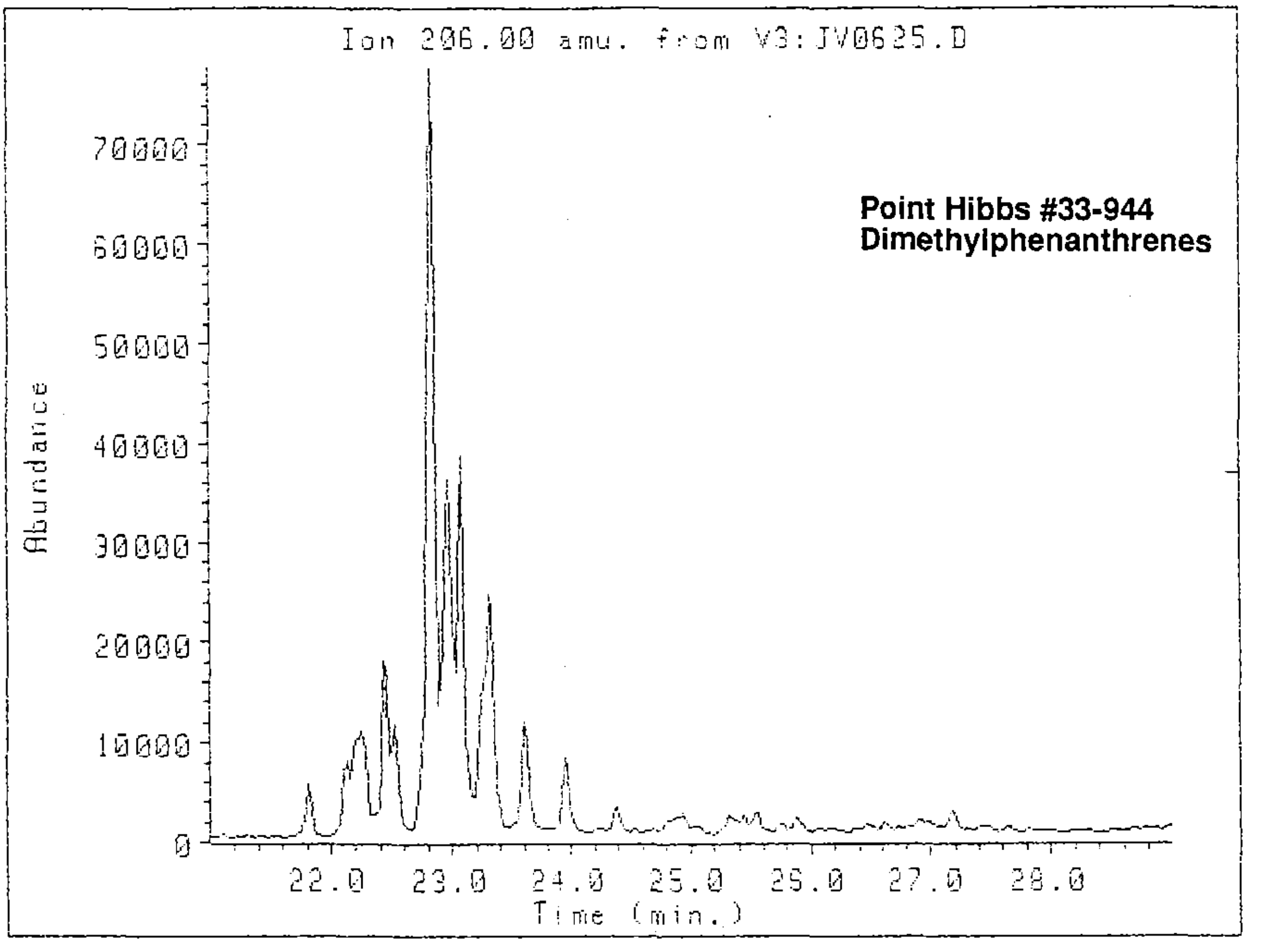

Ion 220.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Trimethylphenanthrenes
Abundance
12000
10000
8000
6000
4000
2000
0
22.0
24.0
26.0
28.0
Time (min.)


Ion 231.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Triaromatic Steroid Hydrocarbons
Abundance
25000
20000
15000
10000
5000
0
32
34
36
38
40
42
44
46
Time (min.)


Ion 245.00 amu. from V3:JV0624.D
Mouth of Deep Creek,
Port Davey #33-362
Triaromatic Steroid Hydrocarbons
Abundance
6000
5000
4000
3000
2000
1000
0
32
34
36
38
40
42
44
46
Time (min.)


Ion 178.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Phenanthrene
Abundance
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
15.0
16.0
17.0
18.0
19.0
Time (min.)


Ion 192.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Methylphenanthrenes
Abundance
70000
60000
50000
40000
30000
20000
10000
0
17.0
18.0
19.0
20.0
21.0
Time (min.)


Ion 206.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Dimethylphenanthrenes
Abundance
70000
60000
50000
40000
30000
20000
10000
0
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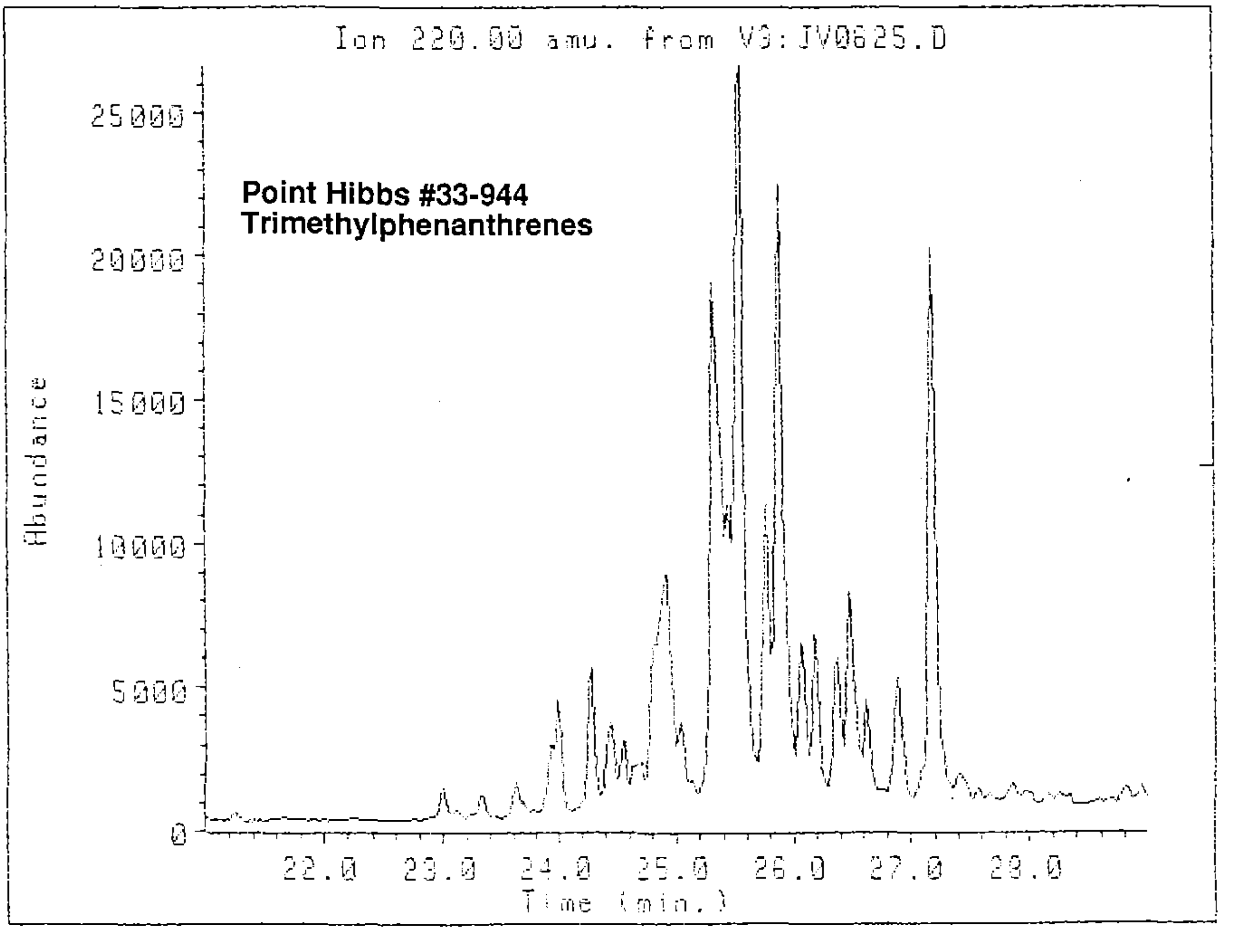
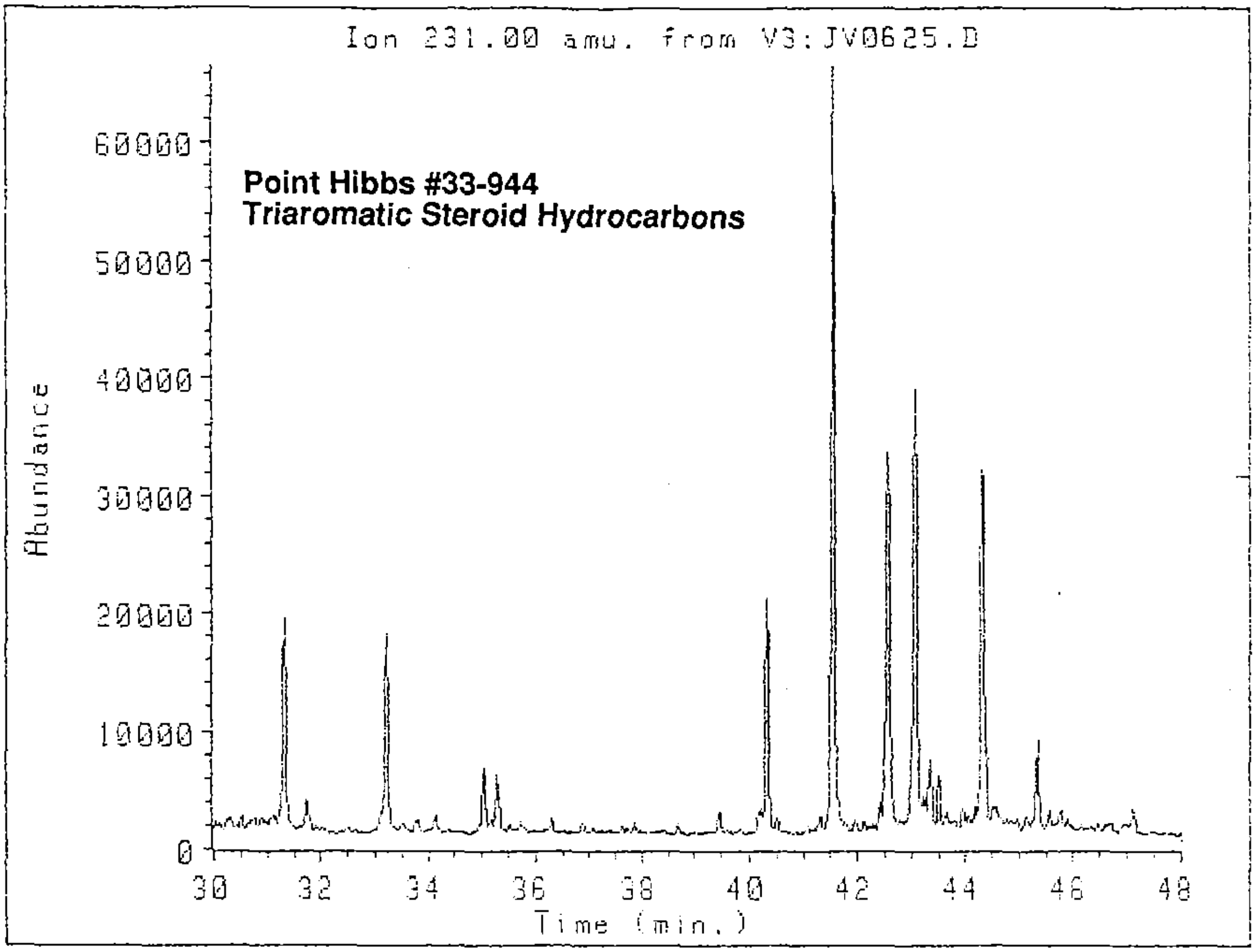
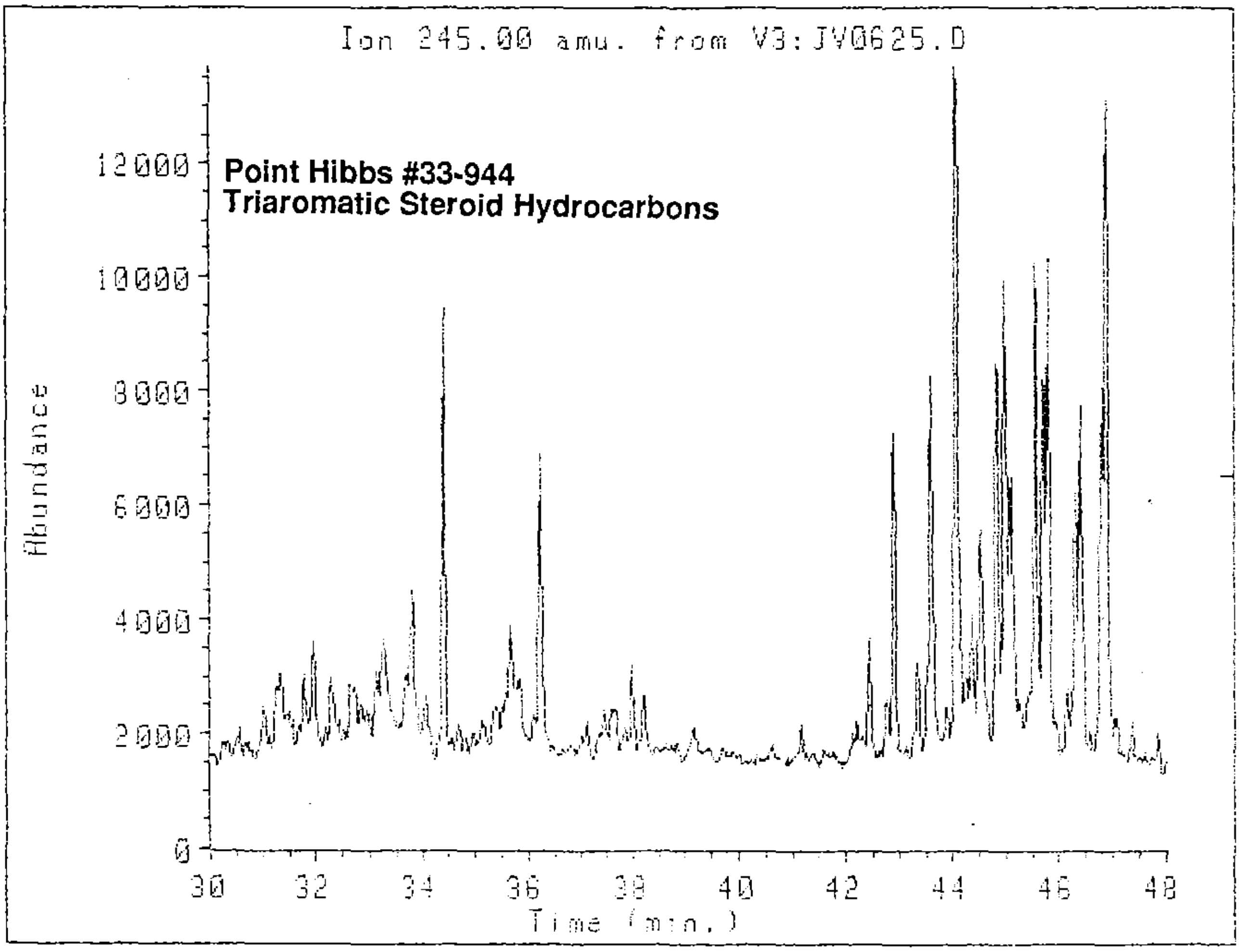
23.0
24.0
25.0
26.0
27.0
28.0
Time (min.)


Ion 220.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Trimethylphenanthrenes
Abundance
25000
20000
15000
10000
5000
0
22.0
23.0
24.0
25.0
26.0
27.0
28.0
Time (min.)


Ion 231.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Triaromatic Steroid Hydrocarbons
Abundance
60000
50000
40000
30000
20000
10000
0
30
32
34
36
38
40
42
44
46
48
Time (min.)


Ion 245.00 amu. from V3:JV0625.D
Point Hibbs #33-944
Triaromatic Steroid Hydrocarbons
Abundance
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10000
8000
6000
4000
2000
0
30
32
34
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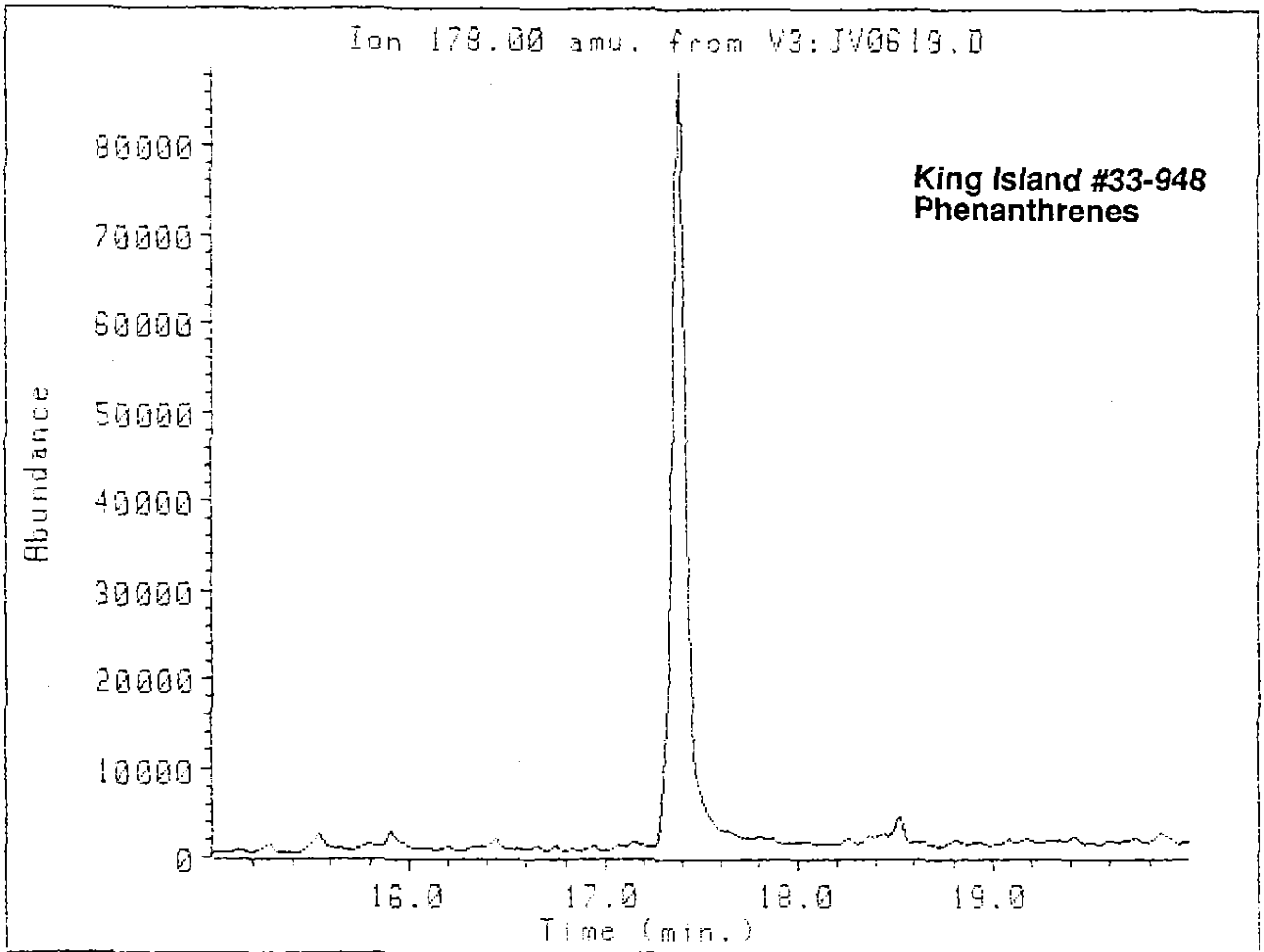
38
40
42
44
46
48
Time (min.)


Ion 178.00 amu. from V3:JV0619.D
King Island #33-948
Phenanthrenes
Abundance
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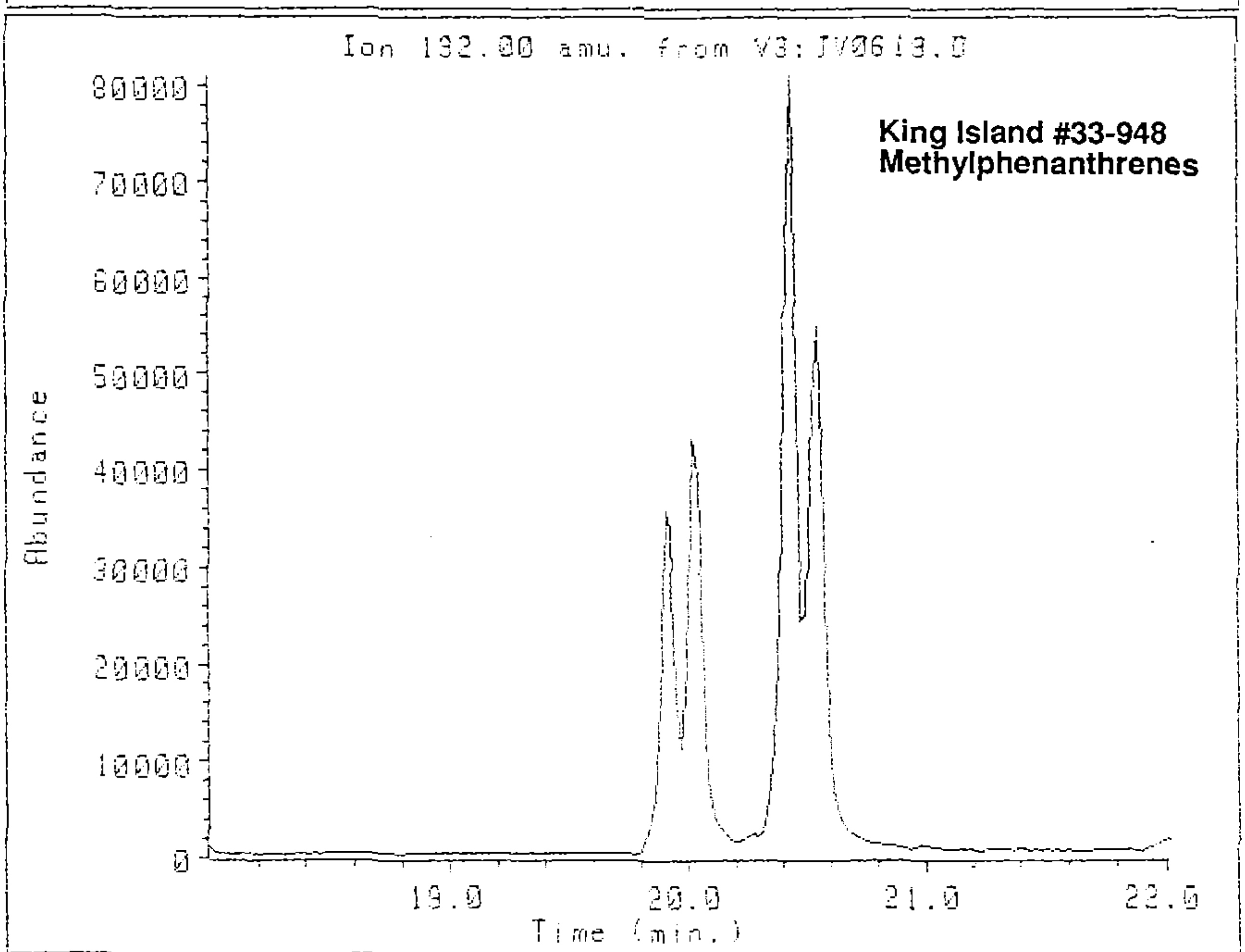
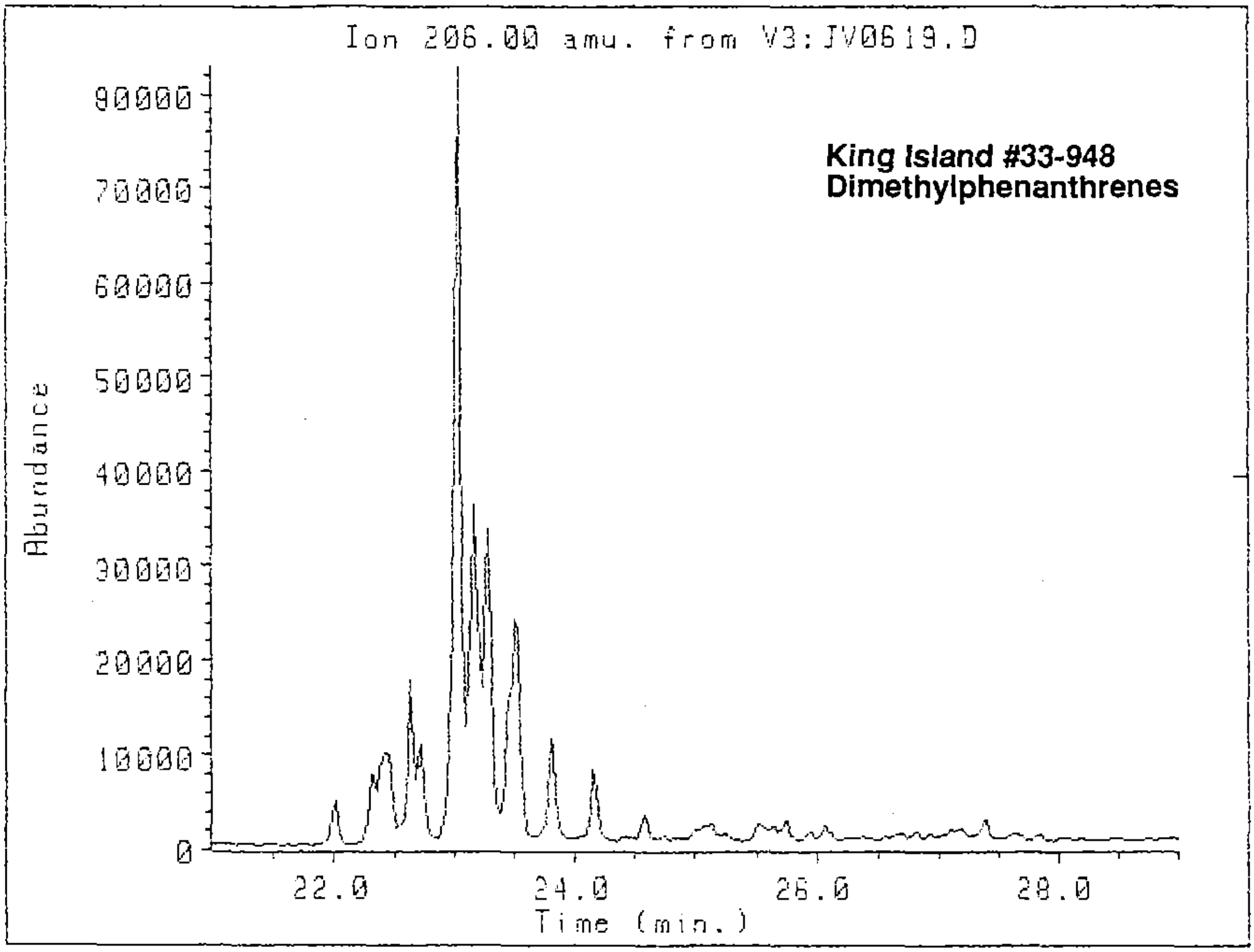
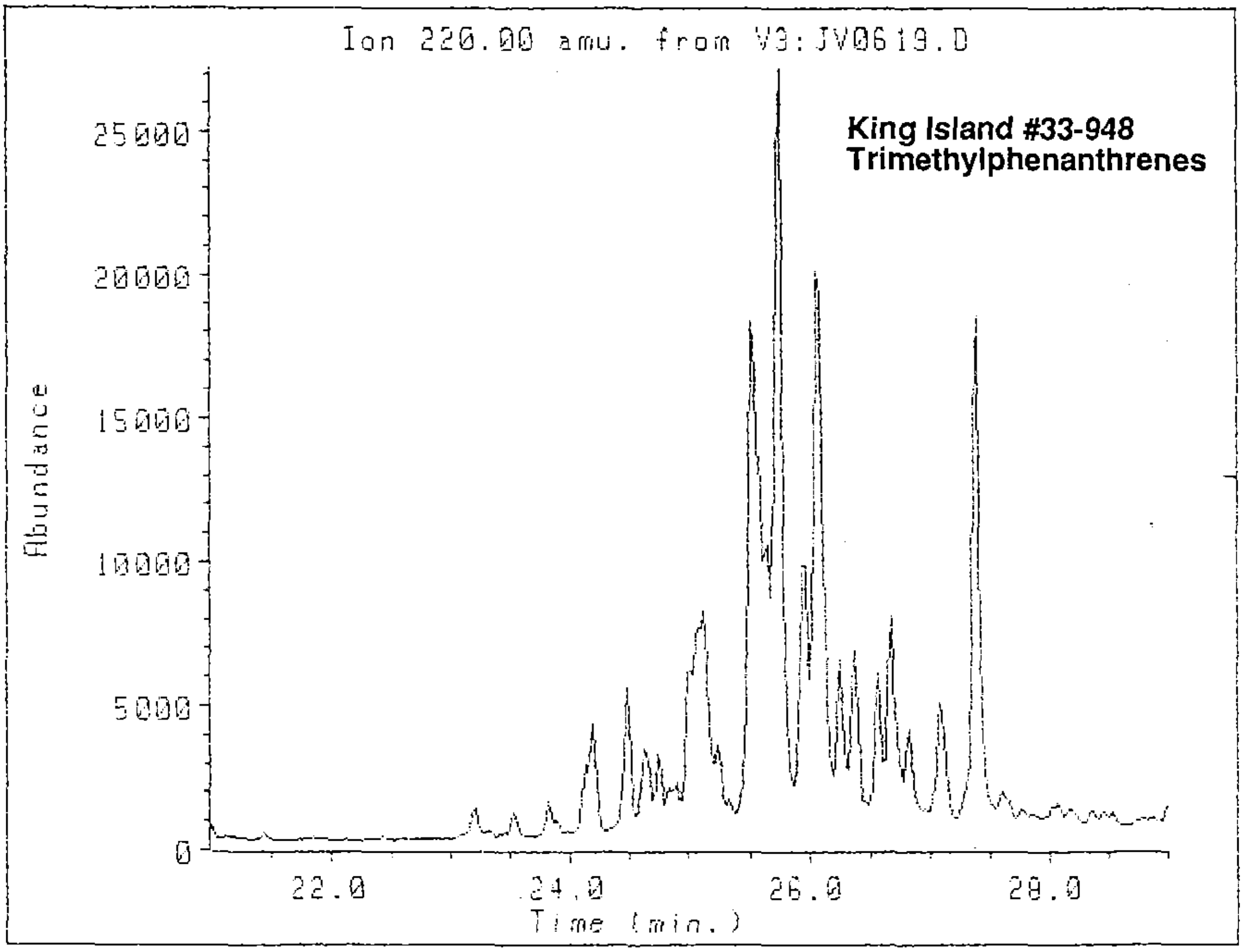
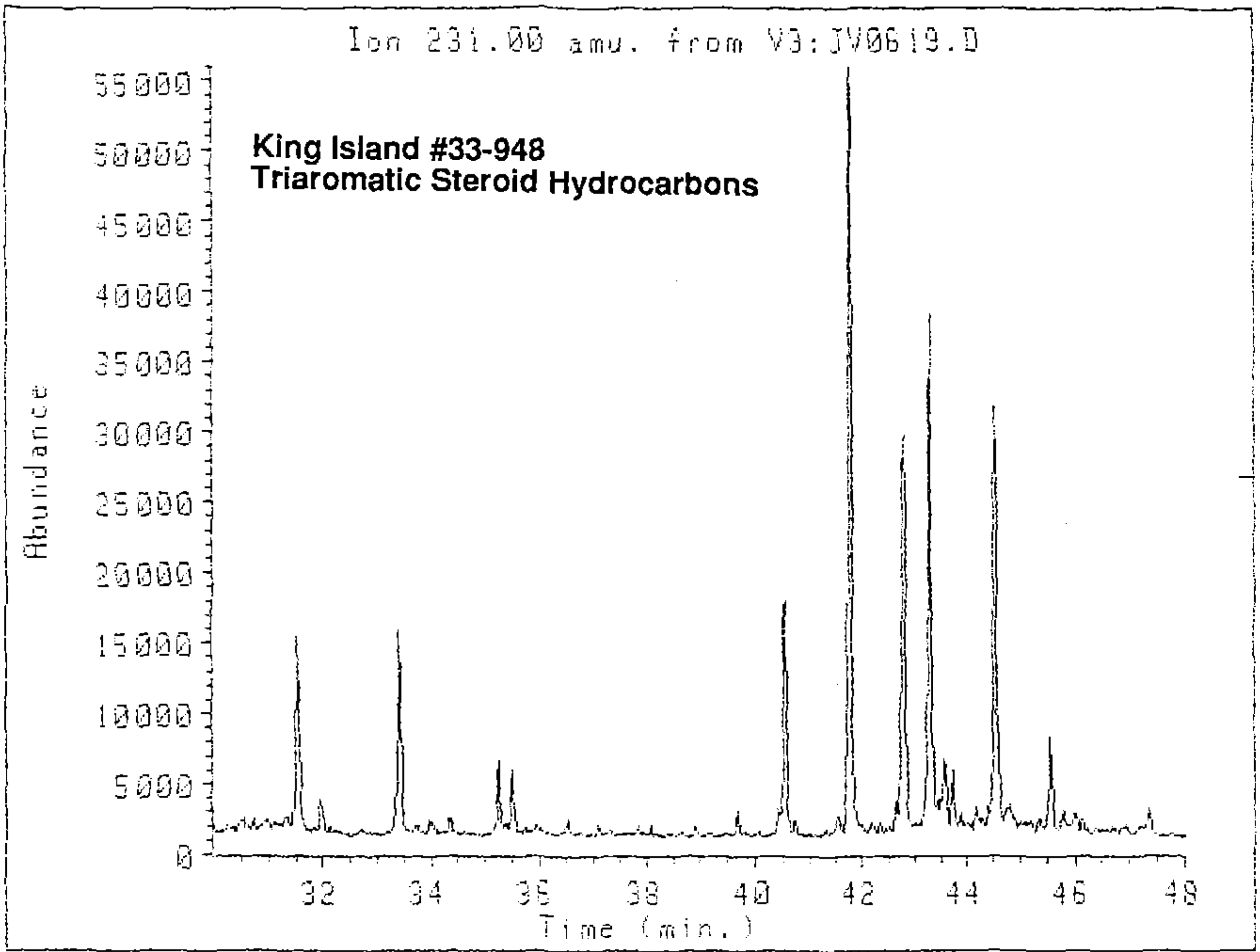
70000
60000
50000
40000
30000
20000
10000
0
16.0
17.0
18.0
19.0
Time (min.)


Ion 192.00 amu. from V3:JV0619.D
King Island #33-948
Methylphenanthrenes
Abundance
80000
70000
60000
50000
40000
30000
20000
10000
0
19.0
20.0
21.0
22.0
Time (min.)


Ion 206.00 amu. from V3:JV0619.D
King Island #33-948
Dimethylphenanthrenes
Abundance
90000
70000
60000
50000
40000
30000
20000
10000
0
22.0
24.0
26.0
28.0
Time (min.)


Ion 220.00 amu. from V3:JV0619.D
King Island #33-948
Trimethylphenanthrenes
Abundance
25000
20000
15000
10000
5000
0
22.0
24.0
26.0
28.0
Time (min.)


Ion 231.00 amu. from V3:JV0619.D
King Island #33-948
Triaromatic Steroid Hydrocarbons
Abundance
Time (min.)
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
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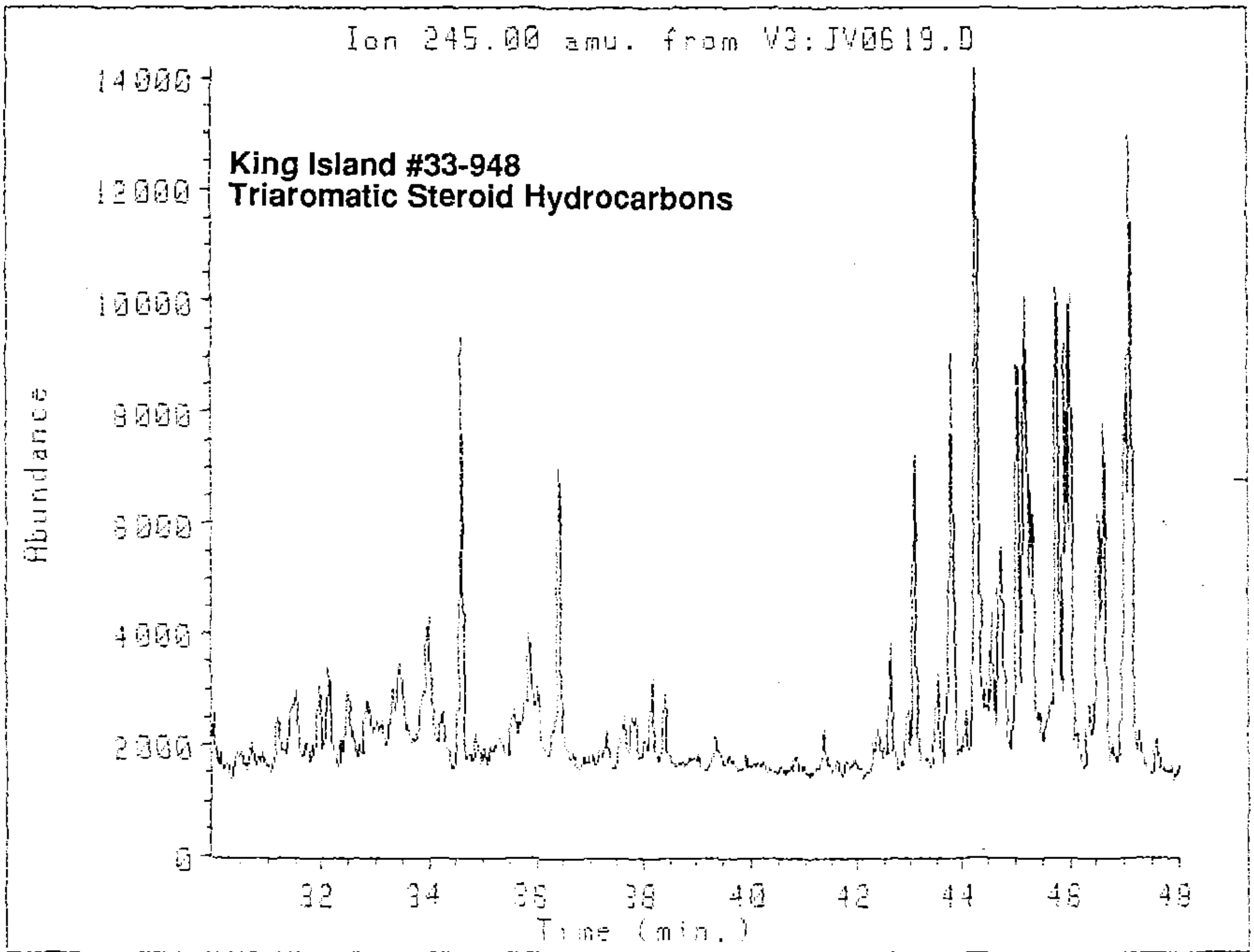
34
36
38
40
42
44
46
48


Ion 245.00 amu. from V3:JV0619.D
King Island #33-948
Triaromatic Steroid Hydrocarbons
Abundance
Time (min.)
14000
12000
10000
8000
6000
4000
2000
0
32
34
36
38
40
42
44
46
48