



Continental Shelf Institute

Institutt for
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REPORT TITLE

Analysis of oil from well 34/10-1.

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SUMMARY

Two oil samples from well 34/10-1 have been analysed by gas chromatography and combined gas chromatography/mass spec. (GC-MS). The oils from the two levels are found to be almost identical, both being immature and no definite proof of biodegradation.

KEY WORDS

Oil analysis

EXPERIMENTAL

Light hydrocarbons

The analytical apparatus, shown schematically in Figure 1, consists of a capillary gas chromatograph which has been substantially modified. In detail, the sample inlet and the carrier gas supply systems have been re-designed, and a backflush system has been introduced. In place of the injection port the 25 cm front length of the capillary extends outside the GC oven and is bent into a U-shape to serve as a cold trap (No. 10 in Figure 1). For the light hydrocarbon extraction procedure, a glass tube containing the freshly crushed rock sample (No. 9 in Figure 1) is placed between cold trap and carrier gas supply. The sample is stripped by passing a measured volume of hydrogen through the tube at ambient temperature. For this purpose the hydrogen flow is held constant by an all-metal flow controller (Siemens type, No. 3 in Figure 1). Since only a fraction of the light hydrocarbons present in the rock is recovered by this method it is essential that measurements be made under constant conditions with respect to temperature, gas flow rate and volume. The development of the backflush technique employed here is based on DEANS' method of column switching. The capillary column is cut into two lengths (ca. 1 : 2 ratio), and connected with a T-union of a low dead-volume which avoids peak broadening by diffusion during the analysis. This has been achieved by a design based on suggestions of SCHOMBURG et al..

Once the sample tube is replaced with an empty glass tube, and a stable baseline obtained, GC analysis is started by removing the liquid nitrogen from the cold trap. The trapped components are then transported onto the capillary according to their volatility. No gas stream split is used with sample introduction. Subsequent GC analysis is performed isothermally, hence the components can be identified by their KOVATS retention indices. Details of hydrogen stripping and GC analysis parameters are summarized in Table I.

In principle, the conditions for light hydrocarbon analysis of crude oils are very similar. But in order to prevent overloading the capillary only trace amounts of hydrocarbons should enter the column. This can be achieved by applying short stripping times (about one minute) on a very

small amount of crude oil, e.g. a quantity which has been drawn into a very thin glass capillary (generally < 0.1 mg). The main advantage of this type of crude oil analysis is the absence of any pretreatment step. Furthermore, any thermal degradation of sensitive compounds in the heated injection ports of normal chromatographs is avoided.

Chromatographic analysis

0.1 l of the whole oil was injected onto a 25 m glascapillary column coated with OV 101, using a split of 1:80 during the injection. The column was programmed from 30⁰C to 270⁰ at 4⁰C, with N₂ as carrier gas.

Aliquots of the oils were introduced onto silicacolumns and eluted with hexane, benzene and methanol, Table II. The C₁₅⁺ fractions, both the saturated and aromatic hydrocarbon fraction, were then analysed gas chromatographically using the same conditions as for the whole oil.

RESULTS AND DISCUSSION

Light hydrocarbons

Two parallel's of each of the two oil samples from well 34/10-1 were analysed for light hydrocarbons and the various results are shown in Table I.

We notice from these analyses that the naphthenic (cyclic) compounds are rather pronounced for the C₆ and C₇ compound while the iso- and n-alkane compounds are minor constituents. Further, we notice that the iso C₄/nC₄ ratio (0.7) and iso C₅/nC₅ ratio (2.0) are very high for crude oils. These results indicate that we here have got immature oils.

The two samples give very similar results for the various calculated parametres, they are almost identical.

Gas chromatographic study of whole oil

Both oil samples were analysed on a glasscapillary GC-column, Fig. 3, a and b. These chromatograms are very different to what is normally found for crude oil i.e. it is very difficult to see any pattern of n-alkanes which are normally the most prominent peaks in the gas chromatograms, and almost nothing is found above nC₂₀. To try to identify some of the types of compounds in the samples, a GC-MS study was undertaken. The oil was run in the same manner as for the GC analyses, and compounds with major fragmentation of m/e 57 + 71 (n-alkanes), 69 (cyclic alkanes and alkanes), 78 (Benzene), 91 (Toluene and Methyl Benzenes), 128 (naphthalene), 142 (Me-naphthalenes), 156 (Dimethyl-naphthalenes) and 170 (Trimethyl-naphthalenes). The two last fragmentations ions will also be molecular ions for nC₁₁ and nC₁₂.

This GC-MS analysis shows that while benzene is only a minor compound in the sample, other aromatic compounds like Toluene, Methyl-benzenes, ethyl-benzenes, methyl, ethyl-benzenes and naphthalene are major compounds. It is not normal in a crude oil to find the various aromatic compounds as dominant as in this sample. We see further that various cyclic compounds are very dominant together with various iso alkanes.

The n-alkanes can easily be picked out using this identification technique, and we see that the iso-alkanes are more dominant than the n-alkanes.

The crude oils were chromatographed on silica column and both the saturated and the aromatic hydrocarbon fractions analysed gas chromatographically.

The two chromatograms of the saturated hydrocarbon fractions are almost identical. The iso alkanes are the most prominent peaks in the chromatogram, with pristane as the dominant peak. The n-alkanes above nC_{20} can, however, be recognized now, while they were completely hidden in the background in the chromatograms of the whole oil.

The chromatograms of the aromatic fractions are very front biased. The various compounds are not identified, but a chromatogram of this type, with a very front biased pattern is typical for an immature sample.

CONCLUSION

The maturity of a crude oil can be determined in various ways, depending of the type of source for the oil. If the source is of herbaceous origin, an immature oil will have a high CPI value ($nC_{24} - nC_{30}$) while a mature oil has a CPI value close to unity. This parameter can not be used for this oil since it is obviously not derived from a herbaceous source.

Another parameter is the pristane/ nC_{17} ratio. This ratio is high for immature oils while it is low for mature oils. Other parametres are the various iso alkanes/ n -alkanes ratios, like iso C_4/nC_4 , iso C_5/nC_5 etc. Immature oils have large iso alkane/ n -alkane ratios. These ratios decrease with increasing maturity and mature oils normally have ratios of 0.3 or less for iso C_4/nC_4 .

If an oil is exposed to bacteriological activity, the n -alkanes around nC_{20} or higher are the first to be attacked. Later iso alkanes and cyclic alkanes will be attacked while aromatic compounds are relatively stable to bacteriological activity. Crude oils which have been exposed to bacteriological activity will therefore have only small amounts if any of n -alkanes, depending on the severeness of the bacteriological attack. The bacteria will, when consuming alkanes, produce other compounds. These will be seen as a large unresolved envelope covering almost the whole chromatogram. Such an unresolved envelope is not recorded for any of the analysed oils, and we do not therefore believe these oils to have been exposed to bacteriological activity. A carbon isotope study of the oil and of the various fractions will probably be the best analysis to prove or disprove whether there has been bacteriological activity.

On the background of the various analysis performed on the oils from well 34/10-1, we conclude that the samples from the two depths are almost chromatographically identical and that they are very immature oils. At present, we have no evidence for a bacteriological activity on these oils but until a carbonisotope analysis is undertaken, we will keep this possibility open.

Code for various calculations

SE Total alkanes in ppb (W/W) in 100 ml elution gas.

SC Dito, normalized to C org.

(4) n-Butane, unity as SC.

(23) n-Heptane, " " "

(15) Benzene, " " "

(28) Toluene, " " "

(25) Methyl cyclohexane, unit as SC.

M ΣC_2-C_5 % of total amount.

T,U,V Paraffin/Naphthene ratio for C₅, C₆ and C₇.

Gx, Ix, Nx, Ax with x = 5, 6, 7 for 100% normalized n-alkanes,

iso-alkane, Napthene and Aromatic content for C₅, C₆ and C₇.

N Ethane / n-Heptane

A Propane / n-Heptane

B n-Butane / n-Heptane

C Benzene / n-Heptane

G n-Heptane / n-Heptane + Cyclopentane + Cyclohexane + 2-Methylhexane
+ 3-Methylhexane + Methylcyclohexane + Σ Dimethylcyclopentane (in %).

E n-Hexane / Methylcyclopentane + 2,2-Dimethylpentane

P n-Pentane / Cyclopentane

Q n-Hexane / Cyclohexane

L n-Heptane / Methylcyclohexane

H n-Hexane + n-Heptane/Cyclohexane + Methylcyclohexane

F Cyclohexane / Methylcyclopentane + 2,2-Dimethylpentane

I 2-Methylhexane + 3-Methylhexane / Σ Dimethylcyclopentane

W Ethane / Propane

X Isobutane / n-Butane

Y Isopentane / n-Pentane

Z Cyclopentane / 2-Methylpentane

Z1 2-Methylpentane / 3-Methylpentane

Z3 2-Methylhexane / 2,3-Dimethylpentane

Z4 3-Methylhexane / 1,cis-3-Dimethylcyclopentane

Z5 1,trans-3-Dimethylcyclopentane / 1,trans-2-Dimethylcyclopentane

K n-Heptane / 2-Methylhexane

1930-35 (B) RI.T.: 4

EW: 1.000 G OC: 0.00%

TF: 1935.

TE: 10.0 MIN HS: 10.0 ML/MIN

MK: 49.0 VPM

MR: 100.

MV: 100.

TM: 68.3

NAME	RET.-IND.	TMS	XEP	XCP
ETHANE	160.6*	70.	0.649E 01	0.000
U	257.4	74.	0.351E-01	0.000
U	270.3	75.	0.112E 01	0.000
PROPANE	300.0*	78.	0.156E 02	0.000
U	348.3	86.	0.363E 00	0.000
MC3	368.3	91.	0.236E 02	0.000
2,2-DMC3	384.3	96.	0.959E 00	0.000
N-C4	400.0*	102.	0.319E 02	0.000
U	410.6	107.	0.363E 00	0.000
U	462.3	130.	0.269E 00	0.000
MC4	474.2	137.	0.604E 02	0.000
U	489.3	147.	0.222E 00	0.000
N-C5	500.0*	155.	0.297E 02	0.000
U	518.8	171.	0.117E-01	0.000
2,2-DMC4	537.6	190.	0.322E 01	0.000
CYC5	564.2	223.	0.156E 02	0.000
2-MC5	569.8	231.	0.474E 02	0.000
3-MC5	585.0	255.	0.239E 02	0.000
N-C6	600.0*	282.	0.223E 02	0.000
MCYC5+2,2-DMC5	628.6	345.	0.638E 02	0.000
BENZENE	643.6	385.	0.730E 01	0.000
U	660.8	438	0.138E 01	0.000
CYC6	663.7	448	0.748E 02	0.000
2-MC6	667.2	460.	0.183E 02	0.000
2,3-DMC5	673.0	481.	0.146E 02	0.000
3-MC6	676.9	496.	0.315E 02	0.000
1,CIS-3-DMCYC5	683.9	524.	0.164E 02	0.000
1,TRANS-3-DMCYC5	688.0	541.	0.182E 02	0.000
1,TRANS-2-DMCYC5	690.6	552.	0.240E 02	0.000
N-C7	700.0*	595.	0.209E 02	0.000
1,CIS-2-DMCYC5	721.4	707.	0.479E 01	0.000
MCYC6	727.6	744.	0.122E 03	0.000
ECYC5	734.1	785.	0.167E 02	0.000
U	739.4	820.	0.339E 00	0.000
1TR2CIS4TMCYC5	742.9	844.	0.940E 01	0.000
U	745.2	860.	0.131E 01	0.000
1TR2CIS3TMCYC5	749.4	891.	0.838E 00	0.000
TOLUENE	751.7	908.	0.660E 02	0.000
U	761.2	983.	0.780E 01	0.000
U	765.4	1018.	0.196E 02	0.000
U	767.4	1036.	0.544E 01	0.000
U	772.3	1079.	0.984E 01	0.000

1930-35 (A) RI.T.: 4

EW: 1.000 G OC: 0.00% TF: 1935.
 TE: 10.0 MIN HS: 10.0 ML/MIN MK: 49.0 VPM MR: 100.
 MV: 100.
 TM: 66.2

NAME	RET.-IND	TMS	XEP	XCP
U	124.5	68.	0.117E-01	0.000
ETHANE	194.7*	70.	0.604E 02	0.000
U	248.9	73.	0.140E 00	0.000
PROPANE	300.0*	78.	0.732E 02	0.000
U	327.0	82.	0.117E-01	0.000
U	352.4	87.	0.937E 01	0.000
MC3	368.7	91.	0.131E 03	0.000
2,2-DMC3	391.7	98.	0.110E 02	0.000
N-C4	400.0*	101.	0.184E 03	0.000
U	448.7	121.	0.936E-01	0.000
MC4	475.7	136.	0.376E 03	0.000
N-C5	500.0*	153.	0.176E 03	0.000
U	518.9	169.	0.105E 00	0.000
U	534.1	184.	0.146E 00	0.000
2,2-DMC4	538.7	189.	0.203E 02	0.000
U	561.6	217.	0.421E 00	0.000
CYC5	565.2	222.	0.986E 02	0.000
2-MC5	570.1	229.	0.296E 03	0.000
U	580.6	245.	0.211E 00	0.000
3-MC5	584.9	252.	0.148E 03	0.000
U	595.2	270.	0.351E 00	0.000
N-C6	600.0*	279.	0.132E 03	0.000
U	624.0	330.	0.702E-01	0.000
MCYC5+2,2-DMC5	628.1	340.	0.384E 03	0.000
BENZENE	642.9	379.	0.417E 02	0.000
U	659.8	430.	0.102E 01	0.000
CYC6	663.1	441.	0.489E 03	0.000
2-MC6	666.9	454.	0.114E 03	0.000
2,3-DMC5	672.5	474.	0.979E 02	0.000
3-MC6	676.3	488.	0.198E 03	0.000
1,CIS-3-DMCYC5	683.7	517.	0.101E 03	0.000
1,TRANS-3-DMCYC5	687.6	533.	0.113E 03	0.000
1,TRANS-2-DMCYC5	690.2	544.	0.156E 03	0.000
N-C7	700.0*	588.	0.129E 03	0.000
1,CIS-2-DMCYC5	721.7	700.	0.308E 02	0.000
MCYC6	728.0	737.	0.822E 03	0.000
ECYC5	734.1	775	0.106E 03	0.000
U	740.0	813.	0.232E 01	0.000
1TR2CIS4TMCYC5	742.5	830.	0.671E 02	0.000
U	745.8	853.	0.131E 01	0.000
TOLUENE	752.3	900.	0.477E 03	0.000
U	762.0	976.	0.500E 02	0.000
U	765.2	1003.	0.248E 02	0.000

1788-1792 (A) RI.T.: 4

EW: 1.000 G

OC: 0.00%

TF: 1792.

TE: 10.0 MIN

HS: 10.0 ML/MIN

MK: 49.0 VPM MR: 100.

MV: 100.

TM: 69.5

NAME	RET.-IND.	TMS	XEP	XCP
ETHANE	199.5*	73.	0.315E 02	0.000
PROPANE	300.0*	80.	0.586E 02	0.000
MC3	365.3	91.	0.993E 02	0.000
N-C4	400.0*	101.	0.134E 03	0.000
U	459.5	128.	0.117E-01	0.000
MC4	473.6	136	0.262E 03	0.000
N-C5	500.0*	154	0.132E 03	0.000
U	518.0	169.	0.585E-01	0.000
2,2-DMC4	538.2	189.	0.149E 02	0.000
CYC5	565.0	222.	0.216E 03	0.000
2-MC5	570.0	229.	0.216E 03	0.000
3-MC5	584.2	251.	0.109E 03	0.000
N-C6	600.0*	279.	0.108E 03	0.000
U	621.4	324	0.585E-01	0.000
MCYC5+2,2-DMC5	628.1	340.	0.306E 03	0.000
BENZENE	642.3	380.	0.330E 02	0.000
U	660.4	432.	0.580E 01	0.000
CYC6	663.4	442.	0.370E 03	0.000
2-MC6	666.9	454.	0.905E 02	0.000
2,3-DMC5	672.7	475.	0.758E 02	0.000
3-MC6	676.5	489.	0.157E 03	0.000
1,CIS-3-DMCYC5	683.8	518.	0.801E 02	0.000
1,TRANS-3-DMCYC5	687.7	536.	0.897E 02	0.000
1,TRANS-2-DMCYC5	690.3	545.	0.122E 03	0.000
N-C7	700.0*	589.	0.108E 03	0.000
1,CIS-2-DMCYC5	721.3	700.	0.248E 02	0.000
MCYC6	727.6	737.	0.656E 03	0.000
ECYC5	734.2	778.	0.857E 02	0.000
U	739.5	813.	0.187E 01	0.000
1TR2CIS4TCYC5	743.0	837.	0.545E 02	0.000
U	745.2	853.	0.114E 01	0.000
TOLUENE	751.7	900.	0.397E 03	0.000
U	761.2	975.	0.411E 02	0.000
U	765.4	1010.	0.110E 03	0.000
U	767.3	1027.	0.239E 02	0.000
U	770.5	1055.	0.761E 00	0.000

1788-1792 (B)

RI.T.: 4

EW: 1.000 G

OC: 0.00 %

TF: 1792.

TE: 10.0 MIN

HS: 10.0 ML/MIN

MK: 49.0 VPM MR: 100.

MV: 100.

TM: 68.7

NAME	RET.-IND.	TMS	XEP	XCP
ETHANE	212.6*	73.	0.233E 02	0.000
PROPANE	300.0*	80.	0.415E 02	0.000
MC3	365.2	92.	0.660E 02	0.000
N-C4	400.0*	103.	0.939E 02	0.000
U	410.2	107.	0.772E 00	0.000
U	460.4	129.	0.117E-01	0.000
MC4	474.1	137.	0.182E 03	0.000
N-C5	500.0*	155.	0.977E 02	0.000
U	518.8	171.	0.351E-01	0.000
2,2-DMC4	537.6	190.	0.104E 02	0.000
CYC5	564.9	224.	0.528E 02	0.000
2-MC5	569.8	231.	0.160E 03	0.000
3-MC5	584.4	254.	0.816E 02	0.000
N-C6	600.0*	282.	0.845E 02	0.000
Y	621.6	328.	0.468E-01	0.000
MCYC5+2,2-DMC5	628.6	345.	0.233E 03	0.000
BENZENE	543.9	386.	0.266E 02	0.000
U	660.6	438.	0.458E 01	0.000
CYC6	663.9	449.	0.276E 03	0.000
2-MC6	667.3	461.	0.704E 02	0.000
2,3-DMC5	673.1	482.	0.560E 02	0.000
3-MC6	676.8	496.	0.120E 03	0.000
1,CIS-3-DMCYC5	684.0	525.	0.619E 02	0.000
1,TRANS-3-DMCYC5	688.1	542.	0.694E 02	0.000
1,TRANS-2-DMCYC5	690.4	552.	0.924E 02	0.000
N-C7	700.0*	596.	0.835E 02	0.000
1,CIS-2-2DMCYC5	721.3	708.	0.181E 02	0.000
MCYC6	727.7	746.	0.482E 03	0.000
ECYC5	734.0	786.	0.639E 02	0.000
U	739.3	821.	0.109E 01	0.000
1TR2CIS4TMCYC5	742.7	845.	0.361E 02	0.000
U	745.1	862.	0.467E 01	0.000
1TR2CIS3TMCYC5	749.4	893.	0.324E 02	0.000
TOLUENE	751.5	909.	0.274E 03	0.000
U	761.0	985.	0.290E 02	0.000
U	765.0	1020.	0.777E 02	0.000
U	767.4	1039.	0.204E 02	0.000
U	771.8	1079.	0.213E 02	0.000

TABLE I
Light hydrocarbon data

Sample	TF	OC	SE	SC	(4)	(23)	(15)
1930-35(A)	1935.	0.00	0.451E 04	0.000	0.000	0.000	0.000
1930-35(B)	1935.	0.00	0.725E 03	0.000	0.000	0.000	0.000
1788-92(A)	1792.	0.00	0.346E 04	0.000	0.000	0.000	0.000
1788-92(B)	1792.	0.00	0.259E 04	0.000	0.000	0.000	0.000

Sample	TF	(28)	(25)	M	N	A	B	C	D
1930-35(A)	1935.	0.000	0.000	22.1	0.47	0.47	1.43	0.32	3.70
1930-35(B)	1935.	0.000	0.000	23.1	0.31	0.74	1.52	0.33	3.16
1788-92(A)	1792	0.000	0.000	20.3	0.29	0.54	1.24	0.31	3.68
1788-92(B)	1792	0.000	0.000	19.3	0.28	0.50	1.11	0.32	3.28

Sample	TF	G	E	P	Q	L	T	U	V
1930-35(A)	1935.	5.7	0.34	1.78	0.27	0.16	5.73	0.68	0.40
1930-35(B)	1935.	6.0	0.35	1.91	0.30	0.17	5.86	0.70	0.42
1788-92(A)	1792.	6.1	0.35	1.85	0.29	0.16	5.51	0.66	0.41
1788-92(B)	1792.	6.3	0.36	1.85	0.31	0.17	5.30	0.65	0.42

Sample	TF	H	F	I	W	X	X	Z	Z1
1930-35(A)	1935.	0.20	1.27	0.78	0.83	0.71	2.14	0.33	2.01
1930-35(B)	1953.	0.22	1.17	0.79	0.42	0.74	2.04	0.33	2.02
1788-92(A)	1792.	0.21	1.21	0.78	0.54	0.74	1.98	0.33	1.99
1788-92(B)	1792.	0.22	1.19	0.79	0.56	0.71	1.87	0.33	1.96

Sample	TF	Z3	Z4	Z5	K	G5	I5	N5
1930-35(A)	1935.	1.17	1.95	0.72	1.13	26.6	58.5	14.9
1930-35(B)	1935.	1.26	1.92	0.76	1.14	27.8	57.6	14.6
1788-92(A)	1792.	1.19	1.96	0.73	1.19	28.4	56.3	15.4
1788-92(B)	1792.	1.26	1.93	0.75	1.19	29.4	54.8	5.9

Sample	TF	G6	I6	N6	A6	G7	I7	N7	A7
1930-35(A)	1935.	8.7	30.7	57.8	2.8	5.5	17.4	56.7	20.3
1930-35(B)	1935.	9.2	30.6	57.2	3.0	5.9	18.2	57.2	18.7
1788-92(A)	1792.	9.3	29.4	58.4	2.9	5.7	17.1	56.1	21.0
1788-92(B)	1792.	9.7	28.9	58.3	3.1	6.0	17.7	56.6	19.7

TABLE II
Chromatographic separation of the oils

Sample	Oil (mg)	Sat (mg)	Aro (mg)	NSO fraction	Light components (mg)
1930-1935 m	1019.0	387.4	252.5	58.1	321.0
1788-1792 m	1042.0	395.6	260.2	59.3	326.9

Legends to Fig. 1.

Schematic representation of capillary chromatographic system for light hydrocarbon analysis of rock samples using hydrogen stripping.

1 = hydrogen inlet, 2 = hydrogen purification trap, 3 = flow controller, 4 = solenoid valve, 5 = needle valve, 6 = pressure regulator, 7, 8 = pressure gauge, 9 = sample extraction tube, 10 = cold trap, 11, 12 = capillary column, 13 = T-union, 14 = flame ionization detector, 15 = gas loop, 16 = six-port valve, 17 = inlet for external standard, 18 = on-off by-pass valve, → : direction of gas flow. → : reversed flow during backflush.

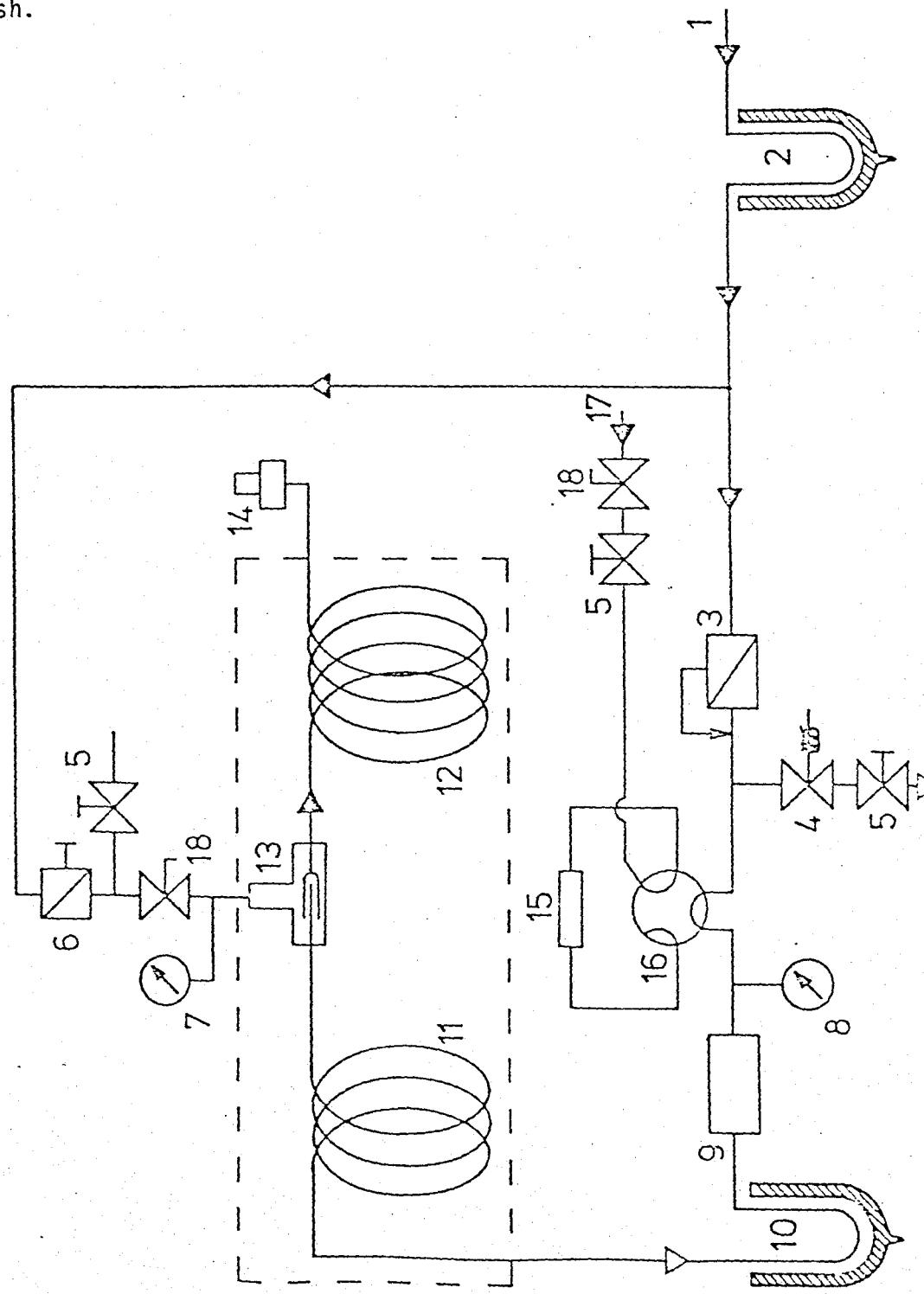


Fig. 2a. (1930 - 35 m)

Light hydrocarbons

Me cyclohexane

Toluene

oC₆C₇

nC₇

Cyclohexane

Me Cyclopentane

Benzene

nC₆

Cyclo-
pentane

iC₅

nC₅

nC₄

iC₄

nC₃ nC₂

Fig. 2b. (1788 - 1792 m)

Light hydrocarbons

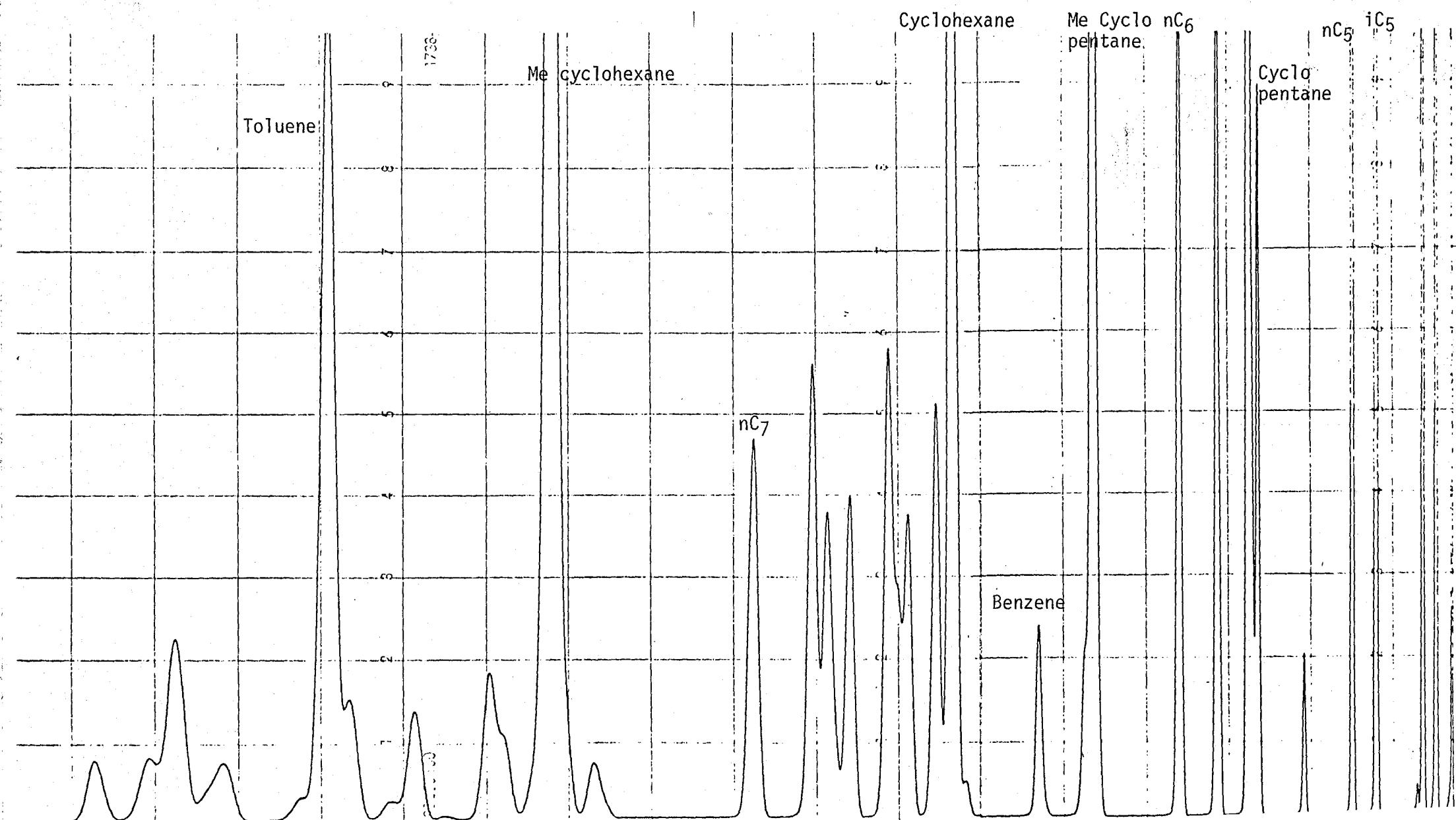


Fig. 3a. (1930 - 1935 m)

GC of whole oil

Pristane

Phytane

nC₁₆

nC₂₀

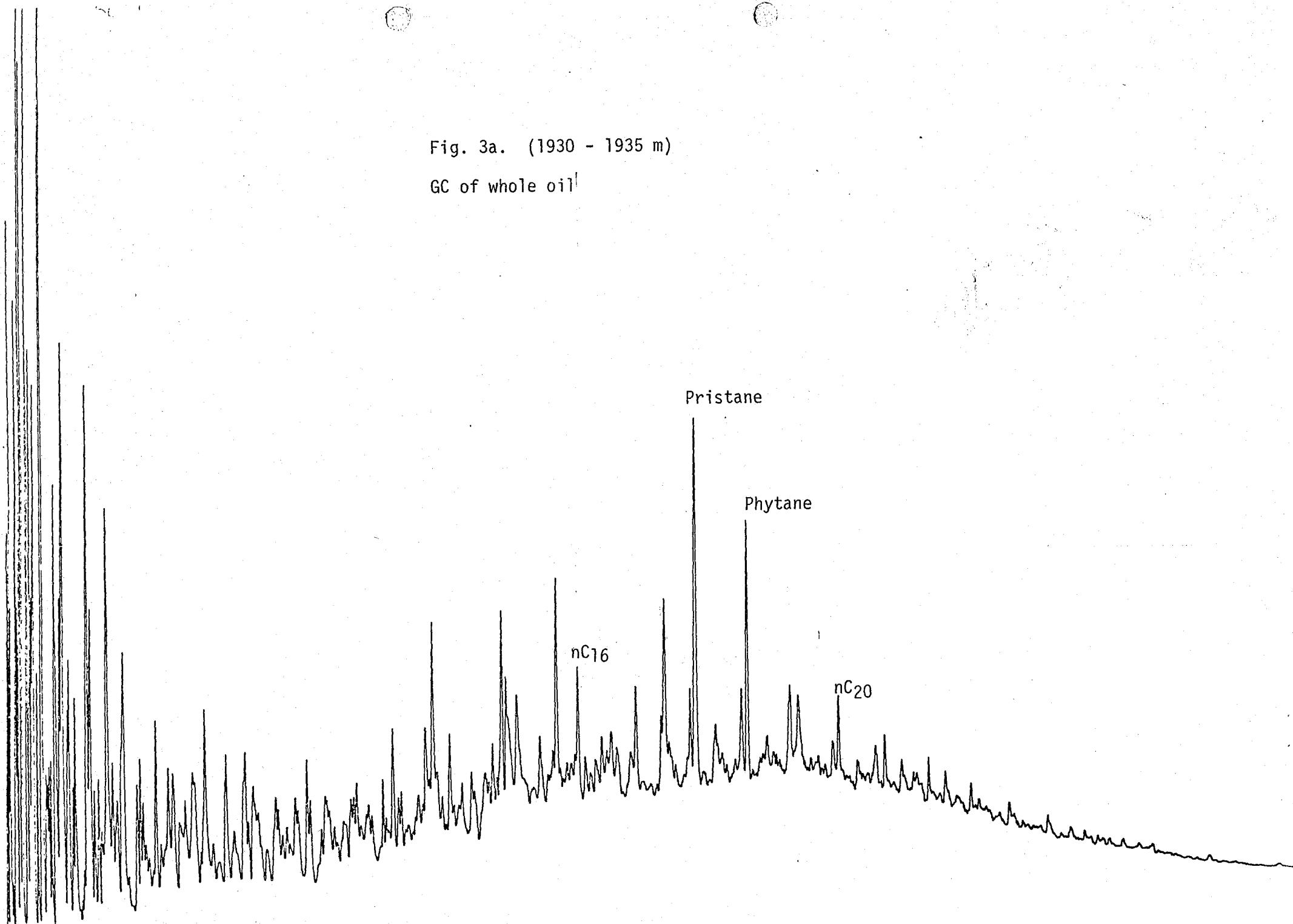


Fig. 4.

GC - MS of whole oil

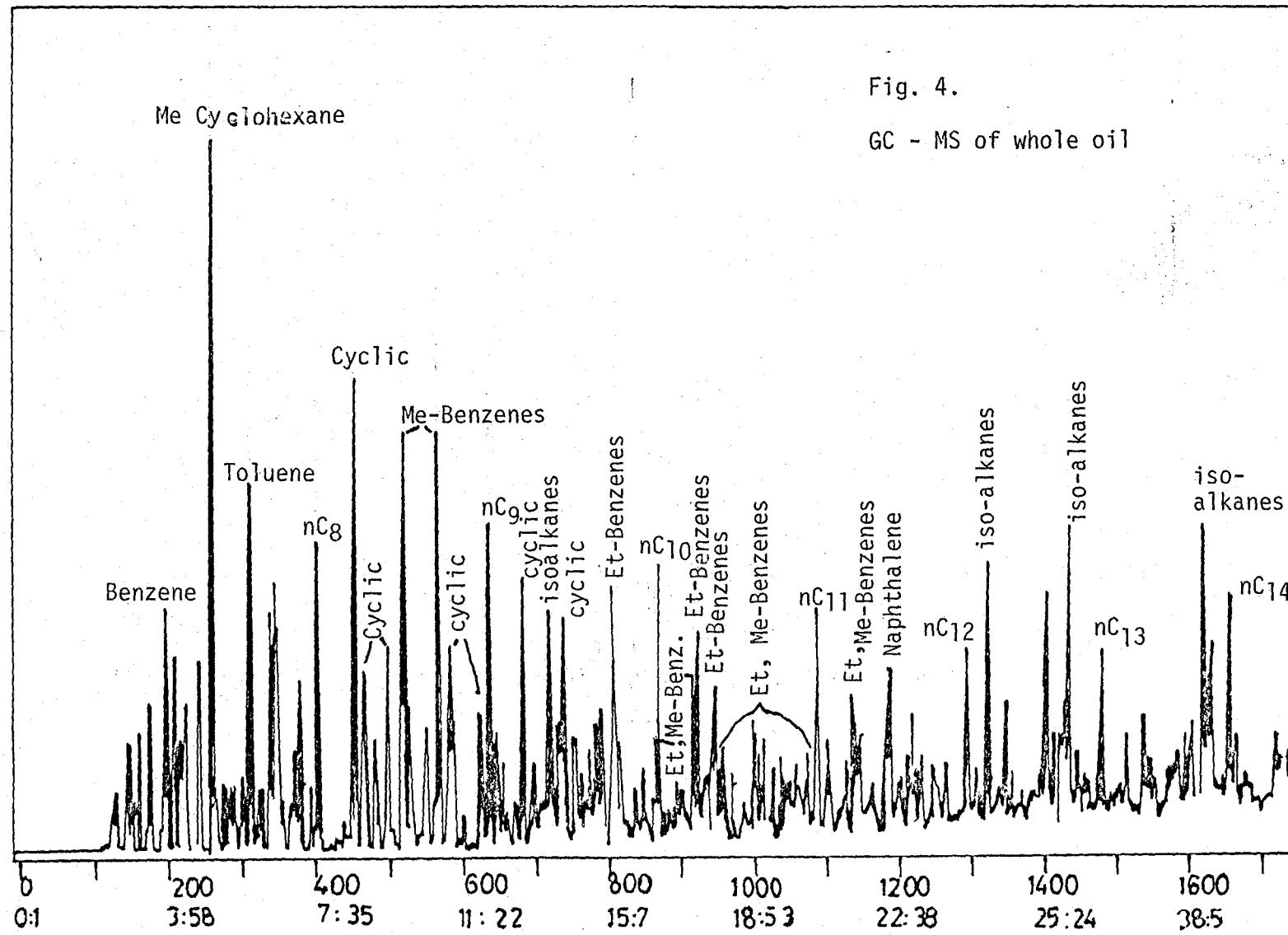


Fig. 3b. (1788 - 1792 m)

GC of whole oil

Pristane

Phytane

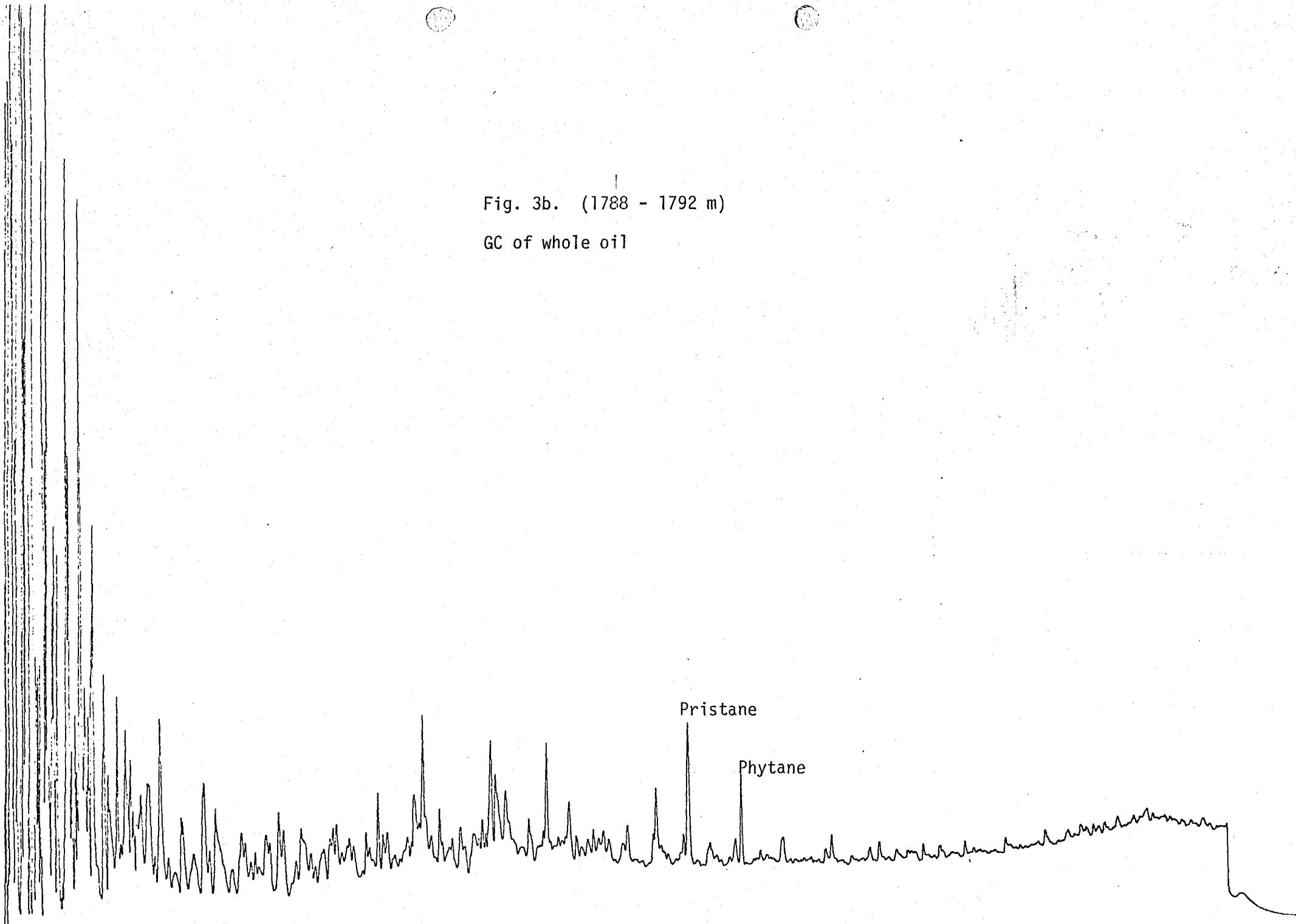


Fig. 5a. (1930 - 1935 m)

GC of saturated fraction

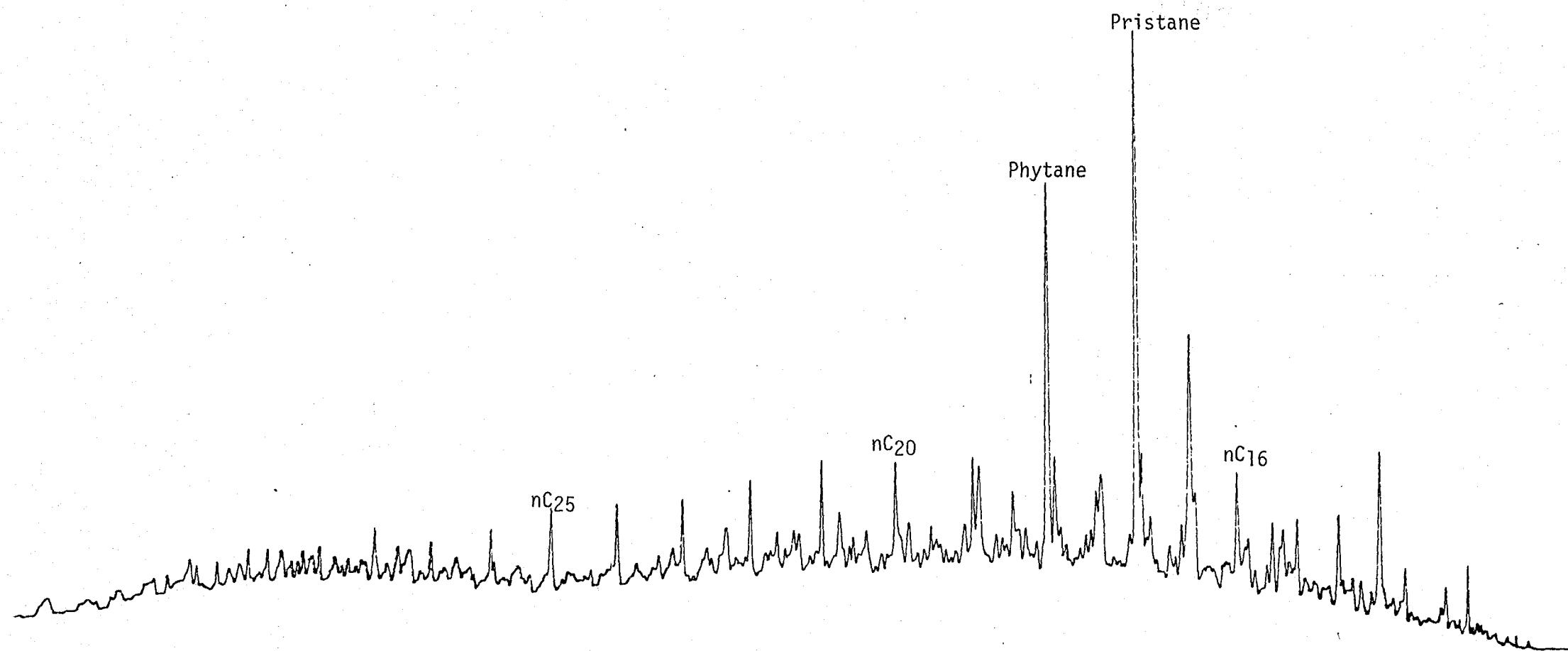


Fig. 5b. (1788 - 1792 m)

GC of saturated fraction

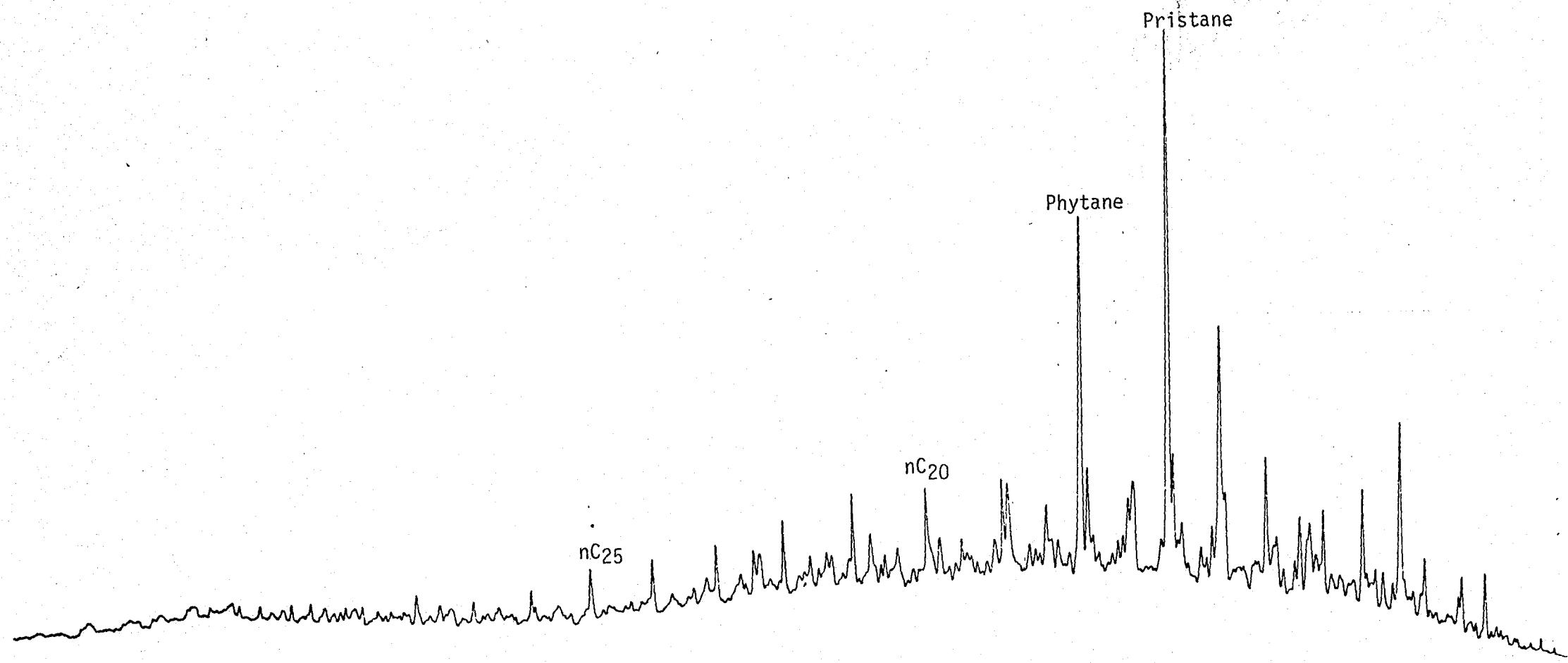


Fig. 6a. · (1930 - 1935 m)

GC of aromatic fraction

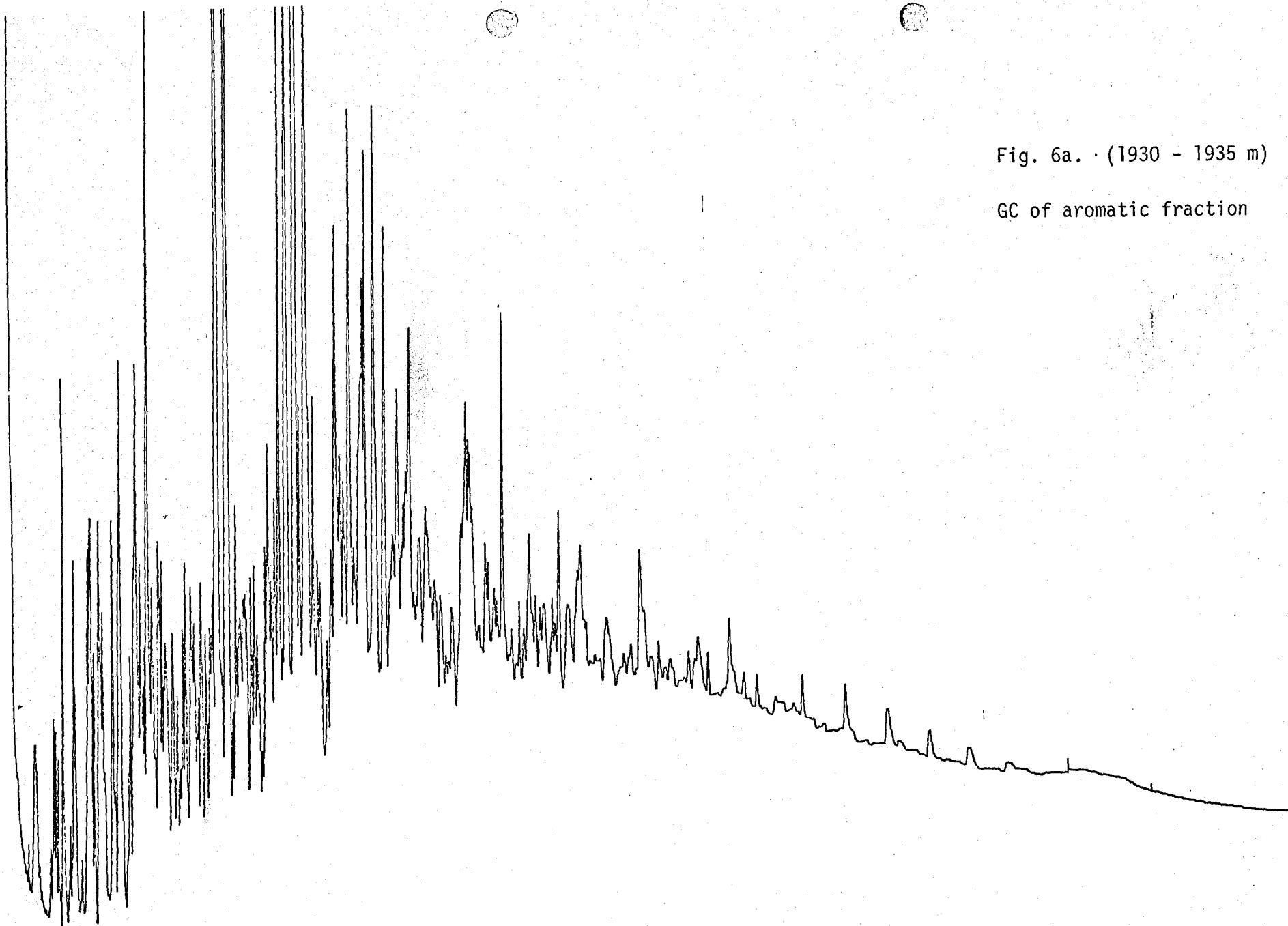


Fig. 6b: (1788 - 1792 m)

GC of aromatic fraction

