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REPORT ON AN OIL TO SOURCE ROCK CORRELATION CARRIED OUT ON SAMPLES OF OIL AND SHALES FROM THE 15/9 - 1 WELL, NORWEGIAN NORTH SEA.

by

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SUMMARY

A study of the source rocks, maturation and produced oil of the section 3,349 to 3,734 metres of the 15/9-1 well, Norwegian North Sea shows that an extensive thickness of some 250 metres of very good oil source rock is present. The source rocks are only just mature and analysis shows that in their present state of maturity they could not have generated the produced oil. However, it has been possible to show that the section from 3,505 to 3,586 metres has a similar set of characteristics to the oil and could have sourced the oil offstructure at a depth of about 4,000 metres.

I

INTRODUCTION

Ditch cuttings and core pieces from the Norwegian North Sea 15/9-1 well have been sampled between depths of 3,349 and 3,672 metres and analysed for their state of maturity and source rock potential. Analyses have been carried out on two samples of oil produced from this well for the purpose of comparison with likely source rocks.

Estimations of maturity are based on measurements of spore colouration and vitrinite reflectivity. Source rock potential has been determined by solvent extraction and column chromatographic fractionation into alkane, aromatic and asphaltene with resene fractions. Gas chromatography of the alkane fractions from both the extracts and oils, has been carried out in order to determine similarities in the distribution of the hydrocarbon components.

The samples were received as ditch cuttings for the interval 3,349 to 3,734 metres and core pieces were made available for 3,523 to 3,672 metres. The ages of the samples are U. Jurassic to L. Cretaceous, and the samples were small but of good quality for analysis.

RESULTS AND INTERPRETATION

A. LITHOLOGIES

The analysed sequence of samples ranges from L. Cretaceous to U. Jurassic in age, with minor chalk caving. For the most part, dark grey shales dominate the analysed sequence from 3,349 to 3,612 metres with subordinate reddish shales occurring between 3,349 and 3,415 metres and between 3,550 and 3,559 metres. Minor sandstone is present between 3,487 and 3,496 metres and between 3,550 and 3,559 metres. From 3,612 to 3,734 metres, sandstones, usually oil stained, are predominant, with intercalations of silty shales, siltstones and coals.

B. MATURATION STATE OF SOURCE ROCKS

1. Spore Colouration (Table 1, Figure 1)

Five samples from 3,349-52, 3,433, 3,487, 3,580.3, and 3,671.85 metres were prepared by normal palynological methods to give preparations of unsieved, unoxidised kerogen mounted on normal palynological slides.

The level of maturity of oil-prone organic matter in the analysed sediments has been assessed by a visual examination of the indigenous sporomorphs. The colouration of spores, with increasing thermal maturity, changes from pale yellow, through orange and brown, to black; spore colour indices used in this report are based on a scale of 1 to 10, with values of 3.0 to 3.5 representing the narrow zone of transition between immature and mature sediments.

The three uppermost samples yielded adequate assemblages of sporomorphs, mostly pale yellow in colour with spore colour indices between 3 and 5, averaging 3.5 and 4. Sporomorphs were less frequent in sample 3,580.3 metres but gave a 4.5 index value, and were absent in sample 3,671.85 metres.

The values obtained for the spore colouration indices suggest that the sediments are at an early stage of maturity. However, it was noted that the

shades of yellow seen were not exactly typical and some bleaching by natural processes might have occurred. Orange fluorescing spores seen in polished coal mounts suggest that indices of 5-6 are reached in the deepest parts of the analysed section.

Sapropel was the dominant component of the kerogens in all but the deepest sample at 3,671.85 metres, which contained mostly inertinite.

2. Vitrinite Reflectivity (Table 1 Figure 2)

The examination of vitrinite particles in shales is now a frequently used extension of coal rank studies. Vitrinite is not an important oil source and its maturation, unlike oil-prone organic matter, is strongly dependent on length of time of heating; however, it is the only reliable maturation indicator in the organically metamorphosed zone. Humic, gas-prone organic matter is considered to be transitionally mature over the reflectivity range 0.4% to 0.5%, but would not be expected to yield prolific gas until levels in excess of 0.8% to 1.0% have been reached. In an early Tertiary sequence, oil-prone organic matter would be expected to generate hydrocarbons at vitrinite reflectivities of 0.4% and above.

Vitrinite reflectivity values have been determined for seven samples including two coals. In the grey shales vitrinitic particles are generally small and rare, but semifusinite and fusinite are common. In coals vitrinite is common but shows widely varying composition and reflectivity. The vitrinitic material in the shales appeared as two populations with low reflecting vitrinite usually more frequent; this commonly occurs in bituminous shales and oil shales. Measurements based on the higher reflecting vitrinite gave values between 0.48% and 0.55%. The coals gave higher values of 0.72%.

Examination of the cuttings as polished mounts in blue light showed that dispersed sapropel was abundantly present giving rise to a general yellowish fluorescent background. Algal cell walls, brightly fluorescing yellow, were also frequently seen.

3. State of Maturation

Both of the maturation parameters indicate a rapid change in thermal maturity at the base of the grey shale sequence before the coals are encountered. Although a normal fault with a throw of about 300 metres could account for this abrupt increase in maturation, it seems more likely that the change is brought about by a rapid increase in palaeotemperature frequently associated with overpressured sections.

The maturation parameters agree in indicating that the analysed section is in the early stages of maturity within which sapropelic kerogen will generate heavy oil with gas and condensate.

C. SOURCE ROCK EVALUATION (Tables 2 and 3)

Thirty six cuttings samples have been analysed for organic carbon content through the grey shale sequence. Organic carbon contents greater than 3% were usual between 3,376 and 3,625 metres. On the basis of the organic carbon contents and availability of samples, eight composites of cuttings were made up and submitted for source rock analysis by solvent extraction and chromatographic fractionation of the extracts. Organic carbon contents of the extracted shales were also determined and were adjusted in relation to the extractable organic matter, to give the organic carbon contents of the shales when fresh; these values are entered in Table 3.

The amounts of extractable organic matter ranged between 0.19 and 1.01% of the shale samples and values relative to the organic carbon content of 8.4 to 15.2%. However the hydrocarbons contents, although in appreciable amounts usually between 300 and 1,050 ppm, made up only relatively small (4-15%) proportions of the extracts. The alkanes were also present in relatively low contents, averaging 60% of the total hydrocarbons.

The analytical results suggest that these source rocks are capable of sourcing prolific amounts of oil at optimum maturity. At their present level of maturity they can yield lesser amounts of heavy oil, condensate and wet gas.

These results are confirmed by microscopic examination of the kerogens from spot samples which show a preponderance of sapropel, with inertinite usually the subordinate component. Even within polished mounted chips the dispersed sapropel gives rise to a general yellow/orange fluorescence in blue light and the remains of algal cell walls are frequently present fluorescing a bright yellow.

D. COLUMN CHROMATOGRAPHIC ANALYSES OF OILS (Table 4)

Two samples of oil were received, one as produced marked "Bubble Hose" and the second which had been heated to 60°C (topped) to remove gases and light vapours. An A.P.I. gravity of 26° for the "topped" oil has been quoted and its associated gas contains 4.8% carbon dioxide. It is noted that the oil as produced has a high GOR of 900.

The oils were fractionated through silica gel filled chromatography columns using pentane, 25% toluene in pentane, and methanol as eluants to yield alkane, aromatic and resene fractions. The asphaltene content was calculated by difference and was close to the value obtained by pentane precipitation. The results show that asphaltenes with resenes make up a substantial proportion (20%) of the untreated oil The increase change in concentration of this fraction to 40% by removal of gas and light vapours ("topped oil) indicates that this latter fraction, composed of gases and the light petroleum fraction, itself contributes approximately half of the untreated oil. The analyses suggest that the oil could be rich in both light and heavy ends.

E. GAS CHROMATOGRAPHY OF SOURCE ROCK AND OIL ALKANES (Table 5, Figures 3a-i)

Gas chromatographic analysis of alkane fractions was carried out on a 20 metre WCOT capillary column with OV1 as stationary phase programmed from 60° C to 260° C at 5°/min.

Analysis of the "bubble hose" oil alkanes shows a distribution of n-alkanes with carbon numbers up to C_{30} with a maximum at C_{22}. Pristane and phytane are sub dominant and a multiplicity of peaks in the C_{25} to C_{30} region indicates

the presence at a low level of polycyclic steranes and triterpanes. A slight tendency to a predominance of even carbon numbered alkanes is seen from C_{20} to C_{26}, a feature of rich algal deposits.

Analysis of the source rock alkanes was more difficult. Amounts of extracted hydrocarbons were rather low and they produced a low signal to weight ratio indicating that the range of carbon numbers present extended beyond C_{35}, the limit for gas chromatographic analyses. This is a feature of some rich source rocks when just mature. In general a range of n-alkanes from C_{15} to C_{33} is seen on the gas chromatograms with a maximum usually at C_{20} but the range of alkanes up to C_{28} are frequently present in almost similar amounts. Pristane and phytane vary in concentration and are present in concentrations similar to C_{17} and C_{18} with pristane/n-C_{17} and phytane/n-C_{18} ratios averaging respectively 1.00 and 0.84. Predominance of either odd or even numbered n-alkanes is never marked in the analyses but C_{26} and C_{29} are usually prominent. Steranes and triterpanes in the C_{25} to C_{32} range are ubiquitously present but at low concentrations. Close examination of the twenty five or so components in this assemblage of polycyclic alkanes shows that each sample has different proportions of some of the members.

F. COMPARISON OF THE OIL WITH THE POTENTIAL SOURCE ROCKS

The analysis of the oil shows that it is a heavy crude, API 26°, associated with a major proportion of gas and condensate. Gas chromatographic evidence also shows that its range of alkanes extends well into the wax fraction. These data show that the oil originated at an early stage of maturity.

Both the oil and the source rock extracts contain 60% alkanes in the hydrocarbon fractions, but the alkanes of the oil when analysed by gas chromatography show only minor points of similarity with those of the source rocks. The main points of dissimilarity are that the pristane/n-C_{17} and phytane/n-C_{18} ratios are much lower for the oil than the source rocks. On the other hand both the source rocks and the oil contain appreciable quantities of n-alkanes in the C_{20} to C_{30} range and appreciable quantities of polycyclics,

steranes and triterpanes.

The conclusion to this point is that some of the source rocks could have generated the oil if they are sufficiently mature off-structure. To be more precise they would have to reach a spore colour index between 5.5 to 6.0 to produce an oil with the characteristics given. Some of the source rocks could not have contributed much to the oil, in particular those with prominent $\underline{n}\text{-}C_{27}$, $\underline{n}\text{-}C_{29}$ and $\underline{n}\text{-}C_{24}$ peaks.

To find a closer correlation, it is necessary to examine on the gas chromatograms the polycyclic alkanes. The polycyclic alkanes, steranes and triterpanes are a fingerprint of the source rock since, although they decrease in concentration as maturation progresses, their concentrations relative to each other remain constant. For this study a visual comparison has been made of the relative heights of the minor peaks in the sterane-triterpane region; that is those which occur between $n\text{-}C_{26}$ and $n\text{-}C_{32}$. Correlations have been judged to be good, fair and poor as follows.

depth (metres)	correlation of polycyclics	negative features
3376-379	fair	prominent $\underline{n}\text{-}C_{24}$
3415-433	poor	
3442-460	fair	prominent $\underline{n}\text{-}C_{27}$, $n\text{-}C_{29}$
3469-496	poor	prominent $\underline{n}\text{-}C_{27}$, $n\text{-}C_{29}$
3505-514	good	
3523-550	fair	
3559-586	fair	
3595-625	poor	prominent $\underline{n}\text{-}C_{27}$, $n\text{-}C_{29}$

III

CONCLUSIONS

1. Very good source rocks, rich in sapropelic kerogen are found in the section from 3,376 to 3,625 metres of the Norwegian North Sea 15/9-1 well.

2. The source rocks are just mature and would produce mainly gas and condensate, with some heavy oil.

3. Maturation increases rapidly in the sandstones with coals at the base of the analysed section.

4. The produced oil is a heavy oil associated with gas and condensate and generated from source rocks which are more mature than those analysed.

5. The oil shows similarities with the source rocks between 3,505 and 3,586 metres and is likely to have been generated from them off-structure at a depth of about 4,000 metres.

TABLE 1

MATURATION EVALUATION DATA

COMPANY: STATOIL WELL: 15/9-1 (N) LOCATION: NORTH SEA NORWAY

SAMPLE DEPTH (METRES) OR NOTATION	SAMPLE TYPE	GENERALISED LITHOLOGY	MAXIMUM PALAEOTEMP-ERATURE °F	VITRINITE REFLECTIVITY %	SPORE COLOURATION (1-10)	LIGHT HYDROCARBONS
3349-352	Ctgs	Med gy sh+10% gy-red sh+mnr chk	-	-	4.0	-
3415	"	Dk gy calc slty sh+mnr med gy sh+ gy-red sh	-	0.53	-	-
3433	"	Ditto	-	0.54	3.5	-
3487	"	Dk gy sh+mnr sst	-	0.48	4.0	-
3550	"	Ditto	-	0.50	-	-
3580.3	Core	Ditto	-	-	4.5	
3586	Ctgs	Ditto	-	0.55	-	-
3647	Core	Coal	-	0.72	-	-
3669.6	"	Coal	-	0.72	-	-
3671.85	"	Grey sand	-	-	5-6	-

TABLE 2

ORGANIC CARBON CONTENTS

COMPANY: STATOIL WELL: 15/19-1 (N) LOCATION: NORTH SEA, NORWAY

Cuttings

Sample Depth (Metres)	Lithology	Organic Carbon %
3349-352	Med gy sh+10% gy-red sh+mnr chk	0.45
3358-361	" +20% " +20% "	0.29
3367-370	Chk(often pyritic)+10% gy-red sh+mnr gy sh	0.39
3376-379	Dk gy calc slty sh	6.71
3385-388	"	10.26
3394-397	" +20% med gy calc sh	2.94
3403-406	" +10% "	2.99
3415	" +mnr "	4.00
3424	" +20% "	2.73
3433	"	4.00
3442	"	4.07
3451	"	4.36
3460	"	3.52
3469	"	5.40
3478	"	4.00
3487	" +mnr sst	5.07
3496	" + "	4.11
3505	"	3.19
3514	"	3.13
3523	"	3.74

TABLE 2 (Cont'd.)

Cuttings

Sample Depth (Metres)	Lithology	Organic Carbon %
3532	Dk gy calc slty sh+mnr sst	3.10
3541	"	4.01
3550	Dk gy calc sh+mnr gy-red sh	3.86
3559	Dk gy slty sh	4.05
3568	Dk gy sh	4.23
3577	"	4.65
3586	"	5.02
3595	"	4.96
3603	"	5.31
3612	" +mnr sst	5.08
3625	Dk gy slty sh+mnr sltst	4.94
3639-3734	Oil stained sandstone with minor shales and coals	

Picked lithologies

3349-352	Med gy sh	0.68
"	Gy-red sh	0.61
3358-361	Med gy sh	0.22
3394-397	Dk gy sh	2.36
"	Med-lt gy calc sh	1.26
3424	Dk gy sh	5.54
"	Med-lt gy calc sh	4.78

TABLE 3

SOURCE ROCK EVALUATION DATA

COMPANY: STATOIL WELL: 15/9-1 (N) LOCATION: NORTH SEA NORWAY

SAMPLE DEPTH METRES OR NOTATION	SAMPLE TYPE	ANALYSED LITHOLOGY	ORGANIC CARBON % OF ROCK	TOTAL EXTRACT P.P.M.	EXTRACT % OF ORGANIC CARBON	HYDRO--CARBONS P.P.M. OF ROCK	HYDRO-CARBONS % OF EXTRACT	TOTAL ALKANES % HYDRO-CARBONS
3376-379	Ctgs	Dk gy slty sh	7.80	11900	15.2	1010	9	51
3415-433	"	Dk gy slty sh+med gy calc sh+mnr gy-red sh	2.71	3700	11.7	190	5	72
3442-460	"	Ditto	3.81	4200	11.0	185	4	59
3469-496	"	Ditto	4.07	3800	9.4	365	10	60
3505-514	"	Ditto	2.74	2300	8.4	310	14	57
3523-550	"	Ditto	3.29	2800	8.4	420	15	71
3559-586	"	Dk gy slty sh	3.81	3400	9.0	340	10	64
3595-625	"	Ditto+mnr sst	4.30	5100	11.9	330	6	66

Organic carbon % determined on extracted rock and recalculated

TABLE 4

OIL FRACTIONATION DATA

COMPANY: STATOIL WELL: 15/9-1 (N) LOCATION: NORTH SEA NORWAY

ELUANT SYSTEM	SAMPLE HEATED TO 60° C	OIL AS PRODUCED (BUBBLE HOSE)
Pentane Fraction (%)	37	49
Pentane/25% Toluene Fraction (%)	20	31
Methanol Fraction (%)	20	7
Residue (%) (by Difference)	41	13
Asphaltenes (%) (by Precipitation)	37	
MOLECULAR RATIOS		
Pristane/n-C_{17}	0.63	0.62
Phytane/n-C_{18}	0.45	0.45
Pristane/Phytane	1.45	1.46
iso-C_{18}/Phytane	0.63	0.58
iso-C_{16}/Phytane	0.65	0.70
iso-C_{15}/Phytane	-	0.50
iso-C_{14}/Phytane	-	-

Pentane Fraction (%) - Contains saturated hydrocarbons (alkanes)

Pentane/25% Toluene Fraction (%) - Contains aromatic hydrocarbons

Methanol Fraction (%) - Contains elutable NSO containing compounds

Residue (%) (by difference) - Contains asphaltenes and other highly polar non-elutable compounds -calculated by difference between total column loading and recovered fractions.

Asphaltenes (%) (by precipitation) - weighed fraction of asphaltene precipitated from solution by addition of pentane.

iso $C_{18, 16, 15, 14}$ - refers to the isoprenoid series with respectively 18,16,15, and 14 carbon atoms in the chain.

All analyses have been carried out in duplicate or triplicate and results are quoted as average values.

TABLE 5

GAS CHROMATOGRAPHIC DATA

COMPANY: STATOIL WELL: 15/19-1 (N) LOCATION: NORTH SEA, NORWAY

Depth (metres)	n-alkanes			even/odd	ratios	
	C.max	range	prominent peaks	dominance	Pr/n-17	Ph/n-18
3376-379	20	17-27	20, 24	even	0.68	0.96
3415-433	20	17-29	20, 24, 29	even	0.98	0.96
3442-460	25	18-29	26, 29	odd	0.75	0.75
3469-496	23	19-29	27, 29	odd	1.40	0.82
3505-514	21	19-26	23, 26, 29	even	0.88	0.70
3523-550	20	17-26	26, 29	-	1.18	0.88
3559-586	20	18-29	26, 29	odd	1.08	0.82
3595-625	20	18-27	27, 29	odd	1.06	0.80
Oil	22	11-26	22, 24	-	0.60	0.44

Key.

C. max : carbon number of n-alkanes in greatest concentration.

range : carbon numbers of n-alkanes with concentrations between 50% and 100% of C. max.

FIGURE I

SPORE COLOURATION INDICES AGAINST DEPTH

COMPANY : STATOIL WELL : 15/9-1(N) LOCATION : NORTH SEA, NORWAY

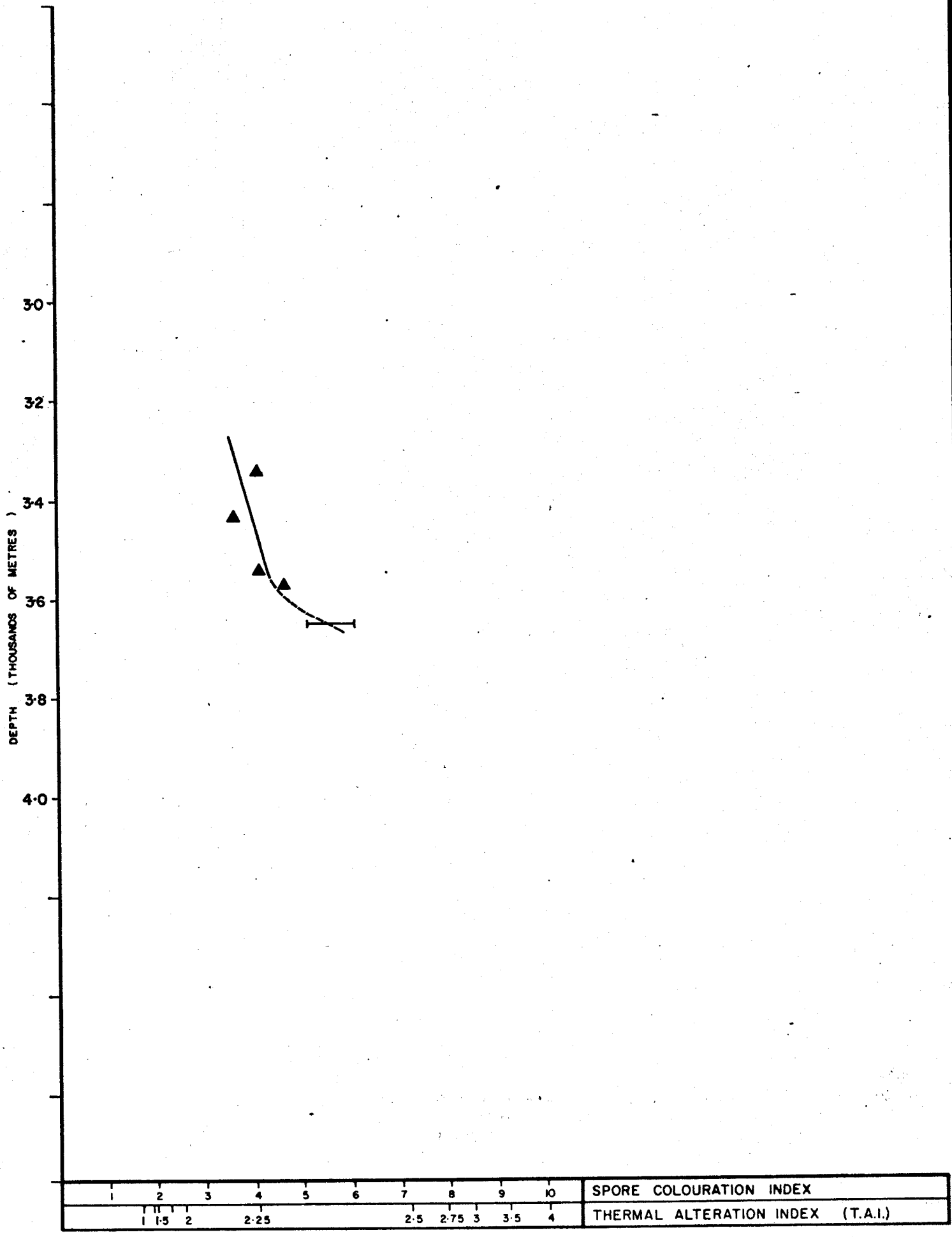


FIGURE 2

VITRINITE REFLECTIVITY AGAINST DEPTH

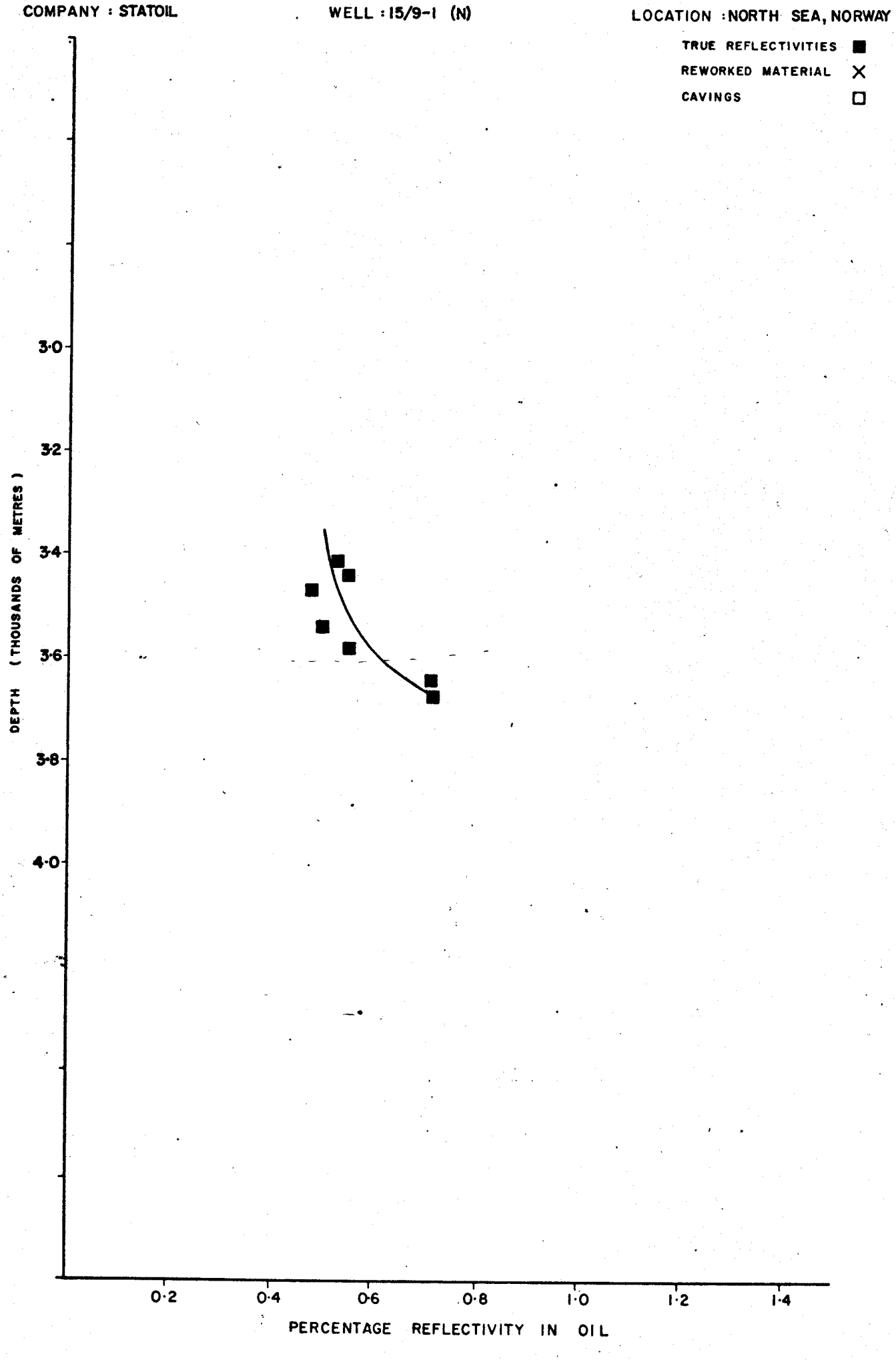


FIGURE 3

GAS CHROMATOGRAMS OF ALKANES FROM SOURCE ROCKS AND OILS

1. (a) 3376 - 379 metres

(b) 3415 - 433 metres

(c) 3442 - 460 metres

(d) 3469 - 496 metres

(e) 3505 - 514 metres

(f) 3523 - 550 metres

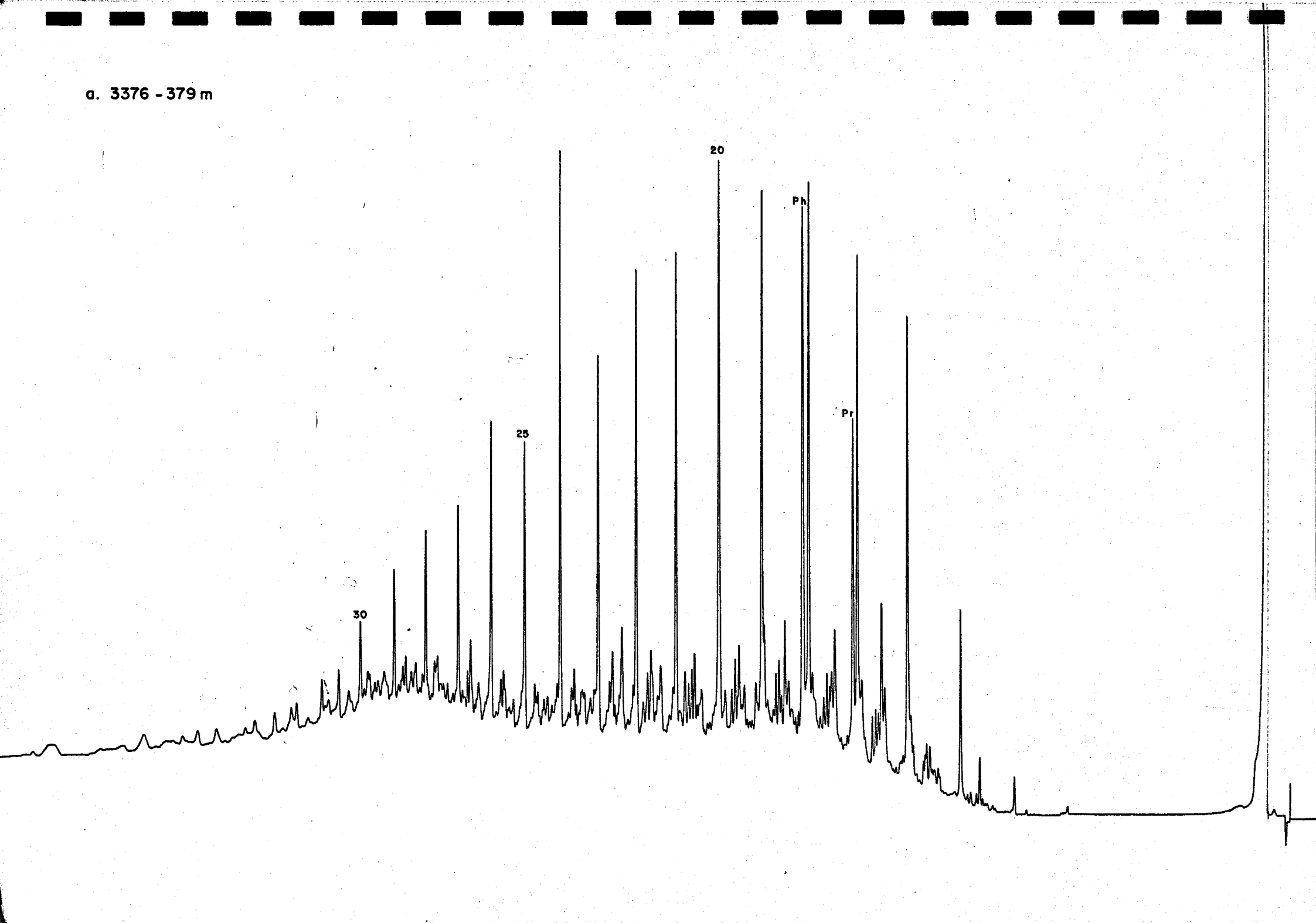
(g) 3559 - 586 metres

(h) 3595 - 625 metres

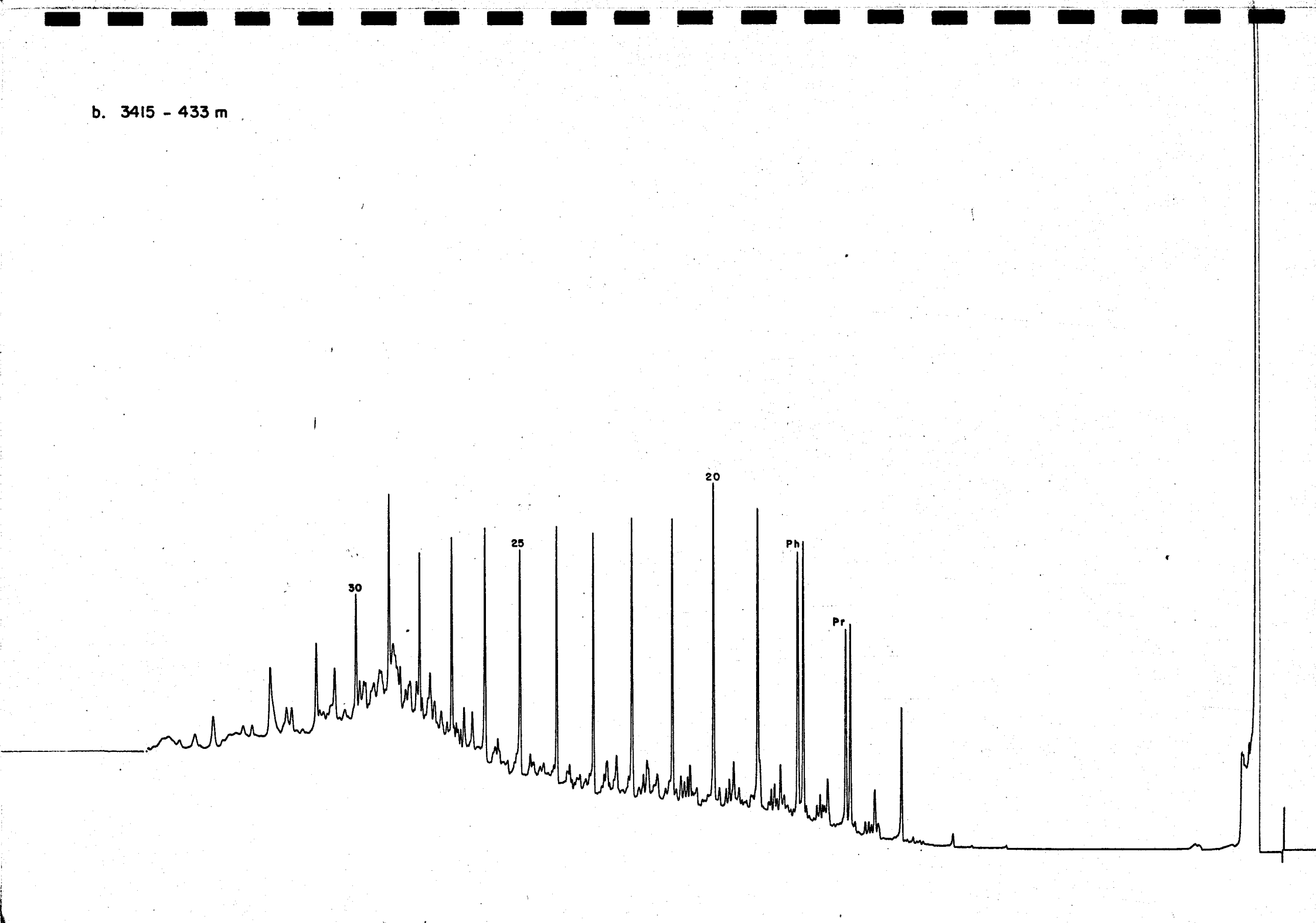
(i) Oil

Analysed on 20 metres wall-coated open-tubular column, OV-1 stationary phase, helium gas flow, programmed from 60°C to 260° C at 5°C/min.

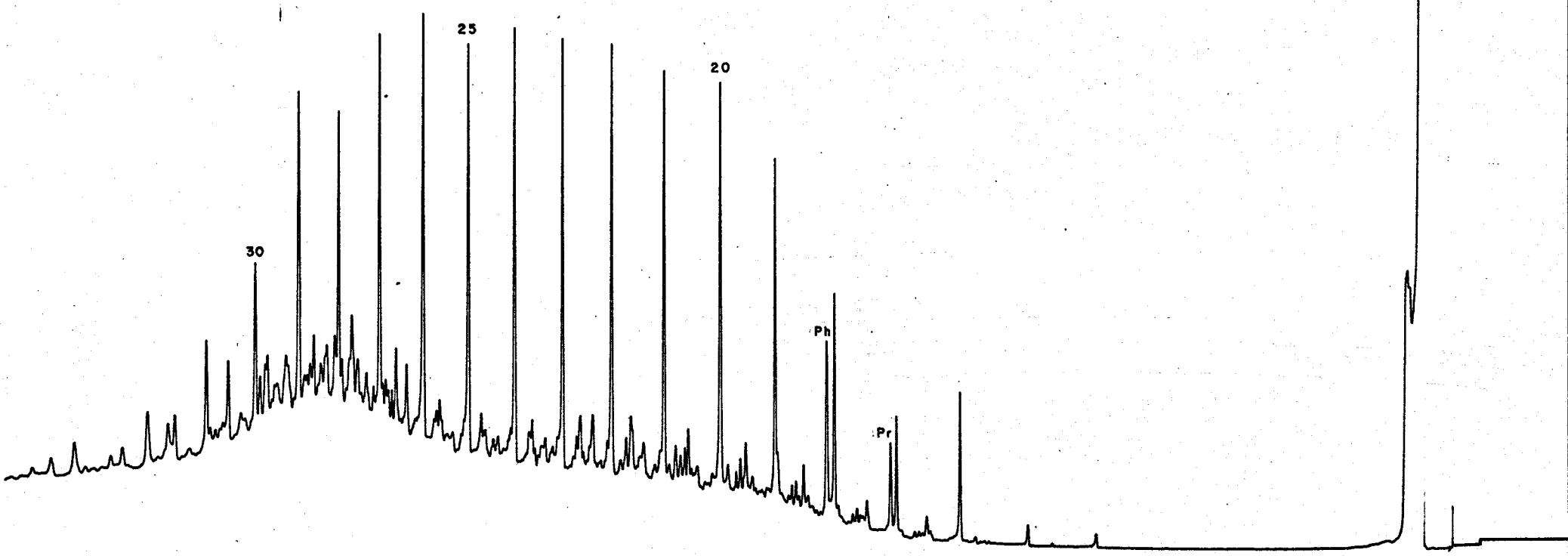
a. 3376 - 379 m

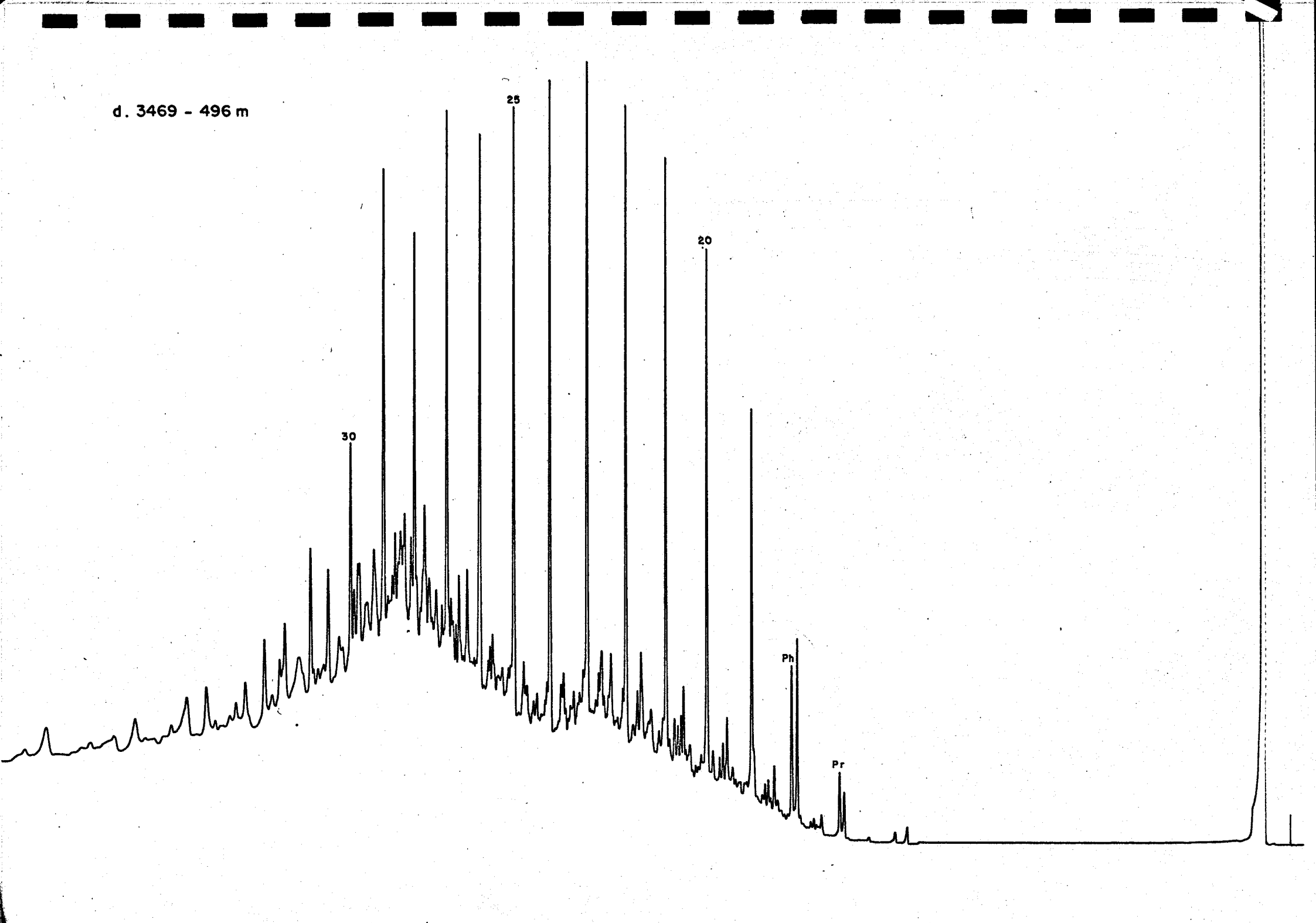


b. 3415 - 433 m

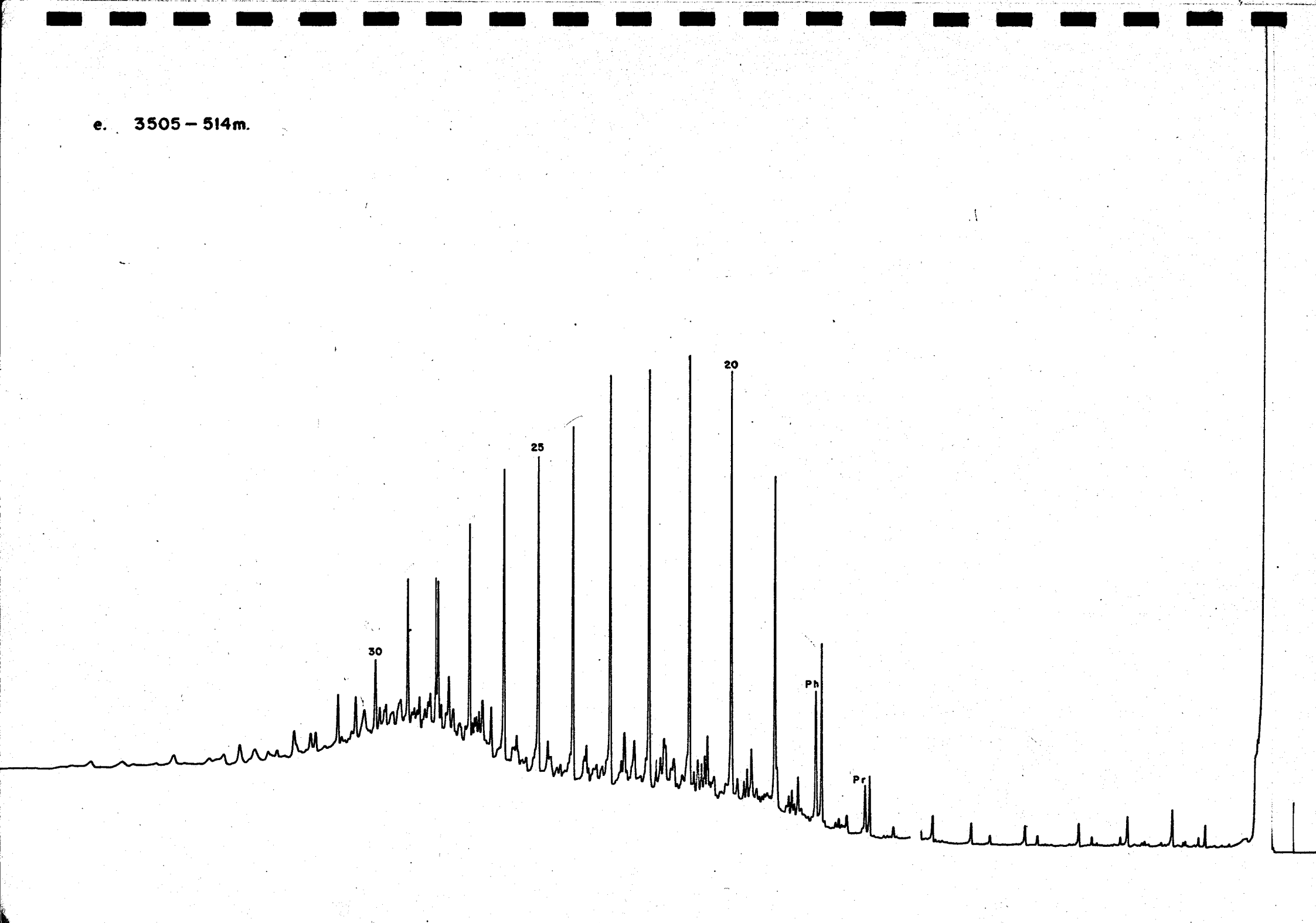


c. 3442 - 460 m

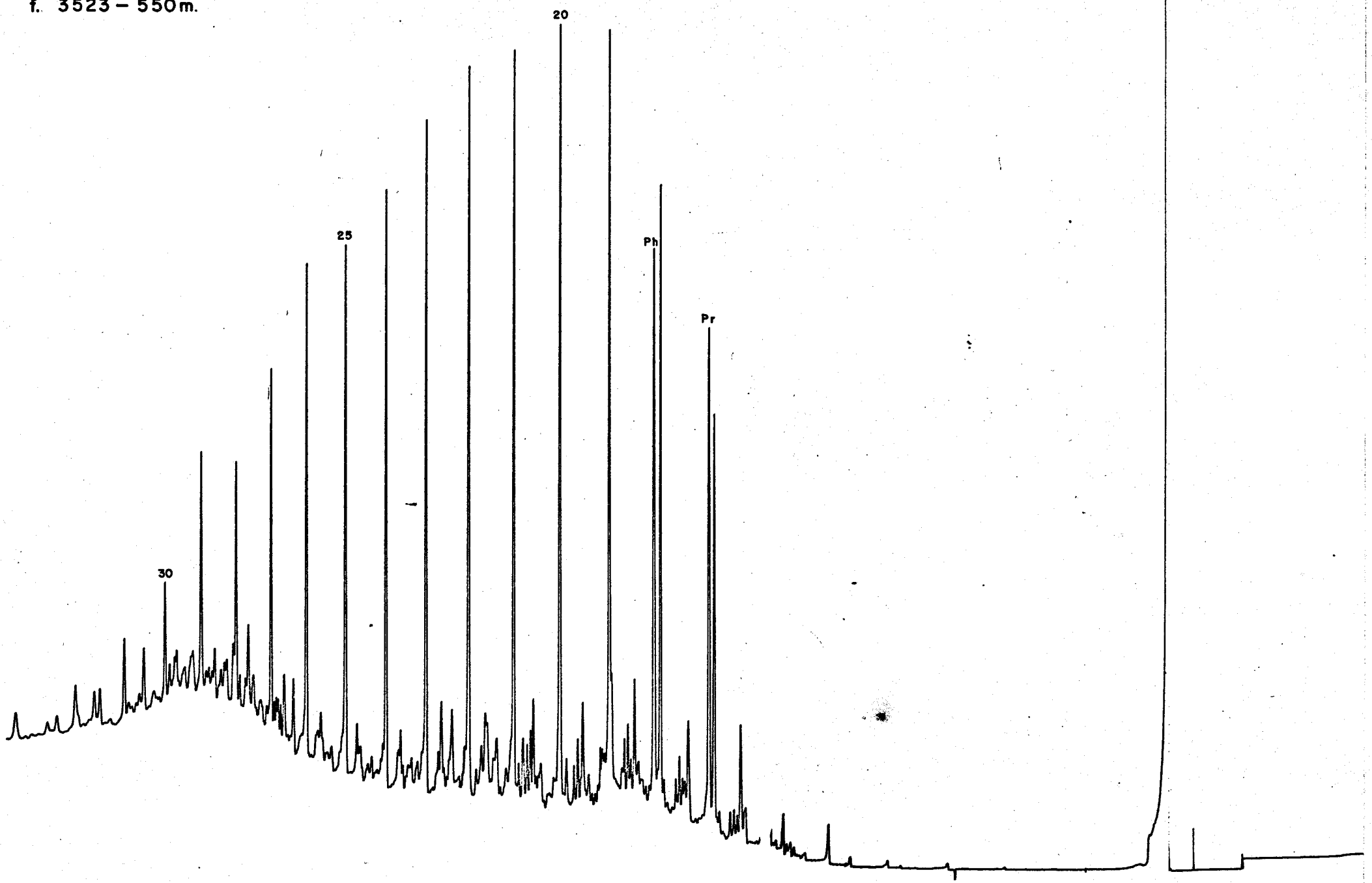



d. 3469 - 496 m
30
25
20
Ph
Pr

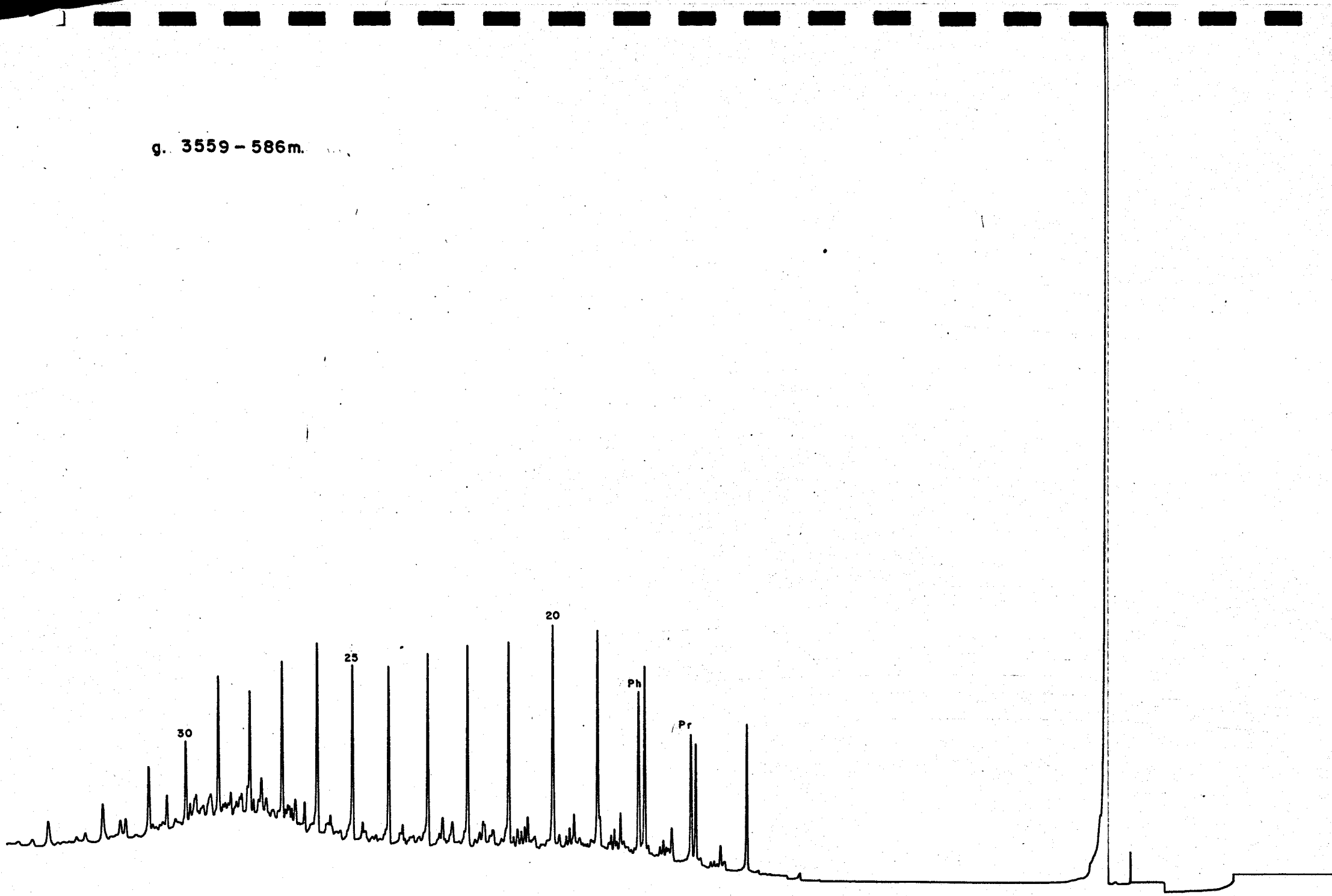
e. 3505 – 514m.



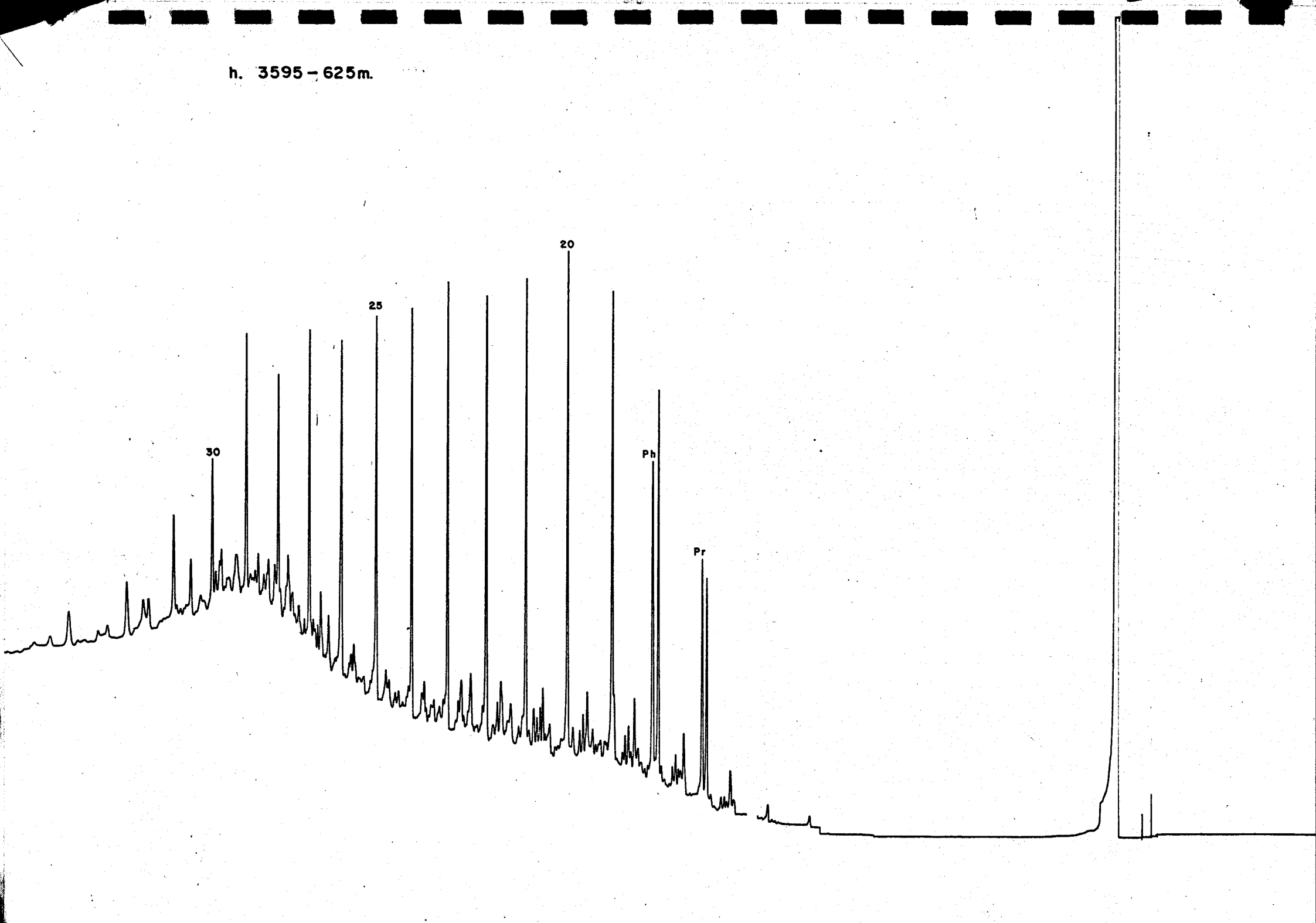
f. 3523 – 550m.



g. 3559 – 586m.



h. 3595 – 625m.



i.. 15/9 – 1 (N)

Oil