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REPORT TITLE/TITTE	Analyses on Sa	mples from We	11 6609/7-1.
CLIENT/ OPPDRAGSGIV	etroleum Co. N	lorway	FILE
RESPONSIBLE SCIENTI	ST/ PROSJEKTANSVADETO	LOGY	
G. van Grae	as, 0.M. Bakke	en and I.K. Al	mås
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SUMMARY/ SAMMENDRAG

Eight claystones and two dolomites from well 6609/7-1 were analysed by thermal extraction/pyrolysis gas chromatography. The claystones were also solvent extracted and the saturated and aromatic hydrocarbons were analysed by gas chromatography.

The results correspond with those based on the screening analyses. The entire interval contains poor type III or type IV kerogen which is immature to marginally mature. There is a poor to fair potential as gas source rock.

The presence of migrated hydrocarbons is suggested in several samples. This is strongest for A-7174 which is located in the sandstone containing interval that showed high amounts of petrogenic gas in the screening analyses.

Source Rock Analysis	6609/7-1	
Trænabanken	Follow-up Analyses	



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INTRODUCTION

This report contains the results of follow-up analyses on samples from well 6609/7-1 (figs. 1 and 2). Selection of samples to be analysed is based on the results of screening analyses from the same well (JKU report 05.0140, August 17, 1983).

The general conclusion from the screening report was that the interval analysed (1500-1969m) has a poor to fair potential as source rock for gas only. The maturity of the sediments is low. A sandstone interval (1830-1880m) contains migrated hydrocarbon gas, probably of petrogenic origin.

Based on these results it was decided to select 8 claystones from zones A, B and C and two dolomites from zone D for follow-up analyses. All samples have been analysed by thermal evaporation/pyrolysis gas chromatography and in addition the extractable hydrocarbons from the claystones have been separated and analysed by gas chromatography.



EXPERIMENTAL

Extractable Organic Matter

Cuttings of the selected lithology were picked from the 2-4mm fraction. The powdered rock was extracted by a flow-blending technique using dichloromethane (DCM) as solvent. The DCM used was of organic geochemical grade and blank analyses showed the occurrence of negligible amounts of contaminating hydrocarbons.

Activated copper fillings were used to remove any free sulphur from the samples.

After extraction the solvent was removed on a Buchi Rotavapor and the amount of extractable organic matter (EOM) was determined.

Chromatographic Separation

The extractable organic matter (EOM) was separated into saturated fraction, aromatic fraction and non hydrocarbon fraction using a MPLC system with hexane as eluant (Radke et al., Anal. Chem., 1980, pp.406-411). The various fractions were evaporated on a Buchi Rotavapor and transferred to glass vials and dried in stream of nitrogen.

Gas Chromatographic Analysis

The saturated hydrocarbon fractions were each diluted with n-hexane and analysed on a HP 5730A gas chromatograph, fitted with a 25m OV-101 fused silica capillary column. Hydrogen (0.7ml/min) was used as carrier gas. The total aromatic fractions were after dilution with n-hexane, analysed on a VARJAN 3700 GC-fitted with a 15m DB-1 fused silica column.

Injections were performed in the split mode (1:20). The temperature program applied was 80° C (2 min) to 260° C at 4° C/min.

The data processing for all the GC analyses was performed on a VG Multichrom System.



Thermal Extraction GC

20-30mg of fine ground whole rock sample was placed in a boat shaped sample probe and heated in a stream of helium at 300° C for five minutes. The extract was flushed directly into a capillary column via a laboratory built interface/splitter.

A 25m OV-1 fused silica column was fitted in the Varian 3700 gas chromatograph, and a temperature program of 40° C/min to 270° C/min at 4° C/min was employed. Nitrogen (1.5ml/min) was used as carrier gas and injections were performed in split mode (1:30).

Py-GC (Progr.)

20-30mg of thermally extracted whole rock sample was pyrolysed in a stream helium (300° C to 550° C at 35° C/min.) using a tube furnace type pyrolyzer interfaced to a Varian 3700 gas chromatograph. The outlet of the pyrolyzer was connected to a fused silica capillary column via an interface/splitter (sample/split ratio; 1:30). The pyrolysis product was trapped in a cooled (liquid nitrogen) U-shaped part at the front of the column.

The outlet of the splitter was connected to a flame ionisation detector (FID) and the course of the pyrolysis could be followed by the detector response of the bulk pyrolysis product which was recorded as a broad peak. At the end of the pyrolysis, the trapped pyrolysis product was injected by removing the liquid nitrogen bath at ambient temperature and analysed under the GC conditions given below.

Instrumental conditions:

Pyrolysis: 300°C to 550°C at 35°C/min.

Column: 25m OV-1 fused silica capillary; ID 0.3mm.

Carrier gas: Helium with inlet pressure 10 psi; Flow ca. 1.5 ml/min.

Split ratio: 1:30.

Oven program: 40°C/1 min. to 270°C at 4°C/min .



RESULTS AND DISCUSSION

Samples

The samples selected for the follow-up analyses are listed in table 1 with their lithology and TOC. Rock-Eval pyrolysis data are shown in table 2. The amount that could be picked of the lithology selected for extraction was in a number of samples too small (<5gr.). By making composite samples with similar lithologies from adjacent samples sufficient material could be obtained for A-6810 (together with A-6809) and A-6821 (with A-6820 and A-6822), but not for A-6825 and A-6827.

Extraction data

Tables 3-6 and fig. 3 contain the results of the extraction and MPLC separation. For most samples the amount of material available was minimal and this resulted in small amounts of extract (table 3). Weighing of these small amounts implies a relatively large error, this will be extra large in the size of the non-hydrocarbon fraction which is calculated from the other values. The relative amounts of hydrocarbons (normalized against sediment weight or TOC, tables 4 and 5) are good to rich for all but the two shallowest samples (A-6803 and A-6805). In fact these amounts are higher than expected based on the immaturity of the sediments involved (see the screening report) suggesting the presence of contamination or migrated hydrocarbons. A similar effect can also be observed in the Rock-Eval data (table 2) where the production index of all samples is higher than expected for immature sediments. Other indications for non-indigenous extract material are the high saturate/EOM and HC/EOM ratios in most samples (table 6).

Saturated hydrocarbons

The distribution of the n-alkanes (Appendix A) is indicative for a contribution of terrestrial material to all samples (as exemplified by the presence of ${\rm C}_{25}$ – ${\rm C}_{31}$ n-alkanes). The pristane/nC $_{17}$ and pristane/phytane ratios (table 7, fig. 4) show a good deal of variation over the samples. All chromatograms show compounds additional to n-alkanes in the nC $_{25}$ –C $_{35}$ region. This is strongest in the middle of the sampled interval. These compounds are probably biomarkers (steranes and/or triterpanes) but their distribution is different from what we usually observe and therefore it



sible to identify them without GC-MS analysis. If they are biomarkers this can be interpreted as another indication for the immaturity of the sediments. The "hump" of unresolved compounds as is visible in most chromatograms and appears strongest in A-7174 is indicative for the presence of hydrocarbons from a more mature source.

Aromatic hydrocarbons

The chromatograms of the aromatic fractions (Appendix B) are all similar in that they contain no or very low amounts of alkylnaphthalenes and the dominant identified peaks are phenanthrene and homologues. This pattern is common in type IV or poor type III kerogens, which is in agreement with the Rock-Eval data. Most samples (except A-6805 and A-7174) contain a number of peaks in the area where aromatic steranes and triterpanes can be expected. This is an indication for the immaturity of the samples.

The very large hump of unresolved compounds in sample A-7174 can be interpreted to represent the presence of migrated mature hydrocarbons.



Thermal Evaporation Gas Chromatography

This method produces a gas chromatogram of the free hydrocarbons in the sample that are released during heating at 300°C . The hydrocarbons measured are identical to the S1 peak in Rock-Eval pyrolysis. The method is qualitative, not quantitative. It enables the inspection of $\text{C}_7\text{-C}_{15}$ hydrocarbons that are normally not analysed as analysis of headspace and cuttings gas is up to C_7 and solvent extraction followed by MPLC analyses mainly C_{15+} hydrocarbons. The sensitivity is very high because of the direct coupling between heated sample container and gc column. Hence, it is possible to analyse samples with low hydrocarbon content, e.g. dolomites A-7177 and A-7181.

The gas chromatograms (Appendix C) show that all samples mainly contain short hydrocarbons (\leq nC₁₂), only the chromatogram of A-7174 contains significant compounds of higher n-alkanes. Most samples have a very complex mixture between $n-c_8$ and $n-c_{11}$ consisting predominantly of aromatic hydrocarbons. Samples A-6820 (composite), A-6821 (composite), A-7174 and A-7177 are different in that these samples contain relatively higher amounts of n-alkanes. The reason for the different chromatograms of A-6810, A-6821, A-7174 and A-7177 is not obvious. These samples are not significantly different from the others based on TOC, Rock-Eval or solvent extractable hydrocarbons. The relatively high abundance of aromatics in the samples can be the result of kerogen type and maturity, i.e. the samples are immature to marginally mature and at this level type III/IV kerogen can be expected to produce relatively high amounts of aromatic hydrocarbons. The similarity between the thermally extracted hydrocarbons from the dolomites and the other samples indicate at least partly a non-indigenous nature of these compounds as it is very unlikely that they have been generated in the dolomites (TOC < 0.2%).

Pyrolysis Gas Chromatography

Pyrolysis-gas chromatography analyses the hydrocarbons that are produced by the kerogen upon temperature programmed pyrolysis. The hydrocarbons measured are those that form the S2 peak in Rock-Eval pyrolysis.



Based on the pyrograms (Appendix D) the samples can be divided into two groups:

- 1. Abundant phenol (P), methylnaphthalenes (C1-N) and a series of n-alkanes extenting to n C_{20} (A-6803, A-6805, A-6808, A-6810, A-6821 and A-7174);
- 2. Poor pyrolysis yield, mainly composed of aromatics and n-alkanes lower than nC_{13} (A-6825, A-6827, A-7177 and A-7181).

Both groups can be characterized as poor type JIJ or type JV kerogens because of the presence of phenol (indicative for terrestrial material) abundant aromatic compounds and a series of n-alkanes with low intensity instead of a dominant series of n-alkanes/n-alkanes which would be indicative for hydrogen richer type JJ and mixed type JJ/JJJ kerogens.

The very poor character of the two dolomite pyrograms (A-7177 and A-7181) is not surprising as these samples were very low in TOC (< 0.2%). The poor pyrograms of samples A-6825 (composite) and A-6827 (composite) are somewhat surprising as these samples are not very different from the other claystones based on TOC and Rock-Eval data.



CONCLUSION .

The results from the follow-up analyses on eight claystones and two dolomites from well 6609/7-1 are in good correspondence with the screening analyses (fig. 5).

Pyrolysis-gas chromatography (Appendix D) confirms the kerogen typing based on Rock-Eval data. The entire interval contains a poor type JJI or type JV kerogen, giving it a poor to fair potential as source rock for gas only.

The presence of terrestrial material (type JJI) can also be recognized from the distributions of the solvent-extracted saturated and aromatic hydrocarbons (Appendix A and B).

Both thermal and solvent extracted hydrocarbons suggest the presence of non-indigenous hydrocarbons (migrated and/or contamination) in the sediments. These indications are strongest for A-7174 (1860-1870m). This sample lies within the sandstone containing zone where high amounts of petrogenic gases were detected in the screening analyses, which could explain the presence of migrated hydrocarbons in the sediment.

The various maturity indicators (vitrinite reflectance, fluorescence colour, Rock-Eval $T_{\rm max}$, composition of saturated hydrocarbons) do not show a consistent change in the sampled interval. The sediments are immature to marginally mature.



<u>Table 1:</u> Samples selected for follow-up analyses (data from screening report).

<u>Sample</u>	Depth (m)	Zone	TOC (%)	<u>Lithology</u>
A-6803	1510-1520	А	1.17	Claystone, light grey
A-6805	1530-1540	Α	0.76	Claystone, different shades of
A-6808	1560-1570	Α	1.02	grey Claystone, different shades of
				grey
A-6810	1580-1590	В	1.14	Claystone, olive grey - grey
A-6821	1690-1700	С	0.76	Claystone, grey - olive grey
A-6825	1730-1740	С	1.93	Claystone, light green
A-6827	1750-1760	С	1.99	Claystone, light green
A-7174	1860-1870	С	0.87	Claystone, grey
A-7177	1890-1900	D	n.d.	Dolomite
A-7181	1930-1940	D	0.19	Dolomite

<u>Table 2</u>: Rock-Eval pyrolysis data of selected samples (from screening report).

Sample	e <u>Depth (m)</u>	<u>TOC (%)</u>	<u>\$1</u>	<u>S2</u>	<u>\$3</u>	<u>H.J.</u>	0.1.	Petr. pot. <u>S1+S2</u>	Prod. Index <u>\$1/\$1+\$2</u>	Temp. max. <u>(^OC)</u>	
A-6803	3 1510-1520	1.17	0.17	0.97	0.53	83	45	1.14	0.15	412	
A-6805	5 1530-1540	0.76	0.10	0.57	0.45	75	59	0.67	0.15	413	
A-6808	3 1560-1570	1.02	0.48	0.62	0.74	61	73	1.10	0.44	426	
A-6810	1580-1590	1.14	0.18	0.98	0.70	86	61	1.16	0.16	420	
A-6821	1690-1700	0.76	0.29	0.11	0.79	14	104	0.40	0.72	339	
A-6825	5 1730-1740	1.93	0.38	1.32	0.84	68	44	1.70	0.22	419	
A-6827	7 1750-1760	1.99	0.25	0.77	0.98	39	49	1.02	0.25	421	
A-7174	1560-1570	0.87	0.09	0.17	0.25	20	29	0.26	0.35	422	



TABLE: 3.

CONCENTRATION OF EOM AND CHROMATOGRAPHIC FRACTIONS

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Ţ	A 6808	:	1570	:	9.7	:	2.6	: 0.8	. :	1.1	:	1.9	:	0.7	:	0.83	I
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ī	A 6809	•	1590	:	9.4	:	2.7	: 1.7		1.0	:	2.7	:	0.0	:	1.11	I
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TABLE: 4.

WEIGHT OF EOM AND CHROMATOGRAPHIC FRACTIONS (Weight ppm OF rock)

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I I	IKU-No	:	DEPTH	:	EOM	:	Sat.	:	Aro.	:	HC	:	Non HC	I I I
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I	A 6805	1	1540	:	138	4	60	:	53	1	113	1	25	Ī
I I	A 6808	:	1570	:	269	:	87	1	112	: :	199	:	70	I
I I	A 6809	:	1590	:	287	:	181	:	106	:	287	:	o	i I
I I	-10 A 6820	:	1710	:	365	:	i 40	:	120	:	259	:	106	I I
I	-22 A 7174	:	18 7 0	:	745	:	280	:	298	:	578	# #	166	I
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TABLE: 5.

CONCENTRATION OF EOM AND CHROMATOGRAPHIC FRACTIONS (mg/g TOC)

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Ţ	IKU-No	: DEPTH	: 8	: : MOE	Sat.	: Aro	. : HC	: Non : HC	I
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I		:	:	:		:	:	*	I
I	A 6808	: 1570 :	: 3	32.4 :	10.5	: 13.5	5 : 23.9 :	: 8.5 :	T T
Ī	A 6809	: 1590	: 2	25.9 :	16.3	9.	6 : 25.9	. 0.0	Ī
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I	A 7174	: 1870	: 6	54.7	24.4	: 25.	9 : 50.3	: 14.5	I
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DATE : 22 - 11 - 83.



TABLE: 6.

COMPOSITION IN % OF MATERIAL EXTRACTED FROM THE ROCK

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Ι	A 6805	:	1540	1	43.6	: 38.2	;	81.8	#	114.3	18.2	450.0	I
I		:		:		=	:		:	:	:	j	Ī
I	A 6808	4	1570	:	32.3	41.5	•	73.8	:	77.8	26.2	282.4	I
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I	A 6809	:	1590	=	63.0	: 37.0	:	100.0	:	170.0	0.0	*****	I
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I	A 6820	:	1710	:	38.2	: 32.7	2	70.9	4	116.7 :	29.1	243.7]	I
I	-22	:		:		:	:		:	:	;	:]	Ι
I	A 7174	:	1870	:	37.6	: 40.0	:	77.6	:	94.1:	22.4	347.4	Ι
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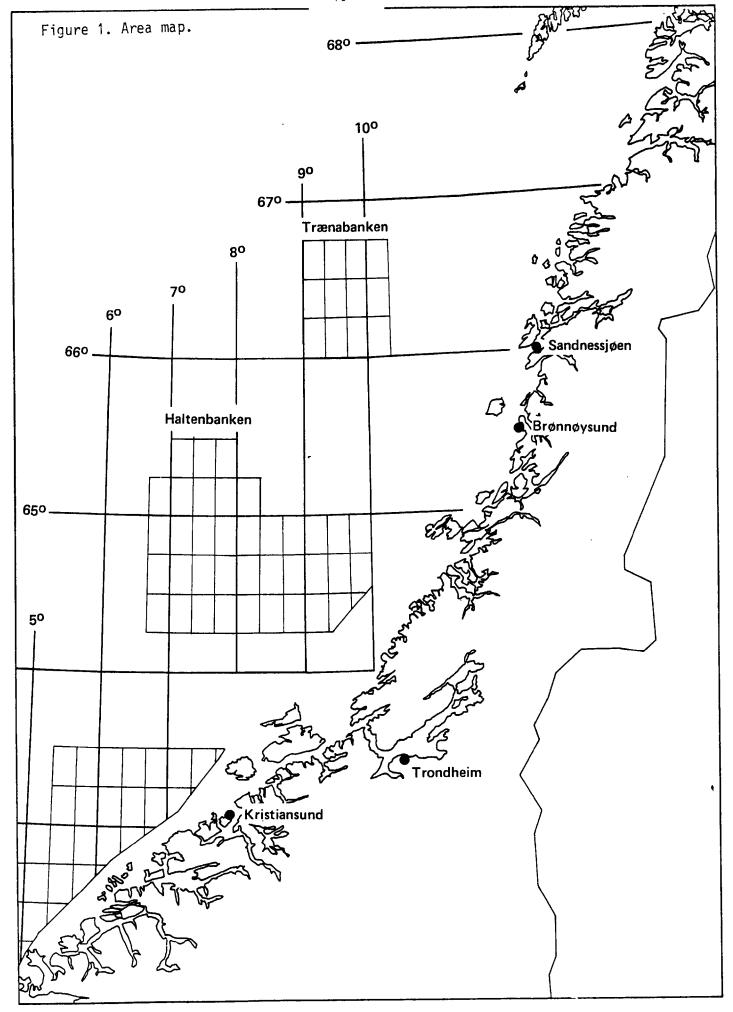


TABLE 7.

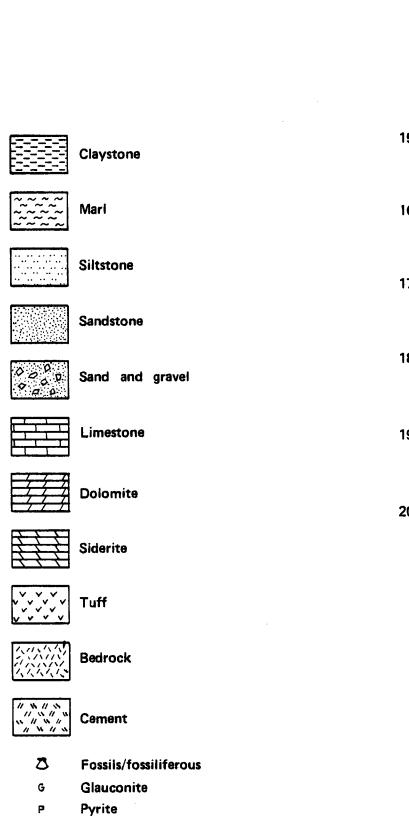
TABULATION OF DATA FROM THE GASCHROMATOGRAMS

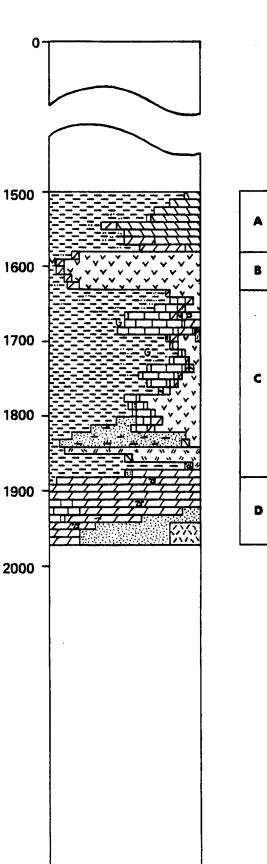
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6609/7 - 1 Phillips Petroleum

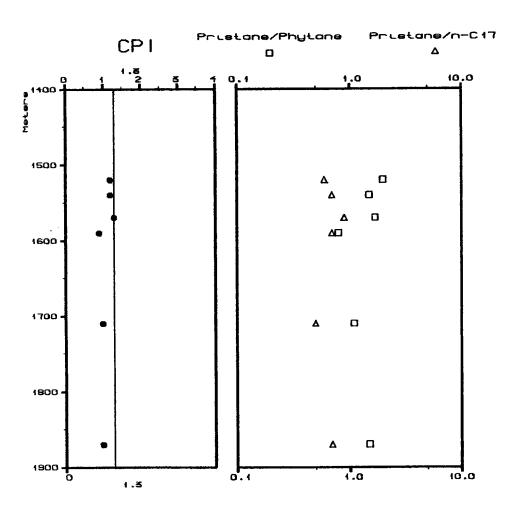




· 21

Figure 4. C_{15}^+ saturated hydrocarbons.

WELL : 6609/7-1



INTERPRETATION DIAGRAM

Figure 5. Interpretation diagram.

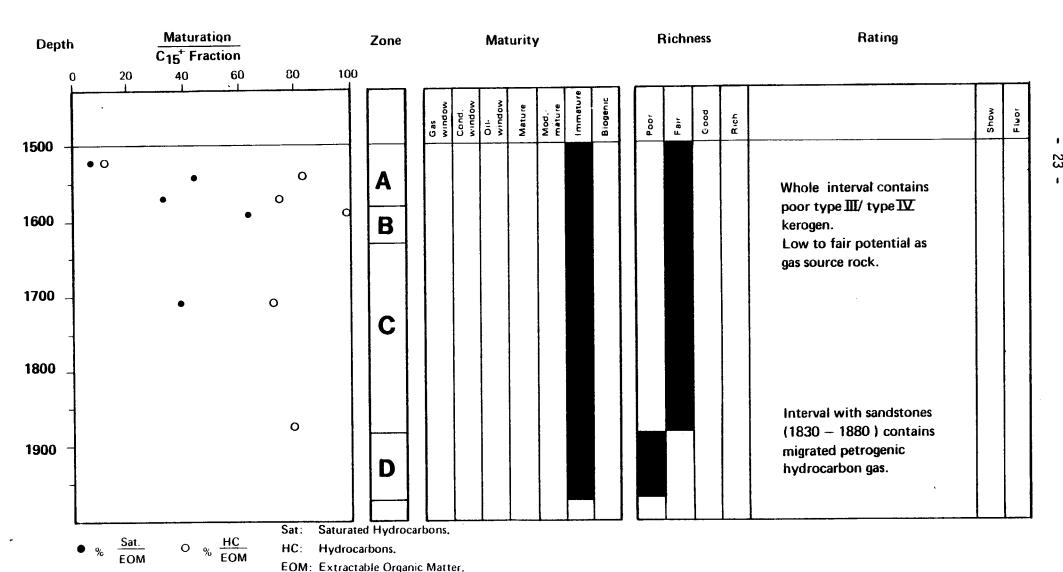
Well no.:

6609/7 - 1

Company:

Phillips

SUMMARY OF SOURCE POTENTIAL





APPENDIX A

Gas Chromatograms of Saturated Fraction

 $a = nC_{17}$ alkane

b = pristane

c = phytane

v,w,x,y,z = unidentified compounds, possibly steranes and/or triterpanes

100 . ₩ 00 Saturated hydrocarbons 90 -A - 6803 1510 - 1520m : :: Вох 80 signal nC₁₅ 70 Injection #: 1 Maximum nC_{20} nC₂₅ 60 50 C 40 Sample 140,0MB 08:33 on 11/Nov/83 Analysis :050140H6B03S Sample Name :8-6803, S, PLOT-CHANNEL 20 Printed at кям рятя երարարարարարարարարարարարարություն արարարարարարարարարարարարություն արարարարարարար

2.24 Saturated hydrocarbons ÷ 90 A - 6805 1530 - 1540m : :: 80 signal 70 Injection #: 1 Maximum b 50 nC₂₀ nC₂₅ # 40 Analysis :050140A6805S Sample Sample Name :R-6805,SAT,OB on 09/Nov/83 30 -PLOT-CHANNEL 20 -13:01 10 -RAW DATA Printed , արտարավատիավատիավարիավարիավարիակարիավարիակարիավարիականակարի հարարակարիականության և բ 73 13

100 🗇 W 16.73 Saturated hydrocarbons 90 -A - 6808 1560 — 1570m : :: Вох 80 signal 70 Injection #: | Maximum 60 50 -40 is: 050140A6808S Sample Name: R-6808, SAT, DMB Printed at 14:42 on 09/Nov/83 30 PLOT-CHANNEL 20 nC₂₅ nC₁₅ DATA Analysi Sample 73 **76** 79 31 19 22 25 R E E 10

100 Saturated hydrocarbons 90 A - 6821 composite 1680 - 1710m : :: 80 signal nC₃₀ Injection #: 1 Maximum 60 b 50 nC₁₅ nC₂₀ 40 Sample 140,0MB 12:41 on 10/Nov/83 :050140R6822S me :R-6822,S, PLOT-CHRNNEL квы ратв Analysi Sample 55

100 27.50 90 Saturated hydrocarbons : ? Вох A - 7174 nC₁₅ 1860 - 1870m 80 -Injection #: 1 Maximum signal nC_{20} 70 С 60 nC₂₅ nC₃₀ 40 74S Sample ,S,140,0MB Printed at 14:21 on 10/Nov/83 PLOT-CHANNEL 20 s:050140B71 Name:8-7174 кам рятя Analysi Sampie 55 52 16



APPENDIX B

Gas Chromatograms of Aromatic Fraction

A = methyl-naphthalenes

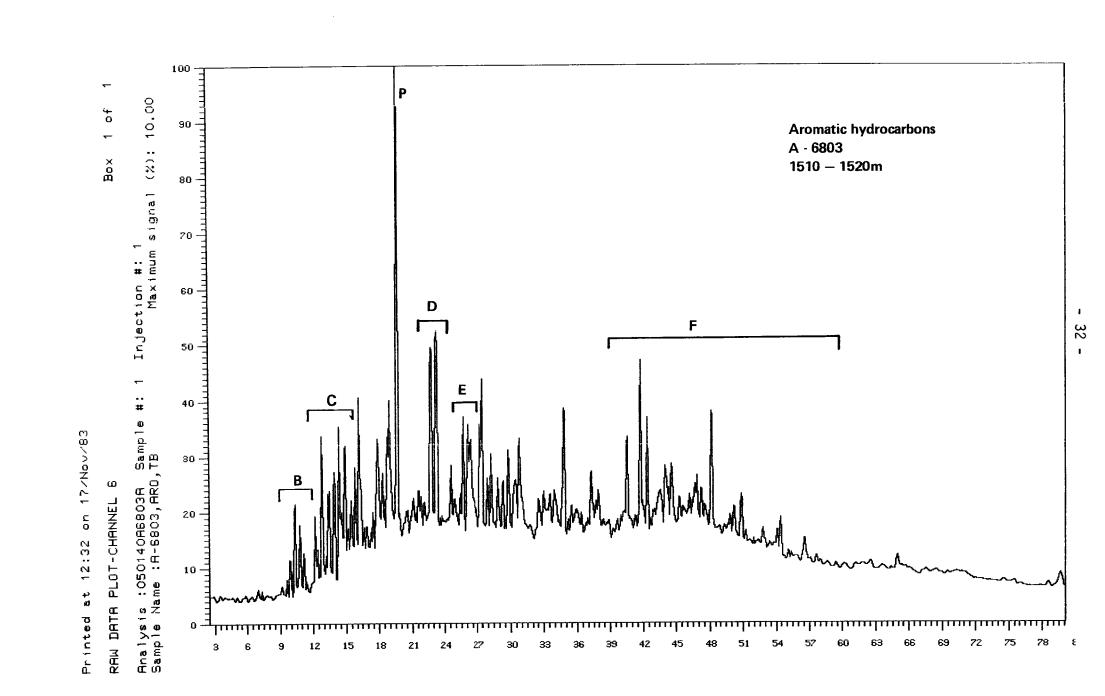
 $B = C_2$ -naphthalenes $C = C_3$ -napthalenes

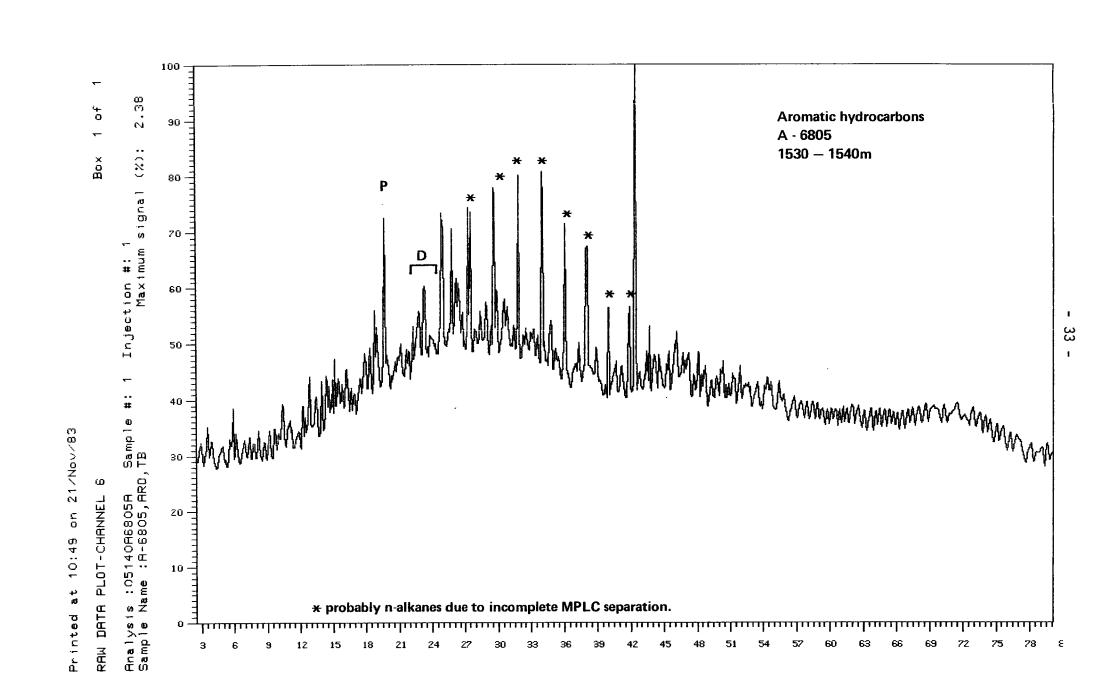
P = phenanthrene

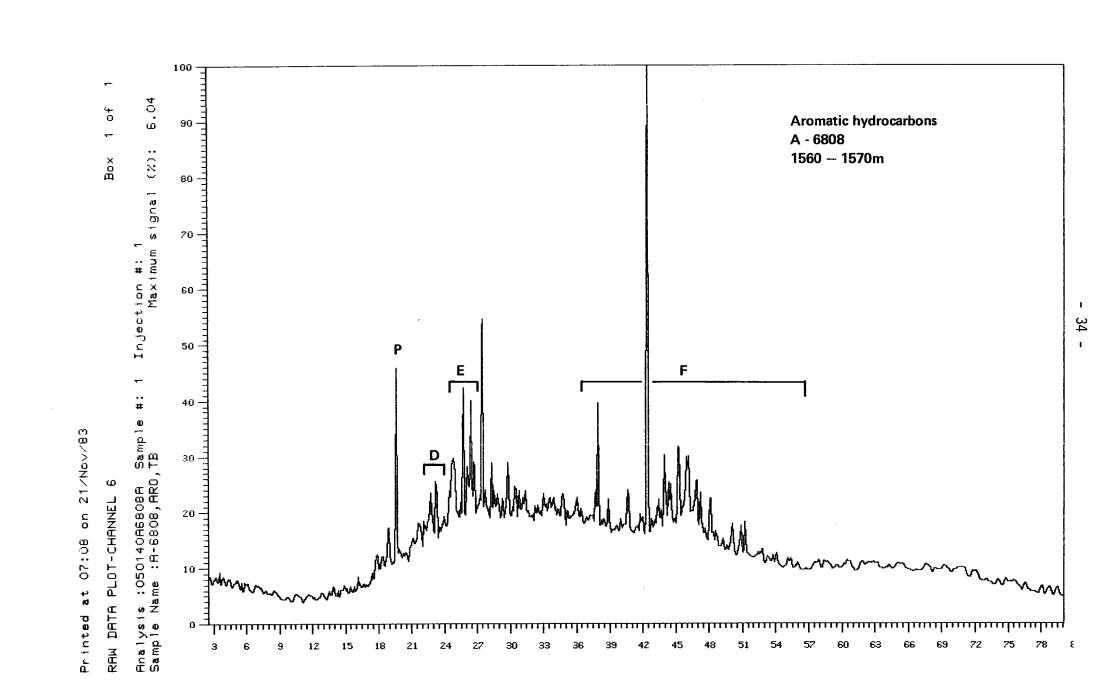
D = methyl-phenanthrenes

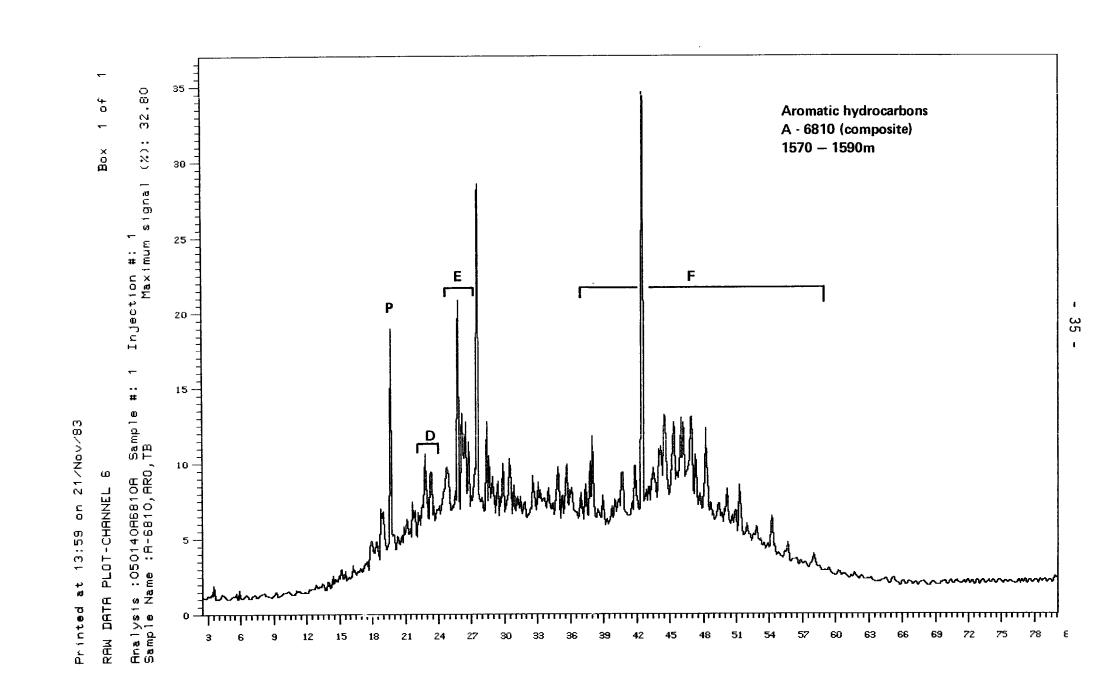
 $E = C_2$ -phenanthrenes

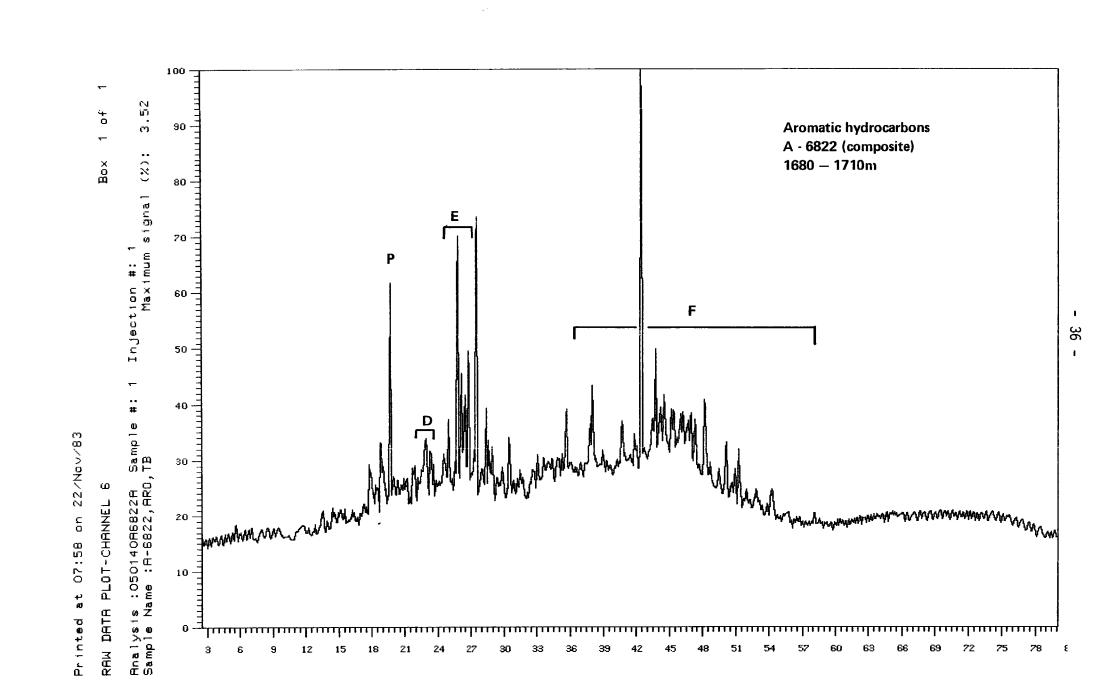
F = area with aromatised steranes and triterpanes

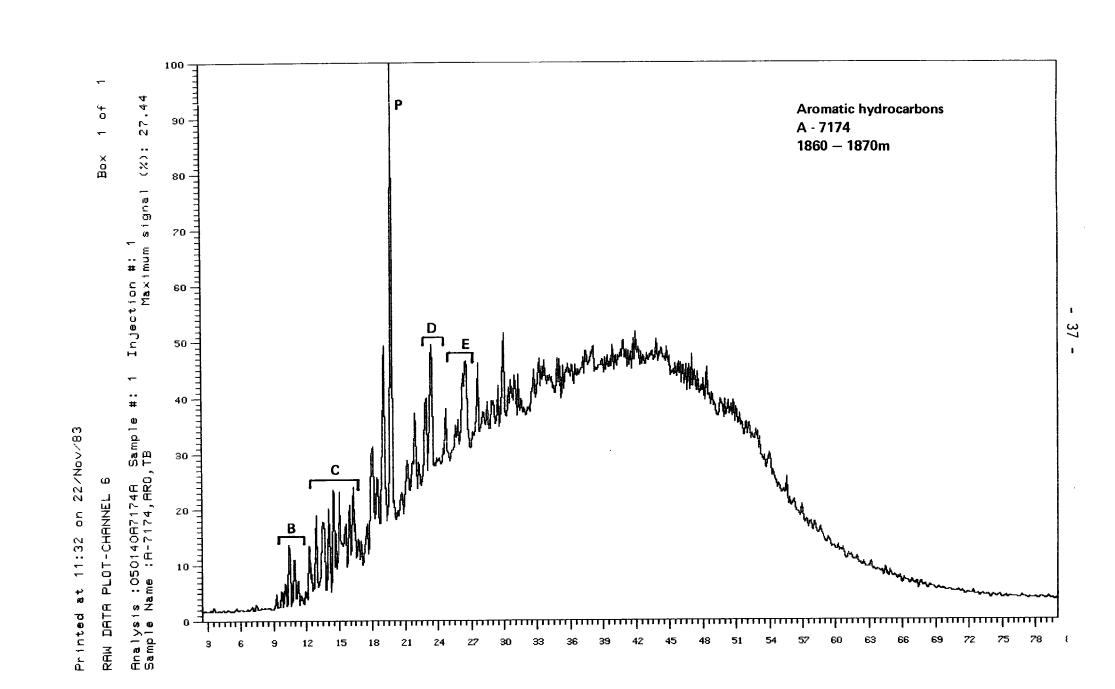














APPENDIX C

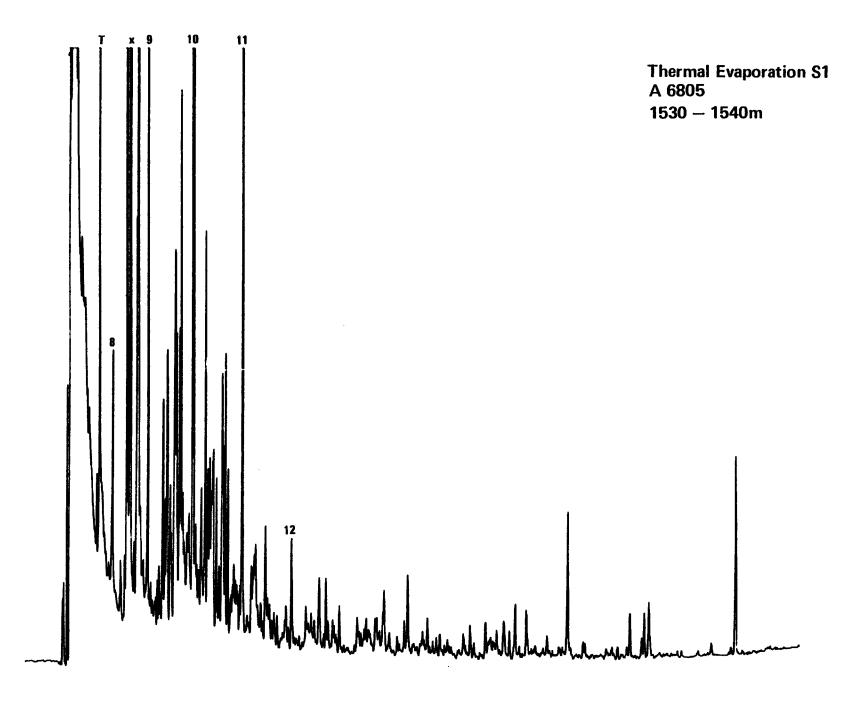
Thermal Evaporation GC (S1 peak) key to the chromatograms

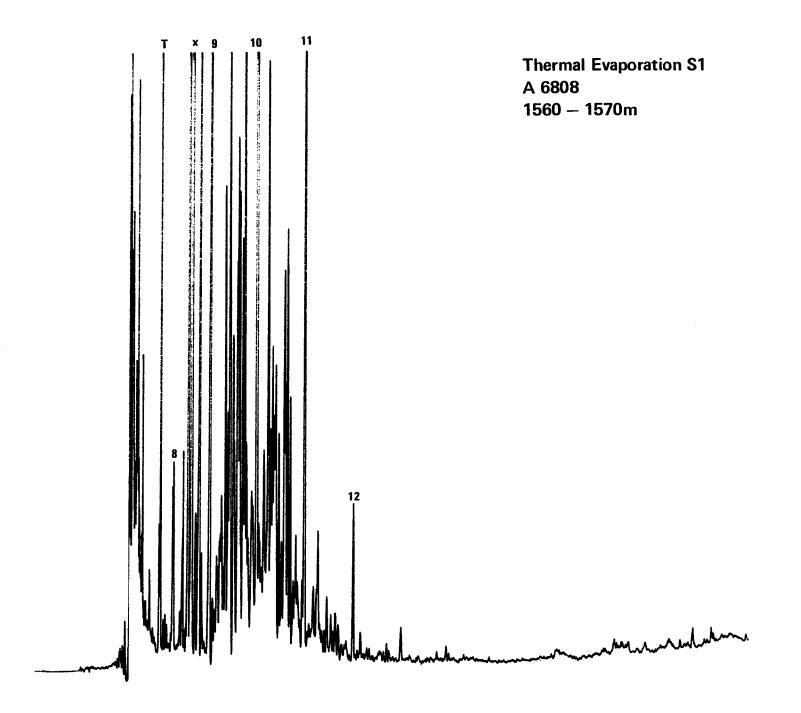
numbered peaks are n-alkanes

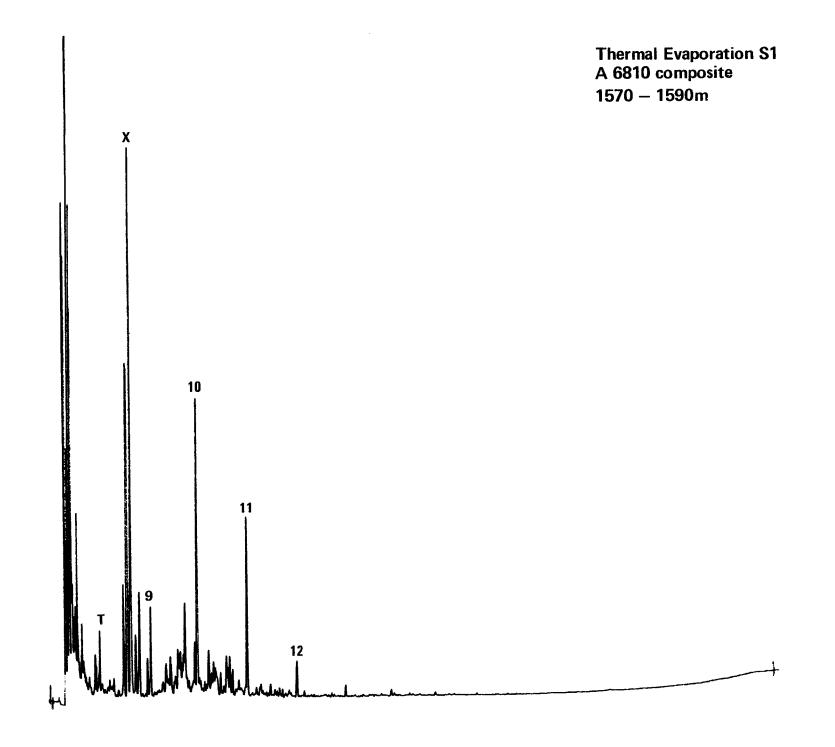
T = toluene

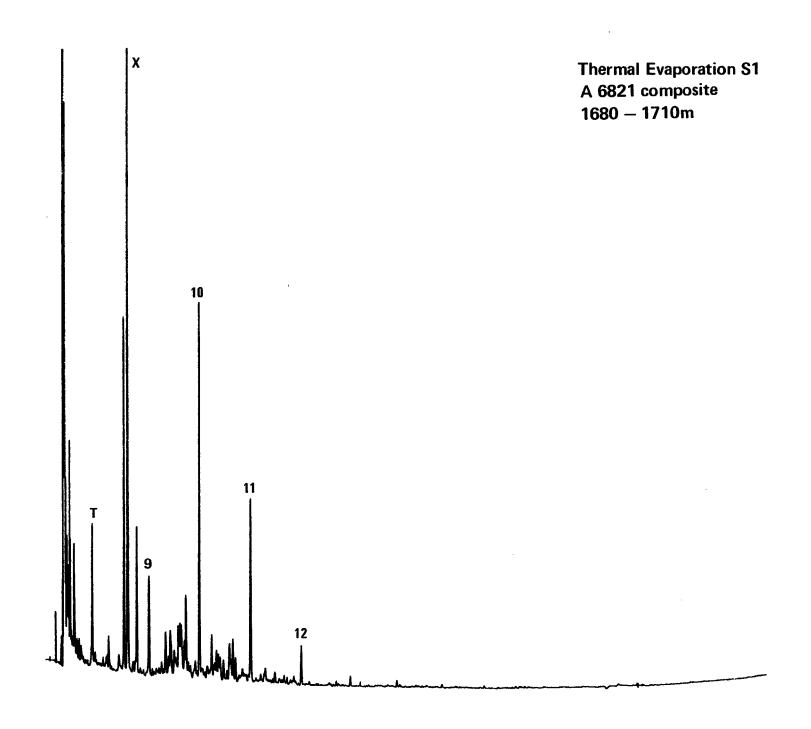
X = xylenes (m+p)

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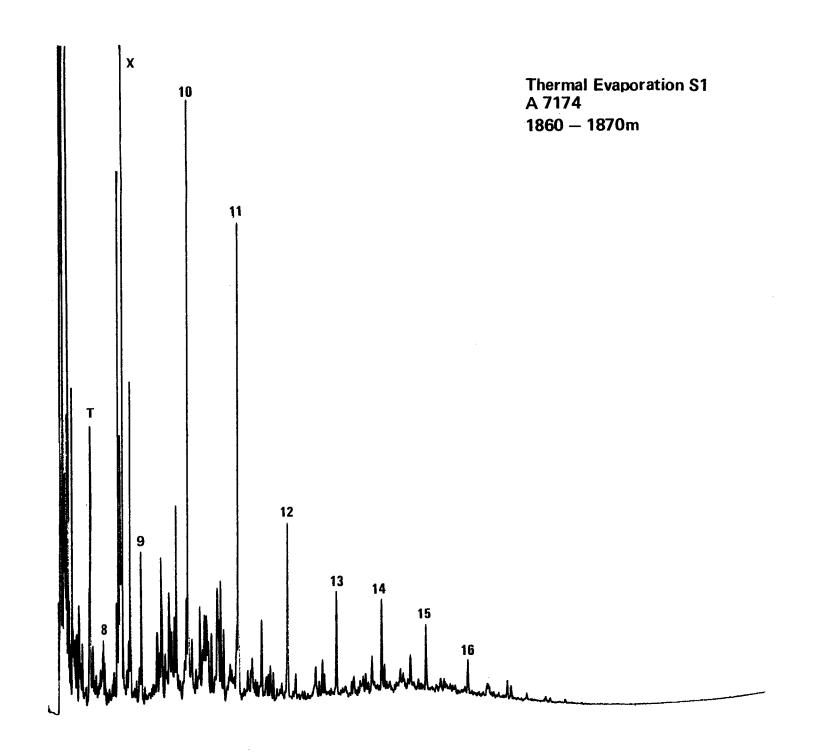


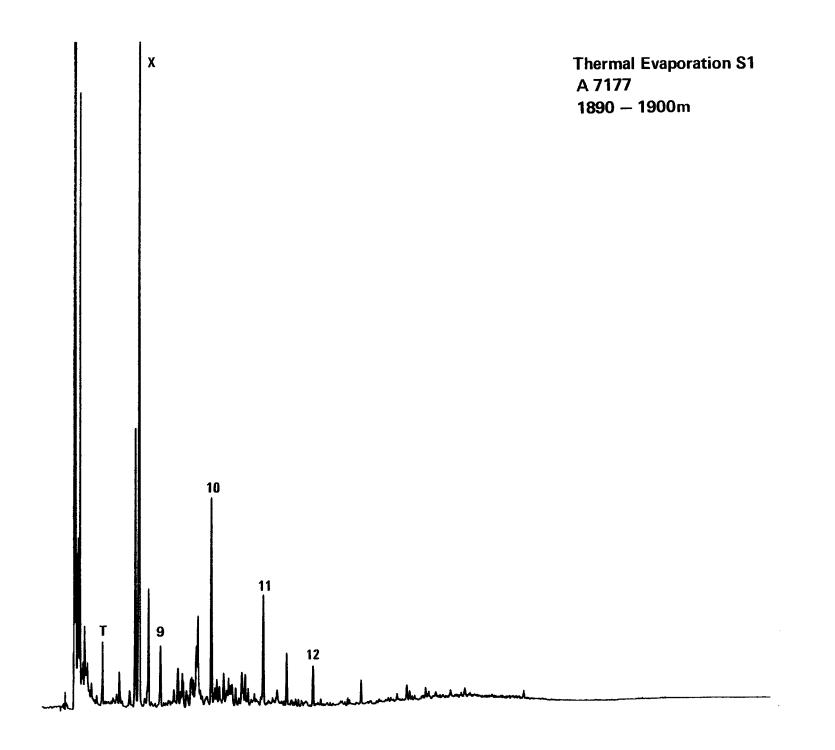


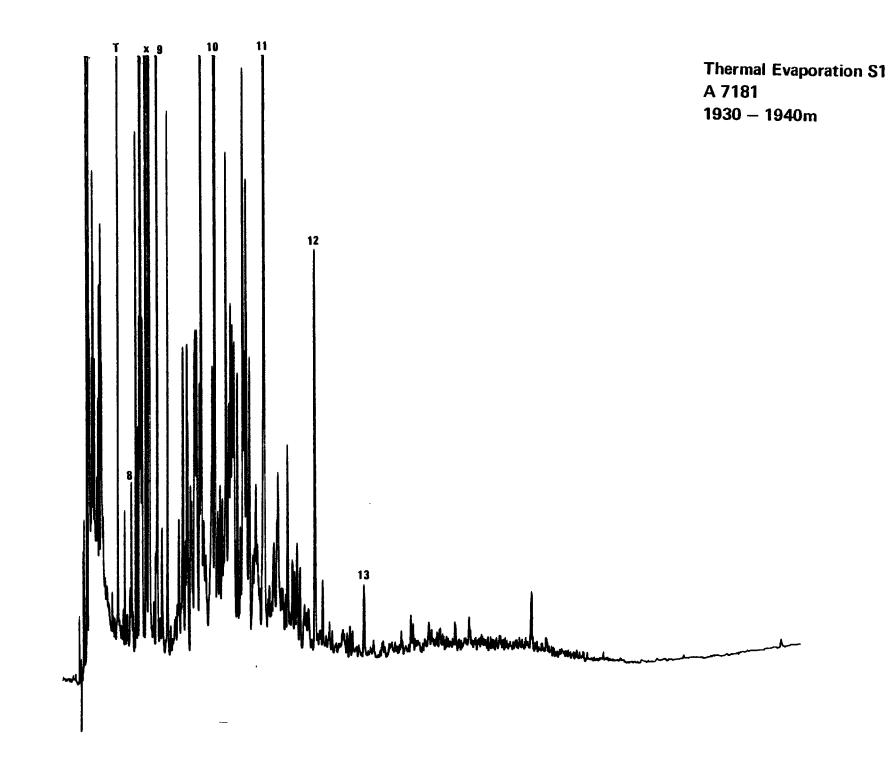


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APPENDIX D

Pyrolysis GC (S2 peak)

Key to the chromatograms:

numbers indicate n-alkene/n-alkane doublets with the n-alkene eluting first

T = toluene

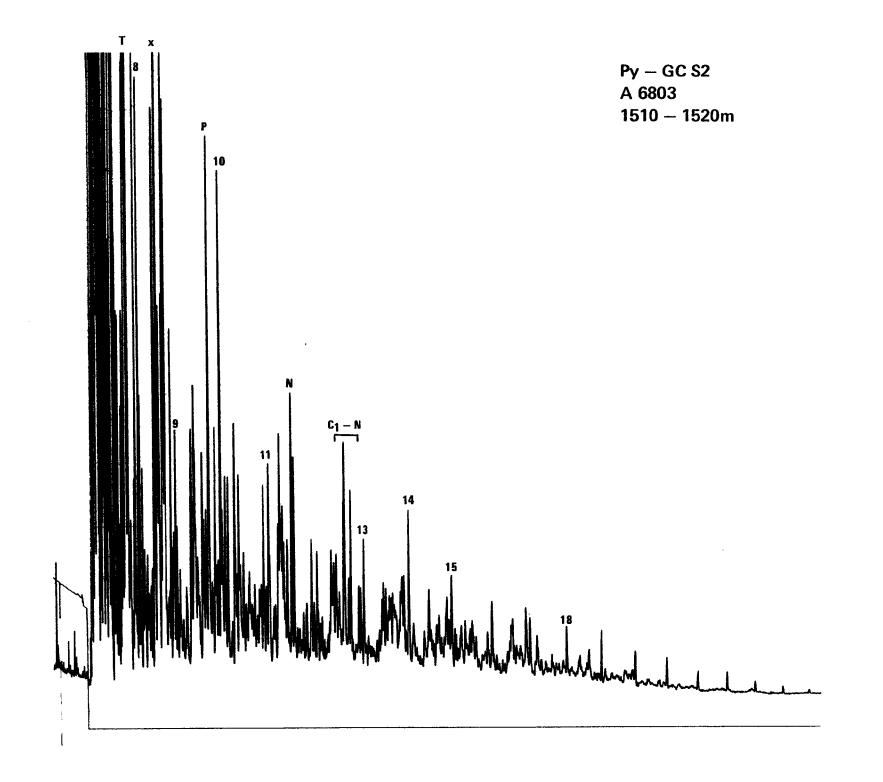
X = xylene

P = phenol

N = naphthalene

C1-N = methyl naphthalenes

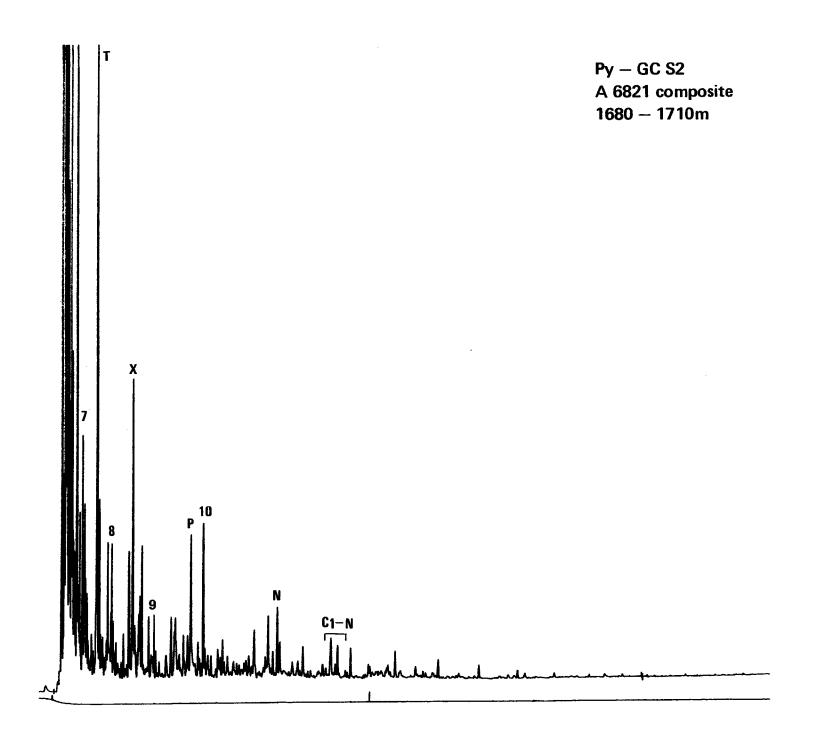
PR = prist-1-ene

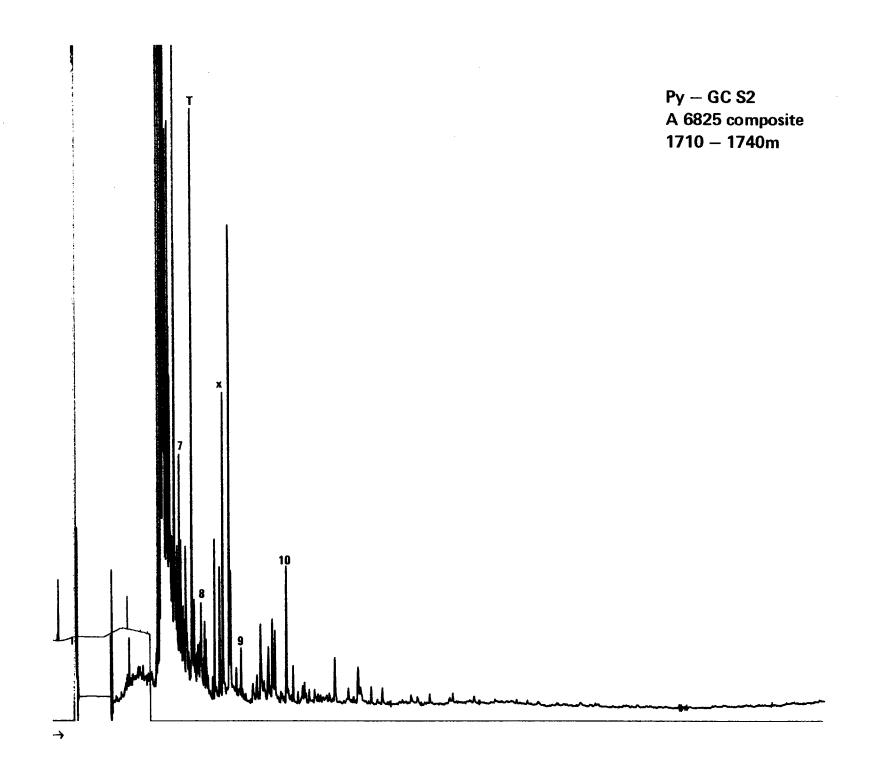


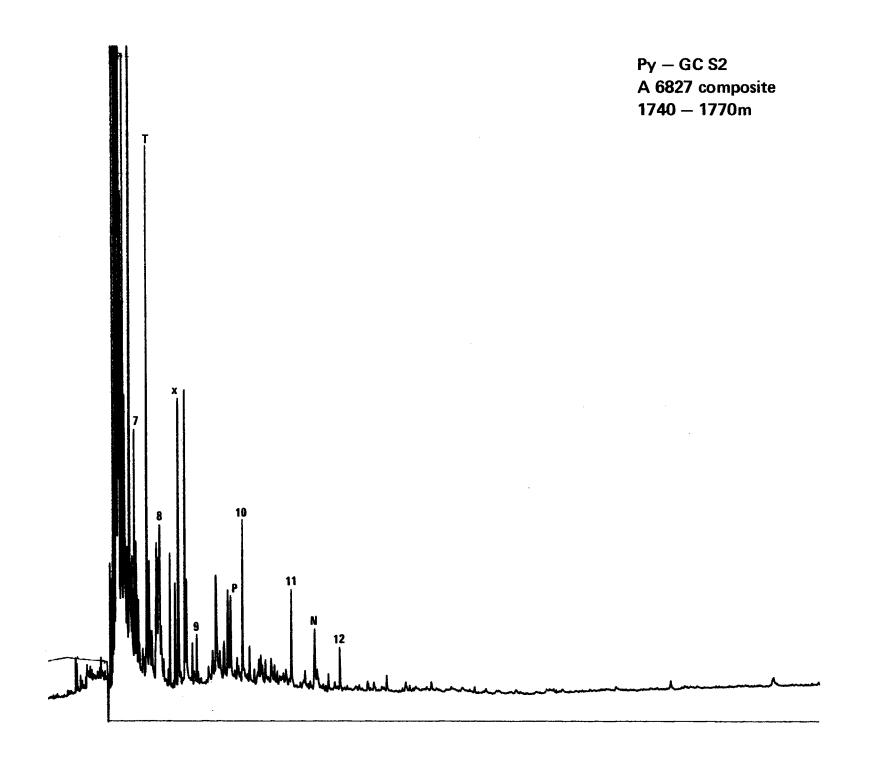
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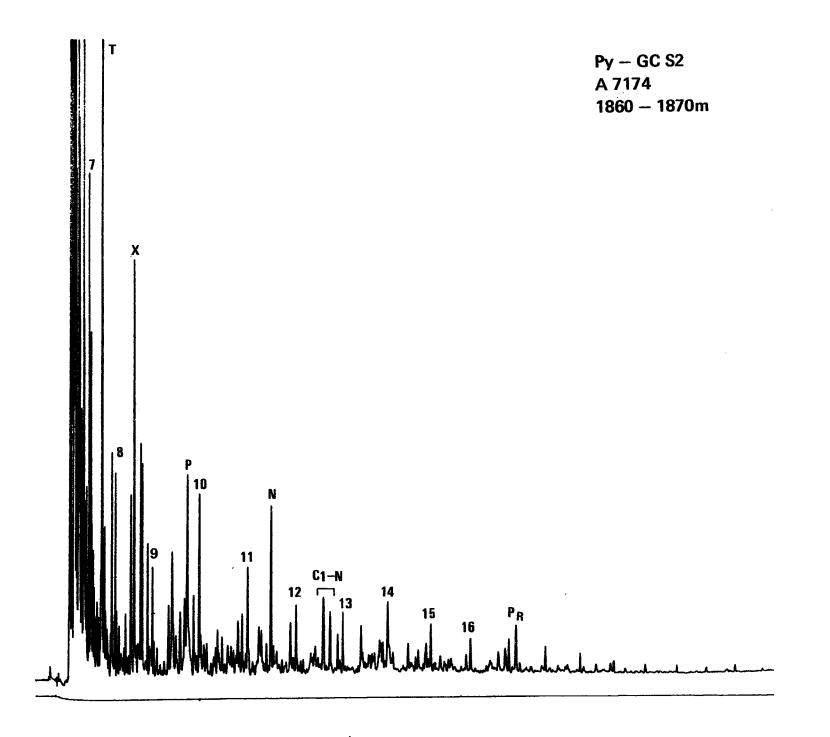
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. 53









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