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# GEOCHEMICAL ANALYSIS OF TWO EXTRACTS FROM WELL 31/2-1, NORWAY

by

F.M. van der Veen and J. Weydema

Investigation 9.12.266

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# KONINKLIJKE / SHELL EXPLORATIE EN PRODUKTIE LABORATORIUM RIJSWIJK, THE NETHERLANDS

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# SUMMARY

Geochemical analysis has been carried out on the extracts of a consolidated core and of a loose sand sample from well 31/2-1.

Both extracts represent impregnations which are slightly bacterially degraded and have been expelled from source material mainly consisting of sapropelic organic matter with probably a slight contribution of land-plant waxes. This type of source material is probably similar to the source material of the Statfjord and Brent oils.

# GEOCHEMICAL ANALYSIS OF TWO EXTRACTS FROM WELL 31/2-1, NORWAY

# 1. INTRODUCTION

The purpose of geochemical typing of crude oils and rock extracts is to assess oil/oil- and oil/source rock correlation. In this context four objectives are of main interest:

- 1. to establish the type of source material from which certain crude oils or extracts originated.
- 2. to find in what type of environment a source rock has been deposited.
- 3. to estimate the maturity of the source material that has generated a certain oil or rock extract.
- 4. to determine whether or not a crude under investigation has been transformed (altered) after expulsion.

The following notes are intended as a guide to the geological interpretation of geochemical parameters. They are keyed to the tabulated results of the current study.

#### 2. SYNOPSIS OF GEOLOGICAL INTERPRETATION OF GEOLOGICAL PARAMETERS

#### **API** Gravity

The API gravity scale for oils is related to specific gravity by the following formula:

degrees API =  $\frac{141.5}{S.G. \text{ at } 60^{\circ} \text{ F}}$  - 131.5

Crude oils commonly range from 10-60° API. Gravity is mainly determined by the maturity of the source material at the time of oil expulsion and by the extent of alteration the oil has undergone (e.g. bacterial degradation, physical or thermal transformation).

## Extract (Ethyl Acetate)

Rock samples are crushed and powdered and subsequently extracted in a soxhlet apparatus using ethylacetate as a solvent. The extract, freed from solvent by evaporation, is used in further analyses.

# Organic Carbon after Extraction

In the extracted rock sample, after treatment with hydrochloric acid to remove carbonates, the insoluble organic carbon content is determined using a LECO instrument. It is generally accepted that an organic carbon content of at least 0.5% defines the lower limit for a source rock. However, this is somewhat arbitrary, dependent on the convertibility of the organic matter type into hydrocarbons and on the expulsion capability of the source beds.

#### Sulphur content

The sulphur content of a crude oil depends on:

- 1. the kerogen type (high or low sulphur) of the source rock, which is in turn related to its environment of deposition.
- 2. the level of organic metamorphism of the source rock at the time of expulsion.
- 3. the degree of transformation (bacterial or physical) of the crude.

The major part of the sulphur in crude oils is present in the heavy ends (higher boiling-point fractions). As bacterial degradation of a crude oil preferentially removes the light ends, the sulphur content of a crude is by mere concentration increased though usually to a not too significant extent. High-sulphur crudes are associated with sulphur-rich source material, deposited in strongly reducing environments (often rich in carbonates or cherts). Sulphur-rich crudes are often heavy, being expelled at a low maturity level. Low sulphur crudes are correlated with low sulphur source material, deposited either in non-marine environments or in marine siliciclastic sequences. Furthermore highly mature oils are always low in sulphur, regardless of their original source material.

#### Porphyrins

Porphyrins are nitrogen-containing ring components often found in petroleum as nickel or vanadyl complexes. It is generally accepted that the porphyrins are derived from chlorophyll during early diagenesis. A predominance of vanadyl over nickel porphyrins is associated with a deep marine environment of deposition, whilst nickel porphyrin predominance is linked with coastal or lagoonal waters with terrestrial influx.

#### Normal-Alkane Distribution

The total of saturated hydrocarbons of an oil (or rock extract) is separated by elution chromatography and then analysed by temperature-programmed gas chromatography. The n-alkane distribution of an oil displayed in the chromatogram provides information on its origin, maturity and possible transformation.

The shape of the n-alkane distribution reflects the original source material. The envelope of the alkane distributions of marine crude oils and source rock extracts are, for instance, concave, whereas landplant-related crudes and extracts usually show a convex or even bimodal n-alkane distribution.

Sometimes there is a marked predominance of odd-numbered n-alkanes over the even ones. This odd/even predominance (expressed as a 'carbon preference index') is often used as an index of maturity. A distinct odd/even predominance in the  $C_{25+}$  region is indicative of a landplant wax contribution in the source material while oils and extracts of marine origin do not exhibit such odd/even predominance. Biodegraded oils are characteristically deficient in n-alkanes. Severe bacterial degradation will result in a complete removal of n-alkanes and finally even of the isoprenoids (see Fig. 1).

#### Isoprenoid isoalkanes

Many crude oils and source-rock extracts contain a series of isoalkanes with structures based on the isoprene unit. They are believed to be derived from phytol, a hydrolysis product of chlorophyll. The most common isoprenoids in crude oil are pristane and phytane. The relative abundances of these two compounds, expressed as pristane/phytane ratio, pristane/n-C<sub>17</sub>, or phytane/n-C<sub>18</sub> is mainly an indication of the depositional environment of the source rock. High pristane/phytane and pristane/n- $C_{17}$  ratios are related to a swampy environment of deposition with low bacterial activity. Low ratios are expected in open aquatic conditions (marine or fresh water), where there is abundant bacterial activity.

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# C<sub>7</sub> Distribution

Crude oil samples are distilled to obtain the volatile fraction boiling below 120°C. This fraction is subsequently analysed by gas chromatography to obtain a detailed distribution of all  $C_7$  hydrocarbon isomers. A triangular plot of straight-chain (normal), polybranched, and monobranched  $C_7$  alkanes is used to distinguish slightly bacterially degraded or transformed crudes from their unaltered counterpart (Fig. 2). A plot of  $n-C_7$  alkanes - branched alkanes naphthenes is used to type the related source material and its environment of deposition (see Fig. 2). Note that this latter plot cannot be used for (even slightly) bacterially degraded crude oils. The relative abundances of  $C_7$  alkanes, naphthenes and aromatics may be used to determine whether waterwashing in the reservoir has occurred.

# Mass spectrometric analysis

# Parameters M1 and M2 (Fig. 3)

From the mass spectra of crude oils and extracts two parameters M1 and M2 can be derived, which are very useful for oil and source rock characterisation. The position in the triangular diagrams of M1 and M2 gives information about the original source material as is indicated in Fig. 3. Note that parameter M1 cannot be used for bacterially degraded crude oils.

# DOM of oils

The maturity of the oil and/or extract can be calculated from mass spectrometric data. The calculated maturity is expressed in DOM (degree of organic metamorphism) units, which cover the following ranges:

MATURITY ZONES
Immature
Mature for oil generation
Mature for gas generation. Post mature for oil generation.
Post mature for both oil and gas.

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\*STANDARD COMPOUNDS ADDED FOR IDENTIFICATION

BACTERIAL DEGRADATION DISPLAYED IN GAS CHROMATOGRAMS OF SATURATED HYDROCARBONS.

FIG: 1.

# C,-ALKANE DISTRIBUTION

poly branched alk.



# C,-ALKANE/NAPHTHENE DISTRIBUTION

branched alk.



# C7 DISTRIBUTION FOR CHARACTERISATION OF RELATED SOURCE MATERIAL.

FIG: 2.



I LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER

II CRUDES OF MIXED ORIGIN

III CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

PARAMETERS M1 AND M2 TO TYPE RELATED SOURCE MATERIAL.

FIG.3

#### 3. RESULTS AND DISCUSSION

The gas chromatograms of the saturated hydrocarbons (Figs. 4 and 5) indicate that both impregnations are possibly slightly bacterially degraded. Due to this bacterial degradation the pristane/ $n-C_{17}$  alkane ratios and the M1-parameters are considered not reliable, so that the environment of deposition cannot be assessed.

The M2 parameter data (Fig. 6) and the gas chromatograms of the saturated hydrocarbons (Figs. 4 and 5) point to expulsion from source material mainly consisting of sapropelic organic matter with probably a slight contribution of land-plant waxes.

The position of the M2 diagram (Fig. 6) is identical to those, of Statfjord and Brent oils (Fig. 7). However, the samples under investigation show a slight difference in n-alkane predominance in the  $C_{23}$  + range as compared to Statfjord and Brent oils, possibly indicating a slightly different facies.

The shape of the gas chromatograms (Figs. 4 and 5) indicates expulsion from mature source mateiral. This is in agreement with the "dom of oil" values of 64 and 63.

## 4. CONCLUSION

Both slightly bacterially degraded oils have probably been expelled from the same or similar type of source material as the Statfjord and Brent oils.

Sample	Denth	artificial	% et.ac. extract	% C <sub>org</sub> ex after C extr.	extr.	% S	porphyrins		pr	_pr_	ph	Parameter M1			Parameter M2			лом
Sampie	Depen	for			Corg	0	V ppm	Ni ppm	ph	<sup>nC</sup> 17	<sup>nC</sup> 18	A	В	С	P	Q	R	of oil
31/2-1 sand sample			1.4			0.6	2	<1	2.0	1.2	0.7	34	49	17	23	52	25	64
31/2-1 core sample			2.7			1.1	2	<1	2.1	1.3	0.7	34	44	22	19	50	31	63

Table 1 - Geochemical data of the impregnations

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lable Z - Geochemical data of Brent and Statijord of
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						<u></u>				C7-distribution					Mass spectometry						
	•								°7 <sup>.</sup>	-alkane	2	C <sub>7</sub> -alk/napth.	C <sub>7</sub> -a	lk/napth/arom	Para	meter	M1	Par	ameter	M2	
Samp.	Depth	API	Spec. grav.	c. % w. v. boil. <120C	% sulph	% 11ph. <u>pr</u> ph	nC <sup>pr</sup> <sub>17</sub>	<u>ph</u> nC <sub>18</sub>	nC <sub>7</sub>	mono br.	poly br.	nC <sub>7</sub> napth. br.	nC <sub>7</sub>	napth. arom	A	B	C	P	Q	R	DOM of oil
211/ 23-2	9249- 9269 ft				0.7	1.2	0.9	0.9				<u></u>			40	41	19	17	51	32	63
211/ 29-2	8713- 9058 ft				-	1.4	0.8	0.7							44	39	17	21	53	26	65
33/9-9	DST-3				0	1.4	0.7	0.6							44	41	15	23	54	23	70
33/12-1	DST-3				0.3	1.5	0.8	0.6							47	37	16	24	51	25	67





I LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER

- II CRUDES OF MIXED ORIGIN
- III CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

# LEGEND

□-31/2-1A SAND □-31/2-1A CORE

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- LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER
- I CRUDES OF MIXED ORIGIN

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II CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

LEGEND

OILS BRENT FIELD

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# GEOCHEMICAL ANALYSIS OF CORE IMPREGNATIONS FROM WELL 31/2-1, NORWAY by

F.M. van der Veen & J. Weijdema

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6. Gas chromatogram of the saturates, core 5, 1483.47	m 13
7. Gas chromatogram of the saturates, core 11, 1573.19	m 14
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12. Parameters M1 and M2 for Statfjord and Brent oils	19

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Geochemical extract analysis has been carried out on core samples from the gas and oil bearing zones in well 31/2-1.

The impregnations appear very similar. They are slightly bacterially degraded, mature expulsion products originating from sapropelic organic source matter with possibly a slight contribution of land plant waxes.

The parent source material resembles that of the Brent and Statfjord oils though the facies may have been somewhat different.

# GEOCHEMICAL ANALYSIS OF CORE IMPREGNATIONS FROM WELL 31/2-1, NORWAY

# 1. INTRODUCTION

The purpose of geochemical typing of crude oils and rock extracts is to assess oil/oil- and oil/source rock correlation. In this context four objectives are of main interest:

- 1. to establish the type of source material from which certain crude oils or extracts originated.
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The following notes are intended as a guide to the interpretation of geochemical parameters. They are keyed to the tabulated results of the current study.

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# API Gravity

The API gravity scale for oils is related to its specific gravity by the following formula:

# degrees API = $\frac{141.5}{5.6. \text{ at } 60^{\circ} \text{ F}}$ - 131.5

Crude oils commonly range from 10-60° API. The specific gravity is mainly determined by the maturity of the source material at the time of oil expulsion and by the extent of alteration the oil has undergone (e.g. bacterial degradation, physical or thermal transformation).

#### Extract (Ethyl Acetate)

Rock samples are crushed and powdered and subsequently extracted in a soxhlet apparatus using ethylacetate as a solvent. The extract, freed from solvent by evaporation, is used in further analyses.

# Organic Carbon after Extraction

In the extracted rock sample the organic carbon content is determined using a LECO instrument. It is generally accepted that an organic carbon content of at least 0.5% defines the lower limit for a source rock. However, this is somewhat arbitrary, dependent on the convertibility of the organic matter type into hydrocarbons and on the expulsion capability of the source beds. In this respect only the percentage of pyrolysable organic matter is of interest.

#### Sulphur content

The sulphur content of a crude oil depends on:

- 1. the kerogen type (high or low sulphur) of the source rock, which is in turn related to its environment of deposition.
- 2. the level of organic metamorphism of the source rock at the time of expulsion.
- 3. the degree of transformation (bacterial or physical) of the crude.

The major part of the sulphur in crude oils is present in the heavy ends (higher boiling-point fractions). As bacterial degradation of a crude oil preferentially removes the light ends, the sulphur content of a crude is increased by mere concentration though usually to a not too significant extent. High-sulphur crudes are associated with sulphur-rich source material, deposited in strongly reducing environments (often rich in carbonates or cherts). Sulphur-rich crudes are often heavy, being expelled at a low maturity level. Low sulphur crudes are related to low sulphur source material, deposited either in non-marine environments or in marine siliciclastic sequences. Furthermore oils expelled at a high level of organic metamorphism of the source rock are always low in sulphur, regardless of their original source material.

# Porphyrins

Porphyrins are nitrogen-containing ring components often found in petroleum as nickel or vanadyl complexes. It is generally accepted that the porphyrins are derived from chlorophyll during early diagenesis. A predominance of vanadyl over nickel porphyrins is associated with a deep marine environment of deposition of the source matter, whilst nickel porphyrin predominance is linked with coastal or lagoonal waters with terrestrial influx.

## Normal-Alkane Distribution

The saturated hydrocarbons of an oil (or rock extract) are separated by elution chromatography and then analysed by temperature-programmed gas chromatography. The n-alkane distribution of an oil displayed in the chromatogram provides information on its origin, maturity and possible transformation.

The shape of the n-alkane distribution reflects the original source material. The envelope of the n-alkane distributions of marine crude oils and source rock extracts are, for instance, concave, whereas landplant-related crudes and extracts usually show a convex or even bimodal n-alkane distribution.

Sometimes there is a marked predominance of odd-numbered n-alkanes over the even ones. This odd/even predominance (expressed as a 'carbon preference index') is often used as an index of maturity. However, this can be done in only a few specific cases. Indeed a distinct odd/even predominance in the  $C_{25+}$  region is indicative of a landplant wax contribution in the source material while oils and extracts of marine origin do not exhibit such odd/even predominance. Biodegraded oils are characteristically deficient in n-alkanes. Severe bacterial degradation will result in a complete removal of n-alkanes and finally even of the isoprenoids (see Fig. 1).

#### Isoprenoid isoalkanes

Many crude oils and source-rock extracts contain a series of isoalkanes with structures based on the isoprene unit. They are believed to be derived from phytol, a hydrolysis product of chlorophyll. The most common isoprenoids in crude oil are pristane and phytane. The relative abundances of these two compounds, expressed as pristane/phytane ratio, pristane/n- $C_{17}$ , or phytane/n- $C_{18}$  is mainly an indication of the depositional environment of the source rock. High pristane/phytane and pristane/n- $C_{17}$  ratios are related to a swampy environment

of deposition with low bacterial activity. Low ratios are expected in open aquatic conditions (marine or fresh water), where there is abundant bacterial activity.

# C<sub>7</sub> Distribution

Crude oil samples are distilled to obtain the volatile fraction boiling below 120°C. This fraction is subsequently analysed by gas chromatography to obtain a detailed distribution of all  $C_7$  hydrocarbon isomers. A triangular plot of straight-chain (normal), monobranched, and polybranched  $C_7$  alkanes is used to distinguish slightly bacterially degraded or transformed crudes from their unaltered counterpart (Fig. 2). In a plot of  $n-C_7$  alkanes - branched alkanes naphthenes oils of similar origin form clusters, while also some information is obtained from this triangular plot about the environment of deposition of the related source rocks (see Fig. 2). Note that this latter plot cannot be used for (even slightly) bacterially degraded crude oils. The relative abundances of  $C_7$  alkanes, naphthenes and aromatics may be used to determine whether waterwashing in the reservoir has occurred.

# Mass spectrometric analysis

## Parameters M1 and M2 (Fig. 3)

From the mass spectra of crude oils and extracts two parameters M1 and M2 can be derived, which are very useful for oil and source rock characterisation. The positions in these triangular diagrams give information about the original source material as is indicated in Fig. 3. Note that the M1 diagram cannot be used for bacterially degraded crude oils.

## DOM of oils

The maturity of the oil and/or extract can be calculated from mass spectrometric data. The calculated maturity is expressed in DOM (degree of organic metamorphism) units, which cover the following ranges:

DOM	MATURITY ZONES
<60	Immature
60-75	Mature for oil generation
79-92	Mature for gas generation. Post mature for oil generation.
>92	Post mature for both oil and gas.



\*STANDARD COMPOUNDS ADDED FOR IDENTIFICATION

BACTERIAL DEGRADATION DISPLAYED IN GAS CHROMATOGRAMS OF SATURATED HYDROCARBONS.

# C,-ALKANE DISTRIBUTION

# C,-ALKANE/NAPHTHENE DISTRIBUTION

poly branched alk.



branched alk.



# C7 DISTRIBUTION FOR CHARACTERISATION OF RELATED SOURCE MATERIAL.

FIG: 2



LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER CRUDES OF MIXED ORIGIN

III CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

PARAMETERS M1 AND M2 TO TYPE RELATED SOURCE MATERIAL.

FIG.3

11

# 3. RESULTS AND DISCUSSION

Geochemical extract analysis has been carried out on core samples from gas and oil bearing zones, well 31/2-1, viz:

- 8

core	1	1453.5	m	gas zone
core	4	1471.20	m	gas zone
core	5	1483.47	m	gas zone
core	11	1573.19	m	(residual-) oil zone
core	12	1581.	m	(residual-) oil zone
core	13	1605.5	m	(residual-) oil zone
core	13	1612.54	m	(residual-) oil zone

Results are displayed in Table 1 and Figs. 4-11.

The amounts of ethyl acetate extract are typically lower for the core samples from the gas zone as compared to those from the oil zone.

The gas chromatograms of the saturated hydrocarbons (Figs. 4-10) indicate that the core impregnations are slightly bacterially degraded. The bacterial degradation is found to level off somewhat at increasing depth as shown by the lower pristane/n- $C_{17}$  and phytane/n- $C_{18}$  ratios for core 13 at 1601.5 m and 1612.5 m.

As a consequence of bacterial degradation the pristane/n- $C_{17}$  ratios and the M1 parameters cannot be considered reliable so that e.g. the environment of deposition of the parent source rock cannot be assessed.

The M2 parameter data (Fig. 11) and the gas chromatograms (Figs. 4-10) point to a parent source material that mainly consists of sapropelic organic matter with possibly a slight contribution of land-plant waxes.

The shape of the gas chromatograms (Figs. 4-10) indicates expulsion from mature source material. For the sample from core 13, 1601.5 m, the level of maturity seems somewhat lower, as also indicated by its 'DOM of oil' value.

The cluster of M2 parameters for the 31/2-1 samples is positioned close to but not coinciding with the cluster of Brent and Statfjord oils<sup>1,2</sup>, see Fig. 12. This might point to a common type of source rock though with some difference in facies as also borne out by a slight difference in odd/even predominance of  $C_{23}^{+}$  n-alkanes and a somewhat higher pristane ratio observed for the 31/2-1 samples as compared to 'Brent and Statfjord oils'.

#### 4. CONCLUSIONS

Impregnations from core samples taken from the gas zone and the oil zone in well 31/2-1 appear very similar.

The impregnations are slightly bacterially degraded, mature expulsion products originating from sapropelic organic source matter with possibly a slight contribution of land-plant waxes.

The parent source material resembles that of the Brent and Statfjord oils though with somewhat different facies.

#### 5. REFERENCES

 van der Veen, F.M. & Weijdema, J., Geochemical analysis of two extracts from well 31/2-1, Norway.

RKER.80.008, February 1980.

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Sample	core 1 1453.5 m	core 4 1471.2 m	core 5 1483.5 m	core 11 1573.2 m	core 12 1581 m	core 13 1601.5 m	core 13 1612.5 m
% ethylacetate extract	0.1	0.1	0.2	1.8	1.0	0.7	0.6
% chloroform extract after ethylacetate extraction	0.01	0.01	0.01	<0.01	0.02	0.01	0.01
% organic carbon after extraction	0.1	0.1	0.0	1.1	1.2	0.6	0.2
% sulphur	1.2	2.9	0.8	0.7	0.7	1.0	0.7
ppm V <sup>o</sup> porphyrins ppm Ni porphyrins	<1 <1	<1 2±1	<1 2±1	<2 <1	<2 <1	<5 <2	<1 <1
pristane/phytane pristane/nC <sub>17</sub> phytane/nC <sub>18</sub>	1.8 2.5 1.5	2.0 2.3 1.2	2.0 2.7 1.4	2.1 2.5 1.2	2.2 2.3 1.3	1.9 1.5 0.8	1.8 1.3 0.7
Parameter M1 A B C	39 42 19	47 35 18	40 43 17	35 47 18	43 41 16	35 45 20	37 43 20
Parameter M2 P Q R	27 49 24	29 48 23	30 44 26	26 49 25	26 51 23	27 49 24	28 51 21
Dom of oil	65	66	65	64	64	63	65
% saturates % aromatics % heterocompounds	59 32 9	54 31 15	53 35 10	49 37 14	51 37 12	50 37 13	50 37 13

Table 1 - Geochemical data of impregnations from well 31/2-1

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31/2-1 CORE 1 1453.5 M



31/2-1 CORE 4 1471.20 M





GAS CHROMATOGRAM OF SATURATED HYDROCARBONS 31/2-1 CORE 5 1483.47 M





GAS CHROMATOGRAM OF SATURATED HYDROCARBONS 31/2-1 CORE 11 1573.19 M



31/2-1. CORE 13 1601.5 M

FIG. 10



GAS CHROMATOGRAM OF SATURATED HYDROCARBONS 31/2-1 CORE 13 1612.54 M



LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER CRUDES OF MIXED ORIGIN

CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

LEGEND

<sup>□</sup> - 31/2-1 1453.5 m. core 1 O- 31/2-1 core 4 1471.20 m. A - 31/2-1 1483.47 m. core 5 +-31/2-1° core 11 1573.19 m. X - 31/2-1 core 12 1581 m. ♦ - 31/2-1 core 13 1601.5 m. ↑ - 31/2-1 core 13 1612.54 m.

FIG. 1

11



LANDPLANT-DERIVED CRUDES WITH SUBSTANTIAL RESIN CONTRIBUTION TO SOURCE MATTER CRUDES OF MIXED ORIGIN CRUDES DERIVED FROM SOM AND/OR ALGAL MATTER

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FIG.

12

LEGEND

STATFJØRD AND BRENJ ØILS

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