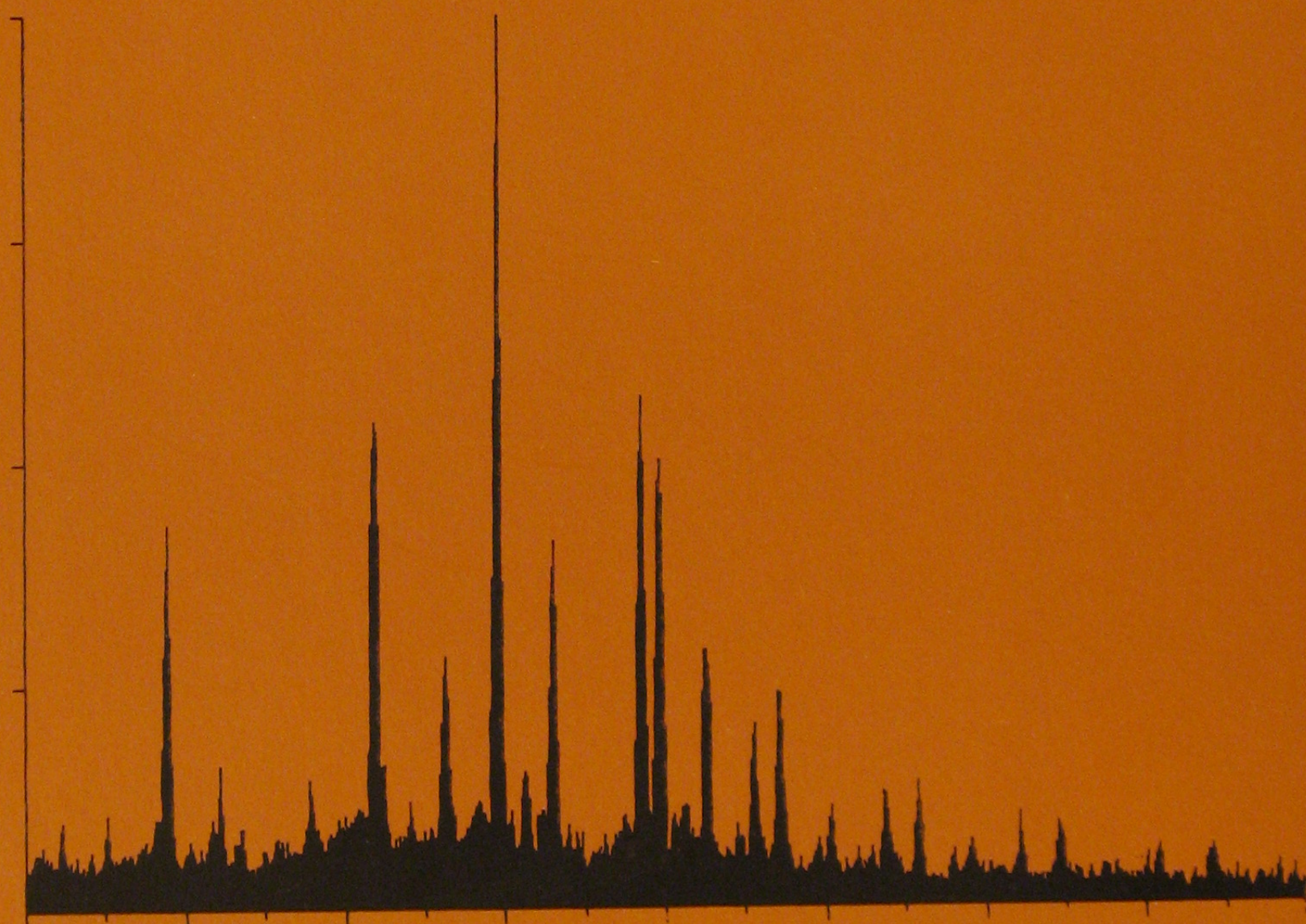


# A Source Rock evaluation of the Mesozoic Sediments of the Well Hyllebjerg-1 Danish Subbasin

By Birthe J. Schmidt



**DGU**

Geological Survey of Denmark  
Ministry of the Environment

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**ABSTRACT**

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The source rock potential of Mesozoic sediments (cuttings) from the Hyllebjerg 1 well, Danish Subbasin, has been assessed using a number of different petrographical and organochemical methods.

Upper Jurassic sediments (Bream Formation) equivalent to the principal source rocks of the North Sea graben structures (Kimmeridge Clay Formation and lateral equivalents) do not show similar prominent source rock characteristics in this well, although a higher proportion of algal material is observed.

Sediments with the most promising source rock characteristics for liquid hydrocarbons were detected mainly in the lower Jurassic sequences of the upper Fjerritslev Formation (F-4 and upper F-3 Member) and in one horizon in the Upper Cretaceous Vedsted Formation which showed a good quality composition and a relatively high content of organic matter. But these sediments may be excluded as actual source rocks in this well as maturity (assuming the threshold value near 0.60 % R) is first reached at approximately 8500' depth i.e. at the top of the Gassum Formation (Upper Triassic/Lower Jurassic). The conditions may only be slightly different off - structure is this area, as the F-4 and F-3 Member sequence according to seismic sections is found at approximately the same depth. But the depth to (and the thickness of) the Fjerritslev Formation is increasing towards the SE into the rimsynclines of the salt domes nearby. While sufficient maturity is reached in the deeper part of the well, no commercial accumulations of hydrocarbons were encountered. This is attributed to the mainly reworked, unfavourable type of organic matter and the generally decreasing organic content downwards in the well, approaching

the lower limit for potential source rocks (set at 0,5% TOC). However, generation and migration of small amounts of gaseous hydrocarbons from Gassum Formation sediments containing more humic-influenced organic matter with only minor reworking cannot generally be excluded either here or elsewhere in the basin. Some more attention should also be paid to the Vinding Formation sediments which contain some algae-rich (Botryocous-type) oil-prone horizons of more favourable source rock conditions.

Mature sediments are found at shallower depths (8500') in this well in the central part of the basin compared to the more marginal areas (8900') where a slightly higher geothermal gradient in Hyllebjerg 1 ( $28^{\circ}\text{C}/\text{km}$  uncorrected) is seen compared with the marginal areas ( $23.5^{\circ}\text{C}/\text{km}$  uncorrected) away from the basinal depocenter. The basinal depocenter also has a higher heat flow.

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## INTRODUCTION

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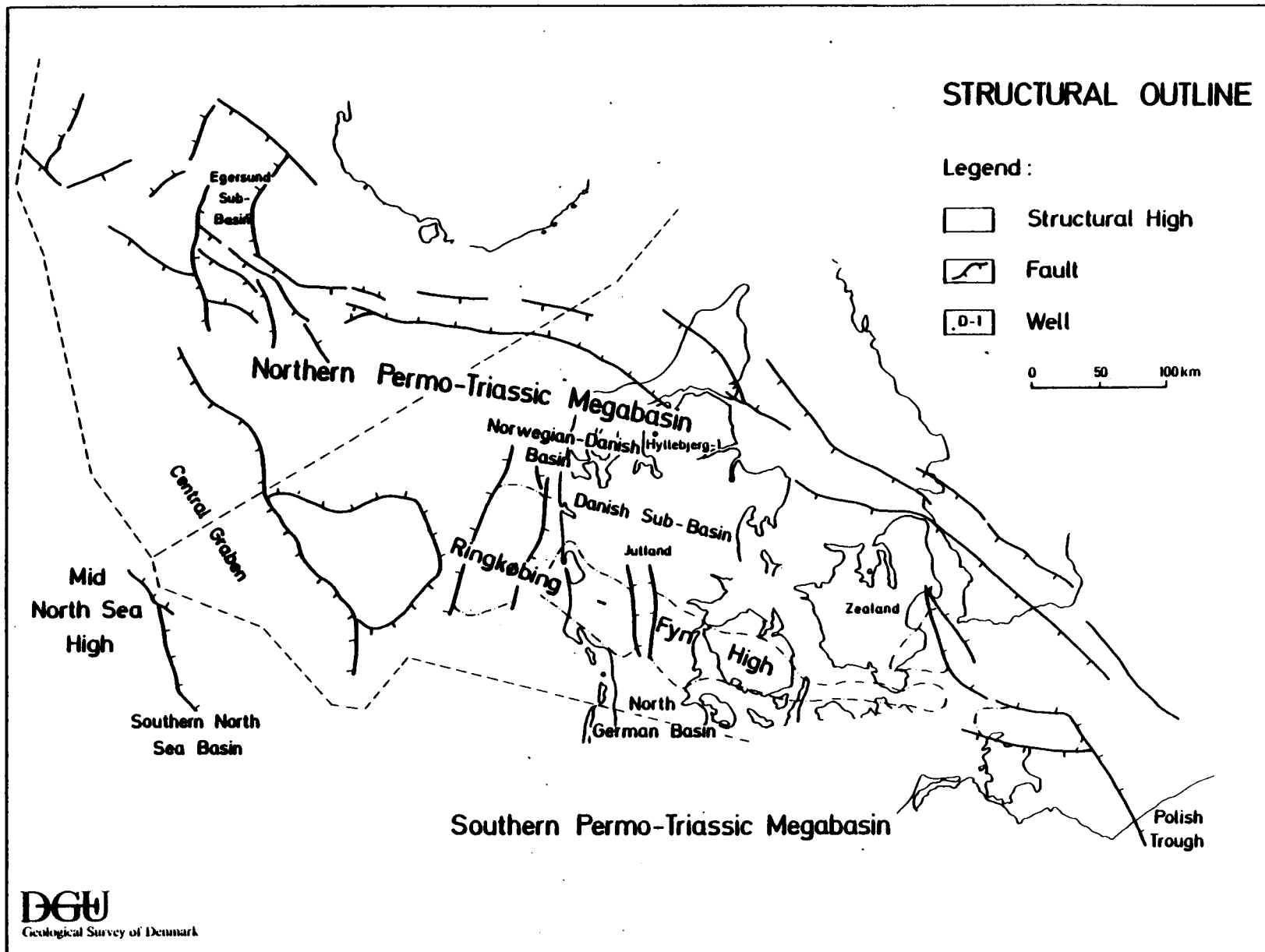
This investigation on the well Hyllebjerg 1 forms part of the larger EFP-85 project "Maturity analyses of the sediments in the Danish Subbasin". The purpose of the project is to investigate the source rock potential of the released deeper wells from the Danish off-shore area (outside the Central Graben), supplemented by a well from the central part of the on-shore area of the Danish Subbasin. Most of the other on-shore wells have been investigated and their oil/gas potential has been estimated (Thomsen et al. 1983, Thomsen et al. 1987).

With financial support for 6 months work initially granted by the Danish Ministry of Energy (EFP-projekt - aktst. 118, 1983) the investigation started in 1984 with a detailed analysis of the well Hyllebjerg 1. The results from Hyllebjerg 1 will contribute essentially to knowledge of the maturity conditions of the Mesozoic sequence of the central on-shore part of the Danish Subbasin, which is generally characterized by relatively high geothermal gradients and some sequences of potential hydrocarbon source rocks.

This report is based on the data available at the end of the short project period, supplemented with analyses performed at the Rheinisch-Westfälische Technische Hochschule in Aachen (F.R.G.) in the spring of 1985 (grant support by SNF) and completed under EFP-85.



Fig. 1 Location of Hyllebjerg 1



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## GEOLOGY

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The well Hyllebjerg 1 was drilled as an exploration well on a low relief collapse structure (solution of salt from Oddesund Formation) in 1975/76 to a depth of 2883 m (b. KB) in the central part of the Danish Subbasin (fig.1) It encountered thick sequences of Mesozoic sediments; 1485 m of Triassic, Jurassic and Lower Cretaceous sediments occurred in the investigated sequence.

The regional and stratigraphic aspects and environments of deposition in the Danish area are described in detail by Larsen (1966), Christensen (1974), Michelsen (1975, 1978), Bertelsen (1978, 1980) and Koch (1983). The lithostratigraphic division used here is based on wire line logs (DGU, unpublished results). Well data are published in the Well Data Summary Sheets vol. 2 (1981). The general lithostratigraphy showing the formations and members encountered in the investigation is seen in fig. 2.

# PRE-TERTIARY LITHOSTRATIGRAPHY

Cretaceous	Upper	Maastrichtian
		Campanian
		Santonian
		Coniacian
		Turonian
	Lower	Cenomanian
		Albian
		Aptian
		Barremian
		Hauterivian
Jurassic	Upper	Valanginian
		Ryazanian
		Volgian
	Middle	Kimmeridgian
		Oxfordian
		Callovian
		Bathonian
		Bajocian
		Aalenian
		Lower
Pliensbachian		
Sinemurian		
Triassic	Upper	Hettangian
		Carnian
		Norian
	Middle	Rhaetian
		Ladinian
	Lower	Anisian
Lower	Scythian	
Permian		

Chalk Group	
Rødby Formation	
Vedsted Formation	
Bream Fm.	Frederikshavn Member
	Børglum Member
	Flyvbjerg Member
Haldager Sand	
Fjerritslev Formation	
Gassum Formation	
Skagerrak Form.	Vinding Formation
	Oddesund Formation
	Tønder Formation
	Falster Formation
	Ørslev Formation
Smith Bank Form.	Bunter Sandst. Formation
	Bunter Shale Formation
Zechstein Group	
Rotliegende Group	

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**MATERIALS.**

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Only cuttings were available from the Hyllebjerg 1 well; Samples were collected at approximately 60' intervals throughout the Mesozoic sequence.

Fig. 2 Generalized lithostratigraphy of the Danish Subbasin.  
(Michelsen 1987).



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## METHODS

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In this study, the source rock potential of the Mesozoic sediments was evaluated by different optical and chemical methods. Coal petrographic optical methods (e.g. reflectance measurements on huminite/vitrinite and spectral fluorescence measurements on extracted bitumens) were used for estimating the maturity of the sediments. Normal reflected light observations combined with fluorescence light techniques, were also used to provide information on the composition (type) of organic matter present. These optical methods were also used to assess the degree of reworking of the organic matter and the presence of cavings or other contaminants.

Combustion in an induction furnace was used to estimate the amount of organic matter. The maturity and type of organic matter was evaluated by several chemical parameters obtained from various analyses of the bitumen fraction (e.g. extraction data, gas chromatographic analyses (GC) and combined gas chromatographic - mass spectrometric analyses (GC-MS)).

Details of the analytical methods and instruments used are given in the appendix.



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## RESULTS AND DISCUSSION

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### Amount of organic matter

Total organic carbon (TOC) values are listed in table 1 (together with the dominant cuttings lithology) and shown in fig. 3. The relative amount of organic matter in the polished specimens was also assessed under the microscope during description of the organic matter composition (table 7).

Most of the samples were claystones/shales or coarser grained silts/sandstones; a few carbonaceous cuttings were included in the uppermost and lowermost samples.

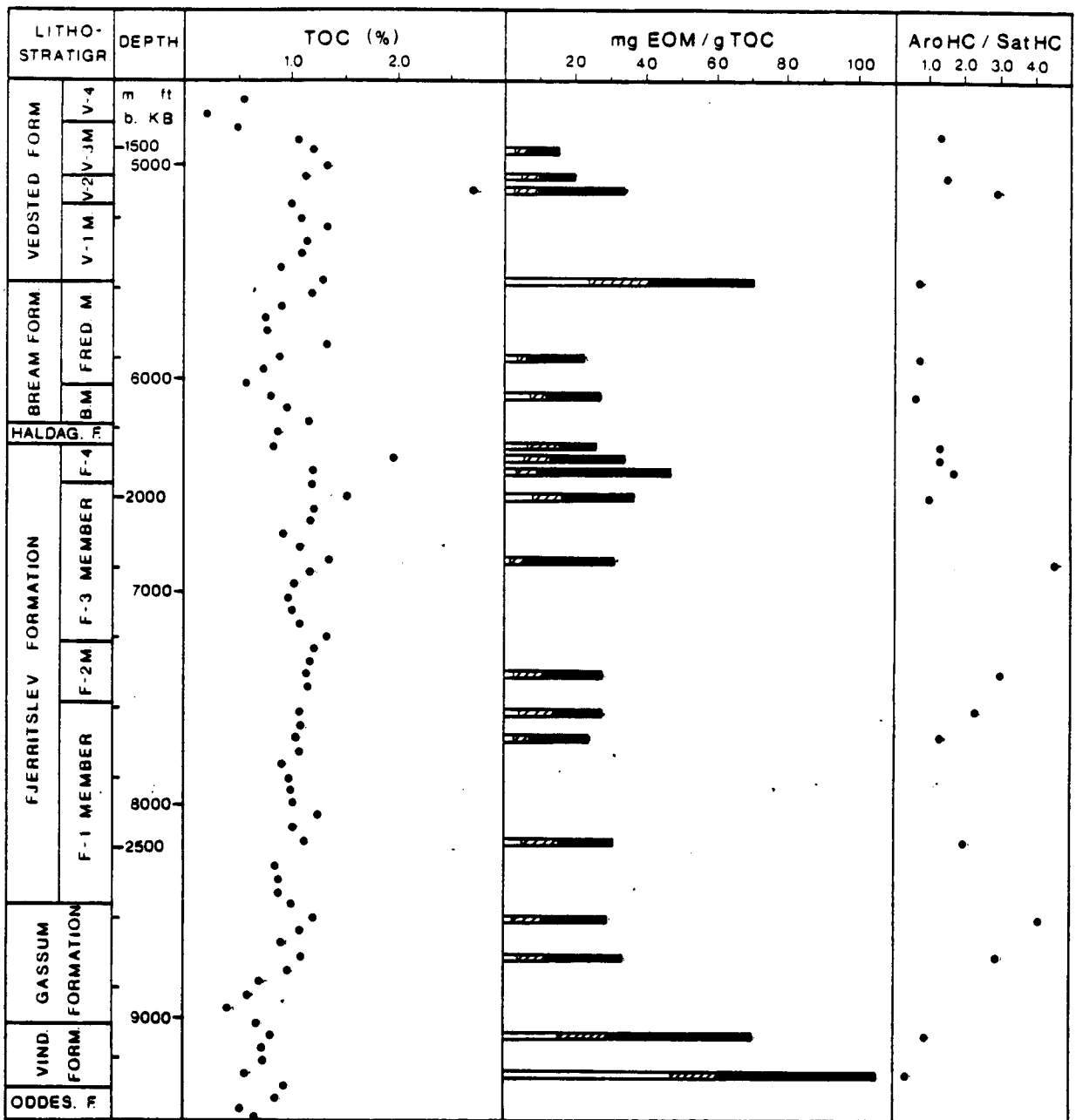
The total organic carbon values varied from 0.19 to 2.71 % wt for the various samples analysed with the lower values in the more sandy and carbonaceous samples. Most samples contained about 1% wt TOC, which is the average value for a "normal" shale (Gehmann 1962, Hunt 1972).

The uppermost 3 samples of the Vedsted Formation had rather low organic contents with values of about 0.5% wt TOC, in part caused by considerable contamination, especially by chalk cavings, especially in 910A and 913A. Some low values are also seen in samples from the Gassum, Vinding and Oddesund Formations with a slightly decreasing trend in TOC-values downwards. Mean values for hydrocarbon source rocks (shales/claystones) are in the range of 1.5-2.5% wt TOC (Tissot & Welte 1984). In the North Sea the range is 1-8% wt TOC (Thomsen et al. 1987).

Only few samples fall within the typical source rock range, as TOC-values > 1.5% wt are only recorded from a few upper Fjerritslev Formation samples and one anomalously organic-rich sample from the Vedsted Formation. While a few coaly particles are present in some of the sandy samples from the Haldager and Gassum Formations, no pronounced high TOC-values are recorded here.



HYLLEBJERG 1



Sat HC      Aro HC      Res + Asph

Sat HC - Saturated Hydrocarbons  
 Aro HC - Aromatic Hydrocarbons  
 Res - Resins  
 Asph - Asphaltenes

Fig. 3 Total organic carbon (TOC) and extractability values (EOM = extractable organic matter)

As minimum values are conventionally set at 0.5% wt TOC for shales and 0.3% wt TOC for carbonates to be potential source rocks (Tissot & Welte 1984), most of the samples meet this demand, but in some of the samples from the deeper part of this well the lower organic content may be critical for the hydrocarbon potential. However the hydrocarbon potential is only affected by the percentage of carbon which is hydrocarbon prone.

If the lower limit is set at 1% and 0.5% wt TOC for shales and carbonates respectively, as is done in many laboratories (Tissot & Welte 1984), the amount of organic matter seems generally too low in the sediments from the Vinding and Oddeund Formations for them to be potential source rocks, whilst in most of the other sediments investigated, TOC-values are close to this critical value.

Thus, on the basis of TOC-values, the hydrocarbon potential must be regarded as relatively low for most of the sediments in the investigated sequence, with the possible exception of the samples noted above.

### Composition of the organic matter

#### Description:

-----

Coal petrographic descriptions of the organic matter and evaluations of its composition are given in table 7. A short summary of the sediments from the different Formations is given below:

The Vedsted Formation is characterized by marine claystone and siltstone. The organic matter is generally dominated by pseudovitrinite with slightly increasing contents of liptinite (alginite) with depth. The uppermost samples 909A-913A are dominated by pseudovitrinite, often showing evidence of reworking and/or oxidation (oxidation rims and shrinkage-

cracks); only a few wisps of huminite/vitrinite and liptinite fragments are present. In 910A and 911A the composition tends to be masked by contamination. In 912A and 913A, some more coarse-grained huminite/vitritines are present. Samples 914A and 915A are still dominated by pseudovitrinite, but with an increased liptinite content (especially in 915A) and only a few small huminite/vitrinite fragments. Among the alginites some dinoflagellates can be identified. Sample 916A is very different from the other material examined from this formation. It is characterized by a very high and very fine grained organic content, mostly composed of liptinite, especially bituminite (associated with many small pyrite framboids) and a few alginites (dinoflagellates). The huminite/vitrinite grains are small and angular. There is a relatively high content of fine grained inertinite. The following samples 917A-922A resemble 914A and 915A.

The Bream Formation is characterized by low energy sediments (Børglum Member) overlain by high energy near-shore sediments (Frederikshavn Member). The organic matter is composed of about equal amounts of liptinite (most alginite) and pseudovitrinite, with only very low huminite/vitrinite contents. Samples 923A-926A have varying organic contents with the somewhat higher organic contents caused mainly by fine grained liptodetrinites and some alginites (dinoflagellates). In the samples 927A and 928A, the liptinite content is slightly increased and large alginite fragments (dinoflagellates) are dominant in 929A, especially in the coarse grained cuttings. Thereafter the grain size and organic content decrease with depth. In 930A-934A the liptinite is mostly fine grains, and more inertinite is observed.

The Haldager Formation is characterized by fluvial-deltaic sediments (Haldager Sand Member) overlain by marine heterolithic shallow water sediments (Flyvbjerg Member). The organic matter is composed of roughly equal amounts of liptinite and pseudovitrinite. Sample 935A resembles the overlying material as fine grained liptinites are prevalent,

but the identifiable liptinites show a somewhat more terrestrial composition (sporinite, bituminite, ?cutinite). Sample 936A (transition to the Fjerritslev Formation) is similar to 935A, but also contains some small angular huminite/ vitrinite particles (a few of them are very large).

The Fjerritslev Formation is divided into four members characterized by marine claystone and shale, representing changing energy environments. The organic matter is dominated by fine grained liptinite (most bituminite and liptodetrinites, some alginite and sporinite) in the upper section (F-4 and upper part of F-3 Members) but the content decreases with depth. However the pseudovitrinite and huminite/vitrinite proportions increase with depth, with pseudovitrinite dominating the lower part of the section (especially in the F-1 Member). In the lowermost part of the Formation, the organic matter becomes coarser, indicating the transition to the Gassum Formation.

The organic content is relatively high in the uppermost of the samples 937A-947A and is dominated by liptinite. In 937A pseudovitrinite and huminite/vitrinite particles still make up a considerable proportion of the organic matter, but their amount clearly decreases in the following samples as the quantity of liptinite increases. The liptinite is very fine grained and composed of many bituminite streaks (with dull orange-brown fluorescence and small greenish fluorescing ?algal-inclusions) associated with small pyrite framboids, some sporinite and some large alginites (Tasmanites) and fragments of alginite. In some of the samples (941-944A) most of the organic matter is composed of bituminite. Slightly varying amounts of alginites are present in all samples. Tasmanites have been observed only in samples 937A, 938A, 940A and 945A. The contents of pseudovitrinite and huminite/vitrinite are again enhanced in 946A and 947A which contain more coarse grained organic matter. Sample 948A-950A are rich in pseudovitrinite with some coarse grained huminite/vitrinite (with cellular structure) whilst the liptinite content is low. The liptinite generally fluoresces dull orange, suggesting some reworking.

In samples 951A-967A pseudovitrinite is generally dominant with less huminite/vitrinite and varying, but small, amounts of liptinite. In 952A-956A almost no liptinite (mostly small unidentifiable liptodetrinites) is observed. In some of the samples (957A, 958A) a few Tasmanites are present, probably in ?cavings. True huminite/vitrinite grains are usually sparse, but are slightly more abundant (coarse grained with recognizable cellular structure) in 954A-956A. In 957A and deeper sample the pseudovitrinite proportion clearly dominates with many grains showing evidence of reworking and/or oxidation (oxidation rims and shrinkage cracks). These samples are also characterized by some small splinters of inertinite, some containing sclerotinites (especially 963A-966A).

In 968A and 969A highly mixed cuttings are observed with compositions like the samples above and below. In 970A-972A (the latter sample belongs to the Gassum Formation) a coarsening of the organic particles composed of about equal amounts of pseudovitrinite and huminite/vitrinite is seen (angular and gnarled) while the liptinite content is very low.

The Gassum Formation is characterized by deltaic - shallow water littoral sand- and siltstones interbedded with claystones. The content of the organic matter fluctuates with coarse-grained pseudovitrinite and huminite/vitrinite particles, low liptinite (terrestrial marker) contents and a few genuine coal grains. There is a decreasing organic content downwards.

Samples 973A-977A are somewhat variable. The upper ones are not essentially different from the overlying samples, but a few genuine coal grains (composed of desmocollinite, corpocollinite, sporinite - 973A) are observed and a low, but more varied content, of terrestrial liptinites, mostly sporinite and bituminite (in some samples (973A, 976A) resinite and cutinite are also observed). In 975A many carbonaceous cuttings are present with angular huminite/vitrinite and pseudovitrinite particles present. In

978A and 979A highly mixed lithologies with some coarse grained cuttings of very low organic content are observed. A low content of organic matter is also observed in 980A and 981A consisting mainly of pseudovitrinite and few angular huminite/ vitrinite particles. There is almost no liptinite present.

The Vinding Formation is characterized by shallow brackish-marine deposits of claystone and limestone. The organic matter is characterized by a generally low organic content, mostly of liptinite, liptodetrinites and alginite of Botryococcus type). Unfortunately only a few samples were available for analysis.

In 982A some small huminite/vitrinite and pseudovitrinite grains are seen together with some liptinite (liptodetrinites and sporinite). In 984A and 985A the low organic content is largely dominated by liptinite; in 984A mostly as liptodetrinites and in 985A as alginite of Botryococcus-type. Botryococcus (bright yellow fluorescence) are especially numerous in a few cuttings, whilst in others the Botryococcus algae show yellow-orange fluorescence and their tousled appearance may be due to reworking and/or oxidation.

The Oddesund Formation is characterized by continental and brackish water claystones and siltstones with carbonates and evaporites. There is only a very low organic content. The general composition in 987A-989A is difficult to estimate because of the very low content and finegrained nature of the organic matter.

Hydrocarbon potential:

-----  
 The composition of the organic matter is affected by the depositional environment of the sediments. Land-derived organic matter is generally found in near-shore areas, whilst phytoplankton represent the main source of organic matter in marine sediments, as noted by many authors (e.g. Dow 1977b, Brooks 1981, Huc 1980, Tissot & Welte 1984,

Glennie 1984). This is also valid for the composition of the organic matter encountered in the Danish Subbasin (Lindgreen & Thomsen 1982, Schmidt 1985) though a relatively high input of terrestrial material, with an obvious reworked appearance, is present in many of the marine sediments of the Mesozoic. This is especially seen in the Vedsted Formation (except for the sample 916A) and the deeper part of the Fjerritslev Formation (F-1, F-2 and deeper F-3 Members). Here the organic matter is dominated by extensively reworked and oxidized pseudovitrinite with only a minor contribution of liptinite. As most of the pseudovitrinite may be thermally inert, the hydrocarbon potential is considered to be low for these sediments.

This may to some degree also be true for the Bream Formation sediments, but the somewhat higher content of liptinites (the small liptodetrinites may be derived from phytoplankton) may increase the hydrocarbon potential as algal material normally has a good potential for oil. The Bream Formation sediments are equivalent to the "black shales" (very organic rich, composed of algal and bituminous organic material) of the Kimmeridge Clay Formation/ Børglum Member in the Graben structures of the North Sea, where they provide the dominant source rock for oil in the central and northern North Sea (Barnard & Cooper 1981, Cornford 1984). However, in the equivalent sediments in this well no comparably favourable organic matter compositions were detected.

Too little information is available from the Haldager Formation to provide a general characterisation of its hydrocarbon potential as only one sample without pronounced organic content was available.

The sediments of the upper part of the Fjerritslev Formation sediments (F-4, upper F-3 Member) with a high organic content dominated by bituminite and alginite have one of the most promising source rock potentials of the sediments investigated. Generally, bituminite (often considered to be bacterial lipids and decomposition products of algae and faunal plankton (Teichmüller 1982)) and alginite are consi-

dered to be good precursors for oil. Bituminite, together with alginite, are the predominant macerals of oil source rocks and oil shales (Tissot & Welte 1984, Hutton et al. 1980). As the sediments investigated locally show these characteristics, they are considered to have a good oil potential (unless the bituminite is composed of degraded humic material of terrigenous origin which may lower the potential). This type of sediment thus indicates reducing depositional environments with good preservation conditions for the organic matter. This is in good agreement with the assumed depositional environment of the upper F-3 and F-4 Member sediments in deep water and lagoonal environments with strongly reducing conditions close to the bottom (Michelsen 1978).

Sample 916A from the Vedsted Formation is of the same sediment type (high organic content of bituminite with some alginite in association with a very high content of pyrite framboids). Thus a change during the lower Cretaceous from more or less "normal" marine conditions to more anoxic environments and the reverse seems to be indicated. The common occurrence of framboidal pyrite seems to attest to the action of sulphate reducing bacteria during this event.

In the Gassum Formation sediments, organic matter is characterized by a higher input of terrestrial matter with only minor signs of reworking. Also the liptinite composition shows a somewhat more terrestrial imprint to the organic matter. The humic composition indicates a major potential for gas whilst the liptinites present may contribute with oil or condensate of characteristic type if a sufficient maturity is reached.

Sediments of the Vinding Formation have a good potential for oil in some horizons as the organic matter is mainly composed of algal material of Botryococcus-type. But as the amount is estimated to be low, the hydrocarbon potential will be reduced.

The organic matter in sediments representing part of the Oddesund Formation reveals no clear evidence of its hydro-



carbon potential, but it is generally assumed to be low because of the low content of organic matter.

#### Vitrinite reflectance measurements

The results of the vitrinite reflectance measurements are presented in Table 2 and the reflectance trend for the well is illustrated in Fig. 4.

The measurements have generally been performed on very small wisps and splinters of huminite/vitrinite. Many of the samples contain low reflecting bituminous and "A"-types (Stach et al. 1975) unsuitable for rank determination and making the analysis problematic (see appendix).

Sample 973A from the Gassum Formation is used as a marker horizon as it contains a few coal particles which unambiguously define the maturity/rank of the sediment. In these coal particles desmocollinite and corprocollinite can be recognized and are used to estimate the lower and upper limit respectively of the realistic reflectance range. These results, together with measurements of the dispersed huminite/vitrinite in the same sample are shown in fig. 5. The range of the dispersed vitrinite particles is slightly broader than for the measurements on the coal particles.

Larger fragments of angular huminite/vitrinite are present in samples 936A and 975A, indicating autochthonous material.

As the reflectance measurements on these particles showed a unimodal distribution with relatively little scattering of the values, and many measurements were possible, these samples are also used as marker horizons. The reflectance range used for these samples proved to be in good agreement with the marker horizon values.

During the analyses much attention was paid to the lithology of the cuttings so as to avoid measurements on cavings or mud additives. The last casing was set in Cretaceous

HYLLEBJERG 1

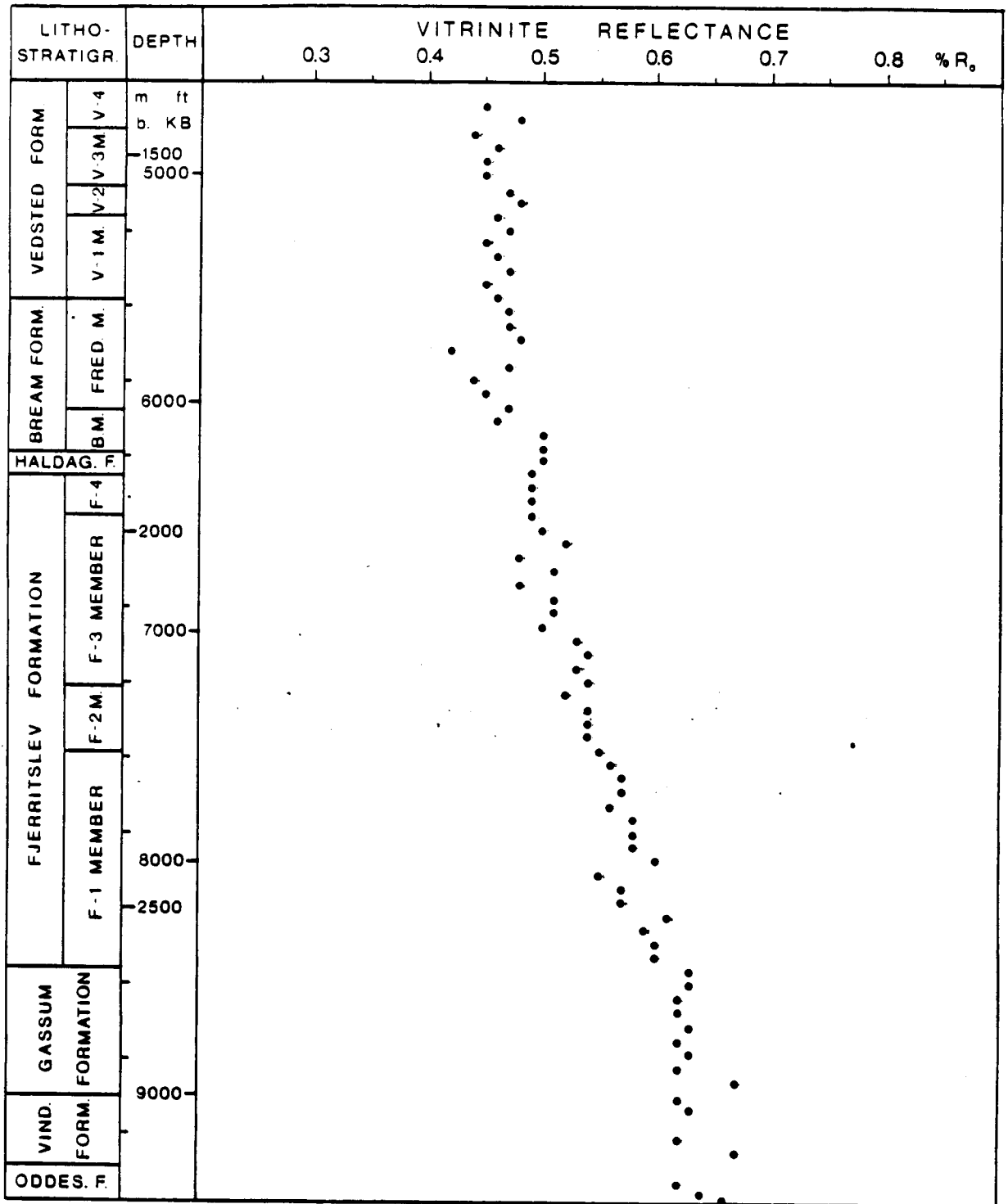


Fig. 4 Vitrinite reflectance measurements.

sediments at 3495 ft.b.KB (Well Data Summary Sheets 1981). The analyses were performed on the samples from the top downwards: Cuttings in samples with different lithology from the overlying one were chosen for measurements if they were considered to represent meaningful lithological change in the sequence, provided that the reflectance range is not significantly lower than in the previous samples. Since the investigated intervals often have almost identical lithologies, the possibility that material from overlying lower rank intervals have been included in the measurements cannot always be excluded. A sequence with rapidly changing lithology with low organic contents of often oxidized and/or reworked material and sometimes large amounts of contaminants (as is seen particularly in the deepest part of the well) is difficult to measure and gives less reliable results than the rest of the well.

All mean  $R_o$ -values are plotted against depth to illustrate the reflectance trend (fig. 4).

The maturity of the organic matter is dependent on time-temperature relationships. Maturation increases approximately linearly with time and exponentially with temperature and a semi-logarithmic plot of huminite/vitrinite values versus depth in a continuously subsiding basin will show a straight line i.e. the maturation profile, assuming a constant geothermal gradient (Dow 1977a).

Consequently, Least square regression analysis has been undertaken. Using this calculation method a very high degree of correlation is achieved ( $r=0.96$ ) and the following  $R_o$ -values are estimated: surface 0.29 %R, 1000 m: 0.38 %R, 2000 m: 0.51 %R, 3000 m: 0.68 %R and 4000 m: 0.90 %R which establishes the reflectance trend.

Reflectance measurements on corresponding Mesozoic sediments from wells outside the basinal depocenter of the Danish Subbasin show a steeper average maturation profile (less maturation per unit depth) than the Hyllebjerg 1 profile (see fig.6). This seems to be due to the slightly higher geothermal gradient in the central part of the basin

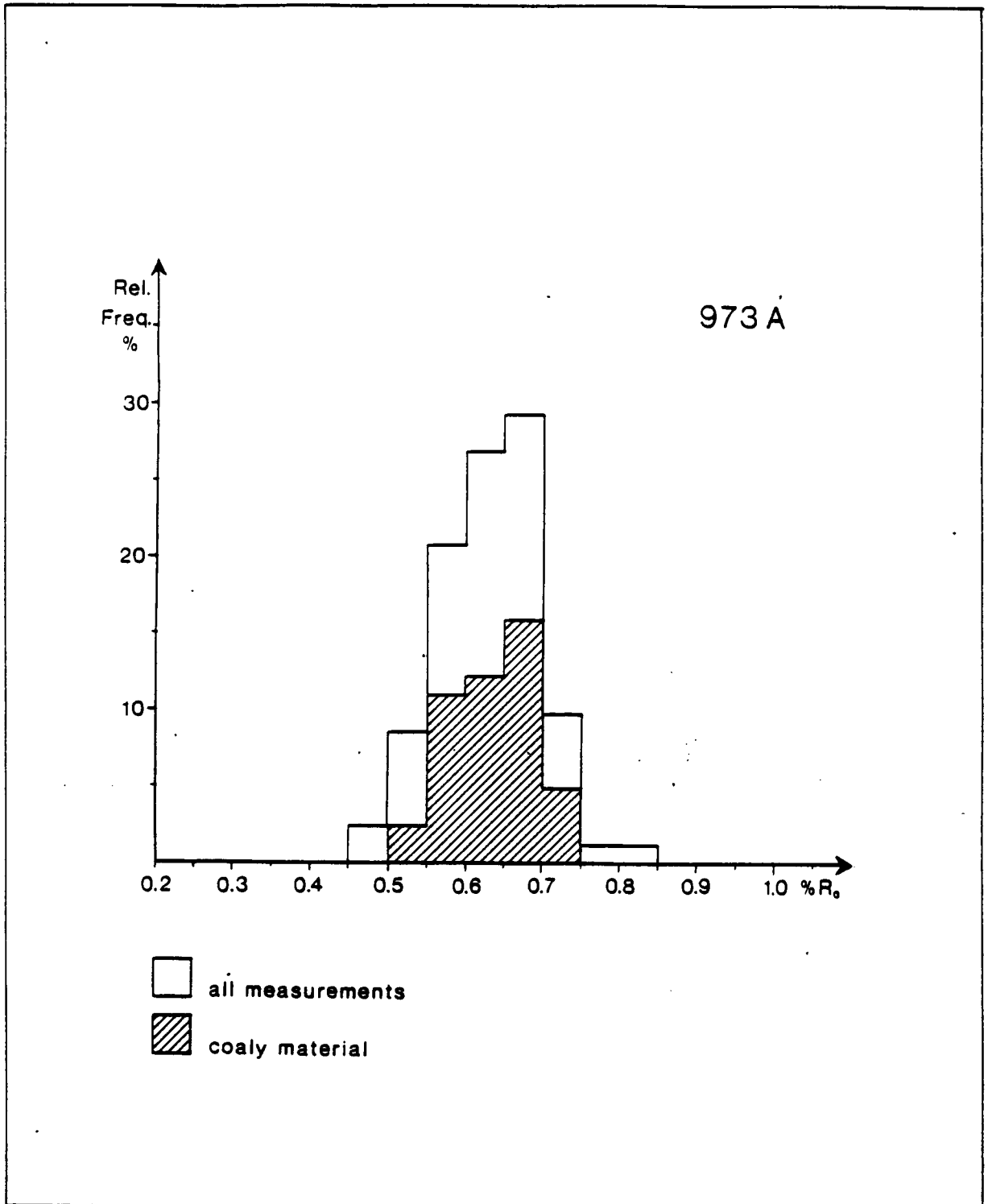


Fig. 5 Reflectogram of the marker horizon 973A, showing all reflectance measurements with measurements on the coaly material indicated (= shaded area).  $R_o = 0.63\%$ .

(Hyllebjerg 1:  $28^{\circ}\text{C}/\text{km}$  uncorrected) compared with the more marginal areas towards the Fennoscandian Border zone and above the Ringkøbing-Fyn-High where it is estimated to average  $23.5^{\circ}\text{C}/\text{km}$  uncorrected (Madsen 1975, 1978, Balling 1978, 1979, Michelsen et al. 1981). In the central part of the basin also a higher heat flow has been calculated (Balling 1979).

Mature sediments may thus be expected at shallower depths in Hyllebjerg 1 than in the other investigated wells outside the basinal depocenter.

The  $R_o$ -values lie between approximately 0.40 and 0.65 % $R_o$  and show a clear depth related trend from immature to slightly matured sediments, reaching 0.65 % $R_o$  at the terminal depth of the well (equivalent to the high volatile bituminous B/C coal rank). Conventionally 0.55 % $R_o$  is used as a threshold value for the onset of oil generation (world-wide accepted "average"), but different maturity levels exist for different types of organic matter with varying rates of transformation with increasing temperature (Tissot & Welte 1984, Vandenbrouke 1980) (see fig. A-1). A maturation level of 0.50 % $R_o$  is reached at a depth of approximately 6400', i.e. at the top of the Fjerritslev Formation. Thus the sediments from the upper part of the well, including Vedsted, Bream and Haldager Formation deposits, are obviously immature as the reflectance trend shows values below 0.50 % $R_o$ . The sediments beneath might have attained maturity but no commercial hydrocarbons have been encountered in the well. The exact threshold value for onset of the oil generation zone, i.e. the top of the so-called "oil window" has not been estimated yet for this on-shore area. However, according to Cornford (1984) the threshold value in terms of vitrinite reflectance is set near 0,60 %  $R_o$  for the North Sea area. With 0,60 %  $R_o$  taken as a threshold value (also used by Thomsen et al 1987) mature sediments might be reached near the top of the Gassum Formation sediments at about 8500' depth. By extrapolation of the reflectance trend the lower limit of 1.3 % $R_o$  (i.e. the base of this "oil

window") can be predicted at approximately 17.400' depth, presumably in Triassic redbeds.

Thus the "oil window" is expected from 8500' - 17.400' (2600 m - 5300 m) at the location of Hyllebjerg 1, which is a narrower interval occurring at a shallower depth than the 2700-6000 m estimated for the marginal area of the Danish Subbasin (Lindgreen & Thomsen 1982). Whilst sufficient maturity seems to be reached in the deeper part of the well, only small traces of non migrated hydrocarbons can be detected. The possible reasons for this will be discussed in the final section.

#### Spectral fluorescence measurements

The fluorescence data from measurements on extracted bitumens are listed in table 3. The calculated trichromatic coordinates x,y from the measured spectral curves are plotted graphically in a two-dimensional diagram (DIN 6164 for the illumination D65, fig. 7) where the maturity can be shown. The straight lines which divide the diagram into 24 parts indicate the shade of colour, while the curved lines characterize the degree of colour saturation.

It is shown that the shade of fluorescence colour of extracts from coals and different source rocks changes with increasing maturity in a clock-wise direction in the diagram (blue line no. 16-19) at the coalification stage of peat, through green (20-24) and yellow (1) to orange (4-6) at the highly coalified stage of anthracite and even higher values) simultaneously with an increase in saturation (Hagemann & Hollerbach 1981a).

According to recent investigations (Hagemann, pers. comm.) the shade of colour can be correlated with huminite/vitrinite reflectance although the changes in fluorescence properties of the extracts are discontinuous, while the reflectance behaviour of the huminite/vitrinite particles indicates a gradual continuous changes. The colour shade between

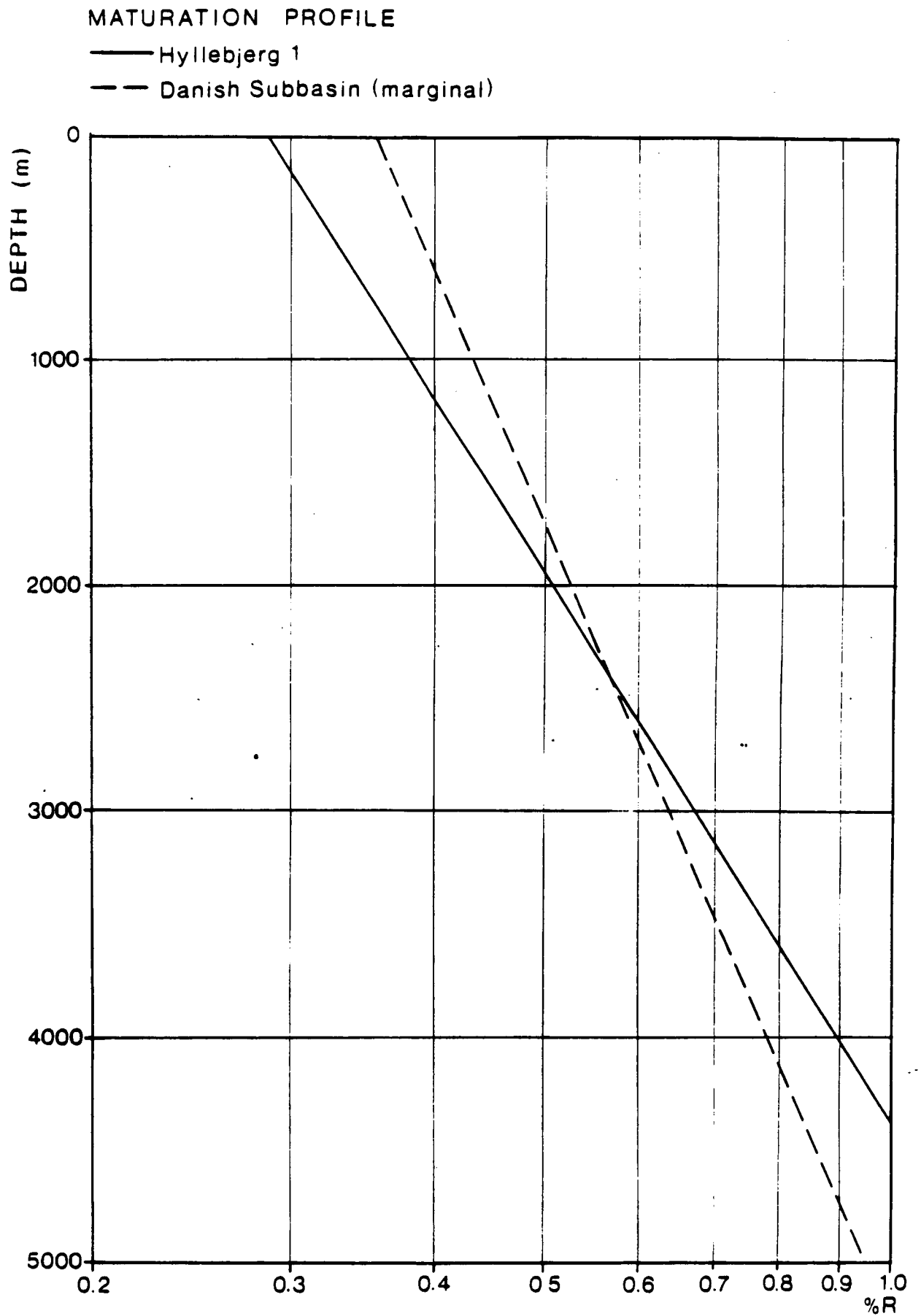


Fig. 6 (Data for marginal Danish Subbasin from Lindgreen & Thomsen (1982)).

lines 21 and 24.5 corresponds to a huminite/ vitrinite reflectance of approximately 0.25-0.50 %R, line 24.5 with approximately 0.50-0.75 %R, lines 1-3 with 0.75-1.15 and lines 3-5.5 with approximately 1.20-1.50 %R. The dividing line between immature and mature sediments is drawn at about the shade of colour between lines 24.5 and 1 (Hagemann, pers. comm.).

The plotted chromaticity values in fig. 7 shows the samples to range from almost unaffected to mature organic matter. The results are generally in reasonable accordance with those of Hagemann as most values plot between the shade lines 24 and 1, indicating reflectance values of about 0.45-0.75 %R. An increasing maturity is seen in a clockwise direction starting from samples of the Vedsted Formation to samples of the Fjerritslev, Gassum and Vinding Formation, together with an increase in saturation from close to curve no. 2 to curve no. 5.

Some lower and higher shade values are observed which do not correspond to the reflectance measurements. The lower shade values are from the Vedsted and Bream Formation samples which consist of unconsolidated material.

The calculated two shade values about line 22 (samples 916A and 923A) suggest unrealistically immature, almost recent sediments (rank equivalent to peat - soft brown coal) but the gas chromatograms show severely biodegraded and possibly contaminated samples respectively. The reflectance measurements show higher maturity with values about 0.45-0.50 %R.

Maturity estimations from chromaticity values of the other samples (913A-932A) of the Vedsted and Bream Formations (between the shade lines 23-24.5) must also be done with some reservation as the extracts from these samples often seem to be more or less contaminated, and only very small amounts were available.

The shade values obtained from the Fjerritslev, Gassum and Vinding Formations are rather similar, mostly between shade lines 24.5 and 1, which indicates the transition zone from immature to mature sediments. A few of the samples from the



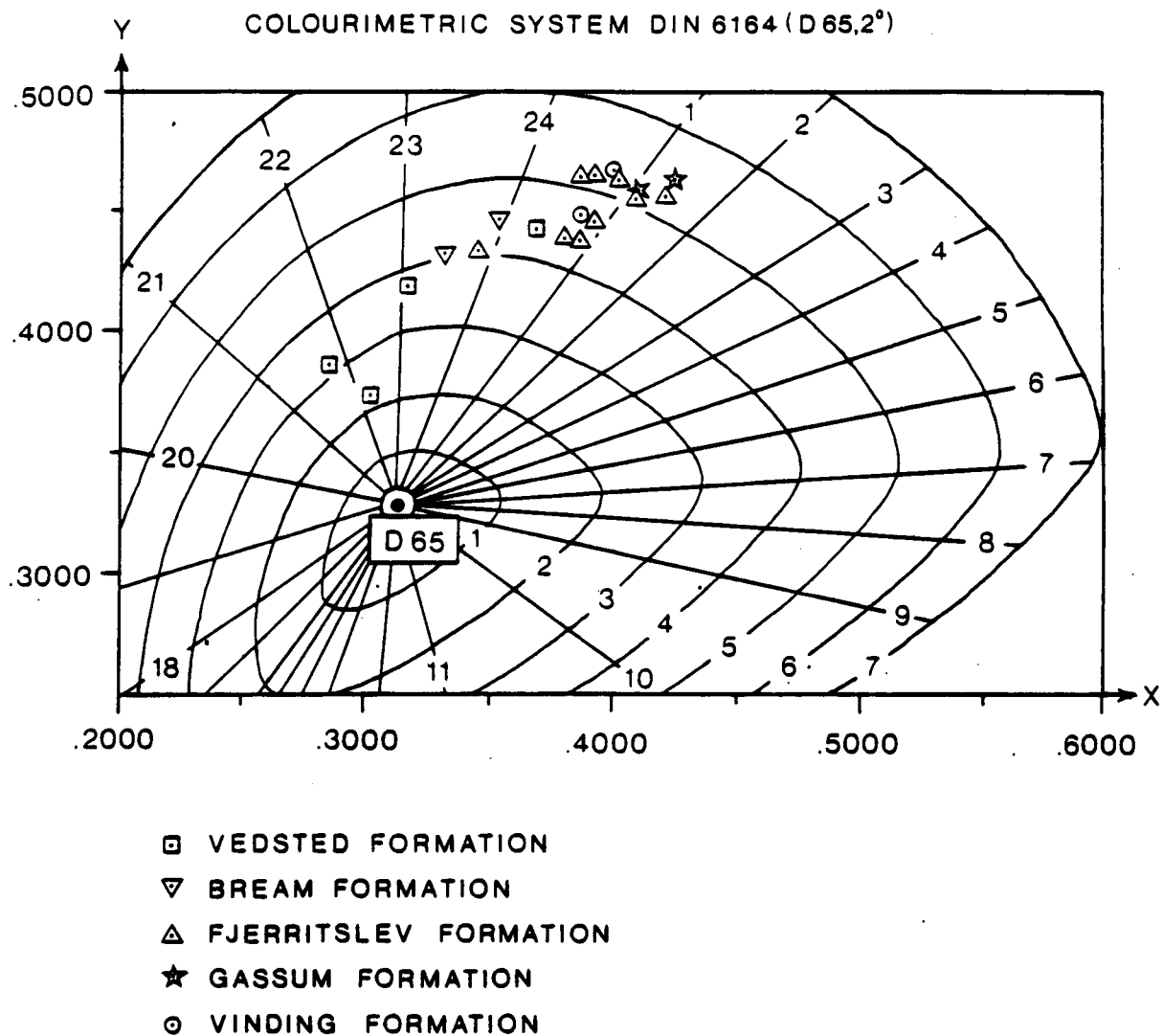


Fig. 7 Chromaticity diagram DIN 6164 showing shade (straight lines 1-24) and saturation of colour (curved lines 1-7) of extracts from different cutting samples.

Fjerritslev and Gassum Formations (945A, 954A, 973A and 976A) clearly lie in the "oil window" (boundaries approximately at shade line 24.5-1 to 4) with their shade values higher than 1. On the other hand the fluorescence analysis indicates that the two lowermost samples from the Vinding Formation (982A, 985A) do not reach this maturity. This clearly contradicts the reflectance measurements and extractability results which both show the highest maturity for the Vinding Formation samples. This is also shown by linear regression analysis. Calculations on Q-values from the spectral curves and  $\lambda_{\max}$  values with corresponding R<sub>o</sub>-values show relatively low correlations ( $r=0.59$  and  $0.58$  respectively; 916A and 923A omitted). (See fig. 8). In the literature (Ottenjann et al. 1974, Teichmüller & Ottenjann 1977, Teichmüller & Wolf 1977, Teichmüller 1979, Ottenjann 1980, Teichmüller & Durand 1983) it is shown that different liptinite macerals have originally different fluorescence colours and distinctively changing spectral fluorescence properties with increasing maturation. Alginites show more shorter wavelength fluorescence colours with higher intensities than most other liptinites (i.e. sporinite, cutinite and bituminite) at equal maturity.

Maturation is characterized by a decrease of fluorescence intensity, and a shift in fluorescence colour to the red part of the spectrum. Spectral composition and intensity vary for the different liptinite macerals, as well as for the different maturity levels.

In this study samples 945A, 954A, 973A and 976A with the highest shade values  $> 1$  and thus a higher proportion of the longer wave length orange red light in the spectra (i.e. high Q-values), also show the highest aromatic hydrocarbons/saturated hydrocarbons ratio  $> 2.5$  (see table 4 and fig. 3).

A high aromatic hydrocarbon/saturated hydrocarbon ratio may indicate terrestrial origin or a high terrestrial proportion of organic matter with fluorescence parameters which normally indicate mature material and thus can resemble increased maturity. In contradiction to this, aromatic

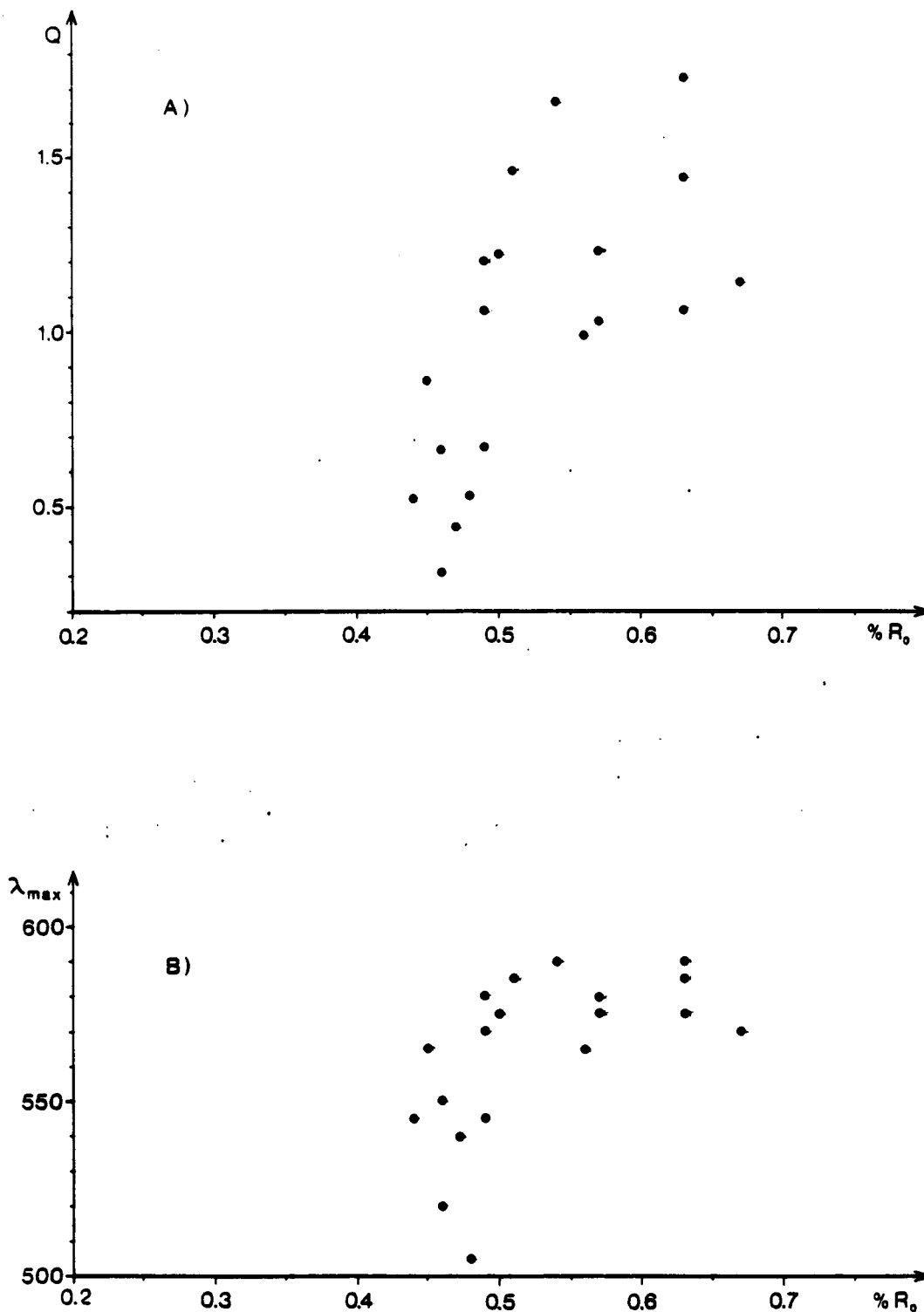


Fig. 8 Correlation tests of Q-values (=relative intensity at 650 nm / relative intensity at 500 nm) and  $\lambda_{max}$  - values (= the position of the maximum wavelength in nm) from the spectral fluorescence curves with the corresponding %R<sub>o</sub>-values (= vitrinite reflectance).

A)  $r = 0.59$  B)  $r = 0.58$

hydrocarbons/saturated hydrocarbons ratio  $< 1.0$  are obtained from samples 929A, 932A, 982A and 985A with algal or marine phytoplankton dominated organic material, giving clearly lower fluorescence parameters. As lipophilic substances, such as the aromatic and heteroaromatic compounds, influence the spectral character of the fluorescing light, it is not surprising that the investigated samples with such varying extract composition show different spectral curves, giving a complex maturation picture, as is also seen for the different liptinite macerals.

The distribution of the fluorescence data in the chromaticity diagram, and the reasonable correlations with the reflectance values, seem to reflect dependence on the organic type which have different fluorescence properties. The latter are superimposed on, and to some extent mask, the maturation effect.

This is clearly in opposition to Hagemann & Hollerbach (1980, 1981a, 1981b) who claimed that the extract composition was independent of the kerogen type. However, it must be pointed out that this method has not previously been tested on cutting material from a basin with often shifting lithologies and thus varying organic compositions (kerogen types), and more data are needed. However, it does seem possible to use this new analytical method for a rough maturity estimation.

#### Extractable organic matter (EOM) and chromatographic - separation

Extraction methods are described in the appendix

The amounts of extractable organic matter (EOM) and the composition of the chromatographic fractions are given in table 4. The EOM and the hydrocarbon content (saturated plus aromatic hydrocarbons) are normalized to total organic carbon content (TOC) to minimize random spread of extraction

results due to the different content of organic matter. The results are shown in fig. 3, where the composition is also indicated.

As relatively minor amounts of sediment were available, only small extracts were obtained (between 8 and 54 mg). As part of this was used for fluorescence measurements very little was left for the chromatographic separation. Evaluation of the results must therefore be done with care as some scatter is to be expected.

The EOM results show increasing extractabilities and hydrocarbon ratios downwards, indicating increased maturity.

Most of the (upper) samples have ratios between 20-50 mg EOM/g TOC with the lowest ratios somewhat below 20 mg EOM/g TOC from the uppermost samples. The same is true for the hydrocarbon content (mg HC/g TOC) as most samples generally have ratios < 10 mg HC/g TOC (especially in the uppermost samples) or slightly above. This seems to indicate immature or flushed sediments as in recent (immature) sediments the extractability and hydrocarbon ratios are fairly low with hydrocarbon contents generally > 10 mg HC/g TOC (Tissot & Welte 1984). In these samples, with approximately equal amounts of extracts, the varying composition of the extract may reflect different types of organic matter, as is seen under the microscope.

In samples 982A and 985A from the deepest part of the well there is a rapid increase in the EOM and the hydrocarbon content. This reflects increased maturity, though it may partly be caused by a change from terrestrial influenced to marine/brackish plant material, as is documented in the microscopic description of the organic matter. According to Tissot & Welte (1984) the extractability is increased with greater maturity to a maximum of about 100-200 mg EOM/g TOC with 70-150 mg HC/g TOC (actual amount dependent on the kerogen type) in the principal phase of oil formation, after which the extract values decrease as the principal phase is passed. Thus sample 985A, with a ratio above 100 mg EOM/g TOC and 60 mg HC/g TOC presumably represents a mature extract, although the hydrocarbon ratio obtained is lower than

the former values. Mature sediments can therefore be found at approximately 9200' depth in the Vinding Formation.

In the literature, extractability values of immature sediments with potential source rock characteristics are typically reported to be 30-40 mg EOM/g TOC (Tissot & Welte 1984). According to this study, most sediments in the investigated sequence show relatively unfavourable source rock conditions. Only sediments from the F-4 and F-3 Member of the Fjerritslev Formation and possibly a few horizons from the Vedsted Formation, the F-1 Member of the Fjerritslev Formation and the Gassum Formation, lie within this range and may have a source rock potential for oil.

#### Gas chromatography

The gas chromatograms of the saturated fractions are shown in fig. 9 a-e, and the different ratios (pristane/n-C<sub>17</sub> alkane), phytane/n-C<sub>18</sub> alkane (phytane/n-C<sub>18</sub>), pristane/p-phytane and Carbon Preference Index (CPI)) calculated from the peak areas are listed in table 5 and shown in fig. 10.

The intensities in the chromatograms are not directly comparable since very small amounts of saturated hydrocarbons were available for analysis and sometimes the chromatograms have been recorded under high detector signal amplification; column bleeding can also be recognized in some of the chromatograms, especially for 929A and 932A and also slightly for some of the other samples.

In addition to this there are contamination and biodegradation problems.

Sample 923 is characterized by higher n-C<sub>16</sub> and n-C<sub>18</sub> peaks together with a large hump in the high molecular range. Because of the absence of the most n-alkanes it is considered to be biodegraded though there is a higher concentration of the n-C<sub>16</sub> and n-C<sub>18</sub> which cannot be explained.

If it is contaminated the presence of contamination is not supported by the EOM/TOC value.

Contamination can be an uncertain factor, especially in the upper samples from the Vedsted and Bream Formation which consist of relatively unconsolidated material which is difficult to separate from the drilling mud by washing. Small amounts of contamination by drilling mud and/or mud additives can considerably affect and mask the results. However severe contamination from the mud itself is not likely as freshwater based mud with bentonite was used during the drilling, but the mud filtrate itself has not been analysed.

Sample 916A on the other hand is anomalous in showing very high pristane and phytane peaks, giving high pristane/n-C<sub>17</sub> alkane and phytane/n-C<sub>18</sub> ratios compared with all the other samples. This may be caused by biodegradation (presumably due to meteoric water or storage of wet samples) which leads to loss of the n-alkane content, especially in the low molecular weight range, while the isoprenoids are less affected and so pristane and phytane become relatively more abundant giving enlarged pristane/n-C<sub>17</sub> alkane and phytane/n-C<sub>18</sub> ratios (Deroo et al. 1974, Bailey et al. 1973).

Since different lithologies and environments of deposition were investigated in this study, a variety of compositions of the organic matter can be expected to be indicated by the gas chromatograms.

#### Characterization:

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A short summary of the main characteristics of the gas chromatograms is given below:

The Vedsted Formation is represented by samples 913A, 915A, 916A and 923A. As 916A and 923A are biodegraded and/or contaminated. only samples 913A and 915A are reliable, as

Fig. 9 a.

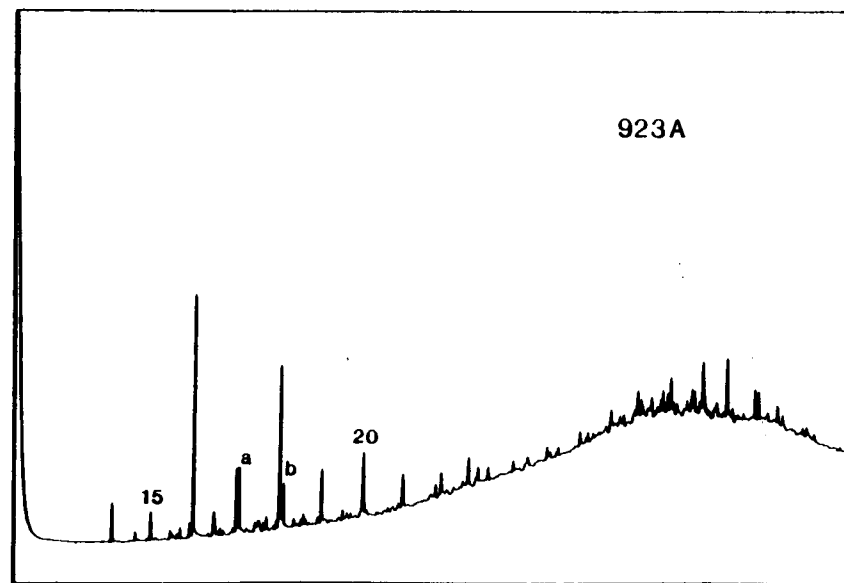
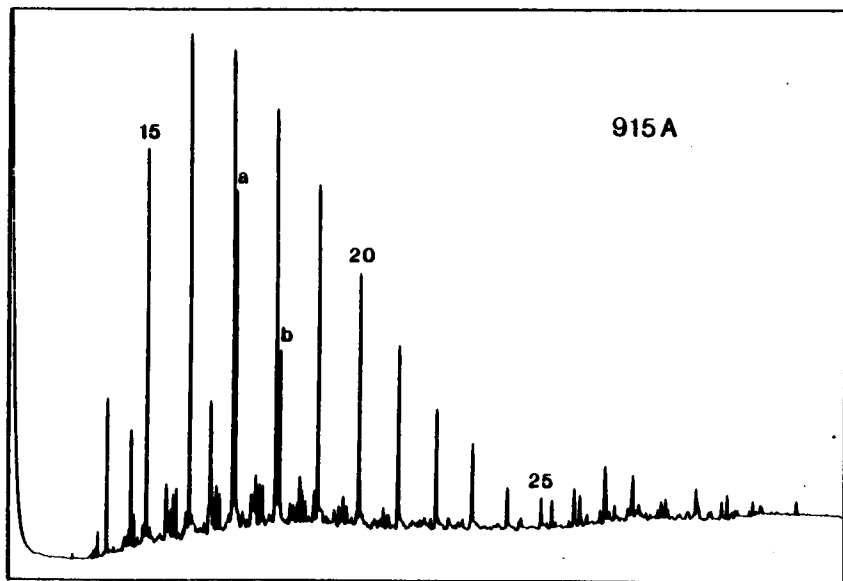
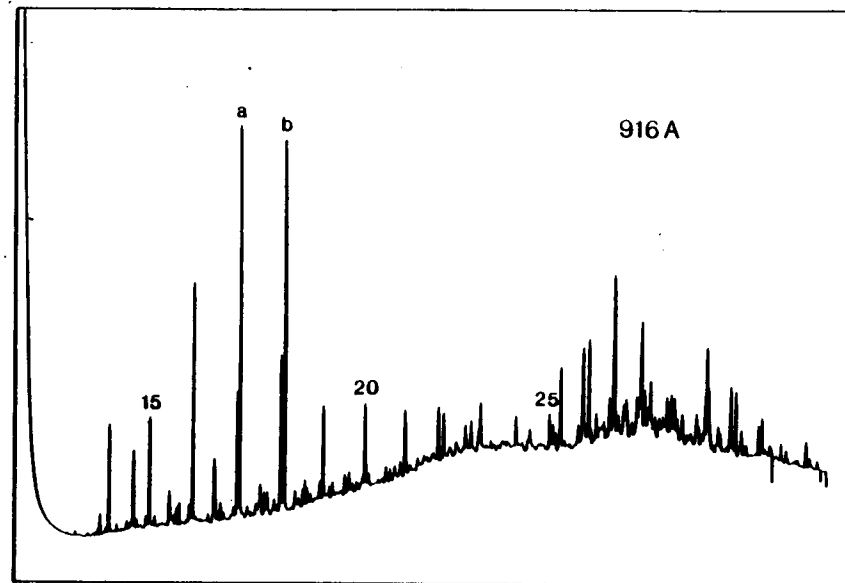
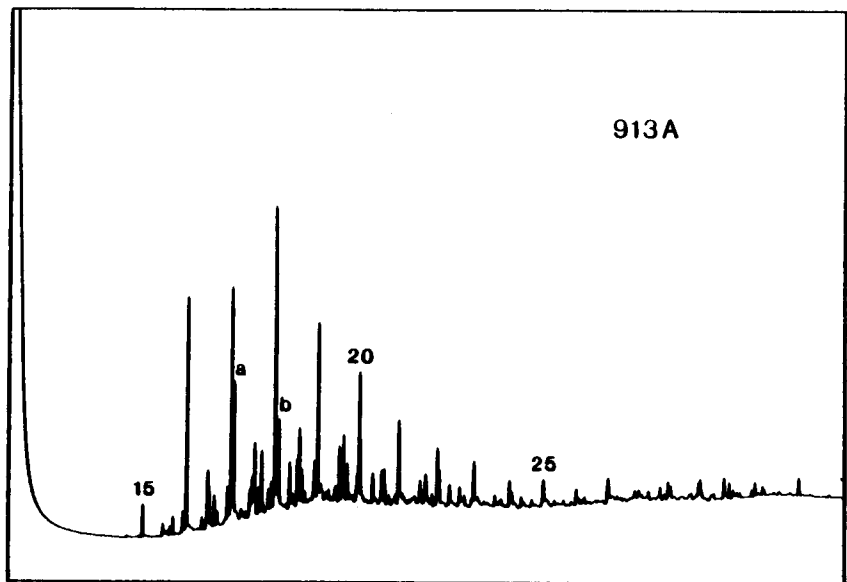




Fig. 9 b.

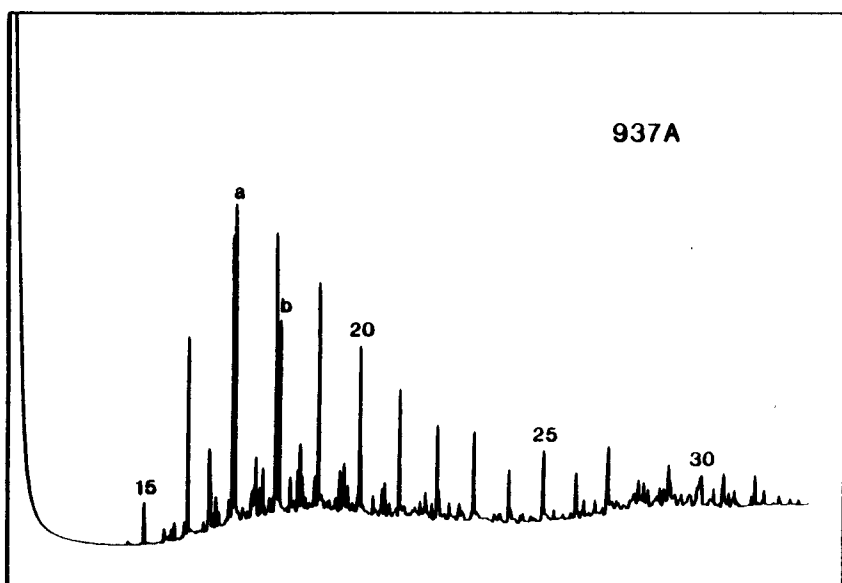
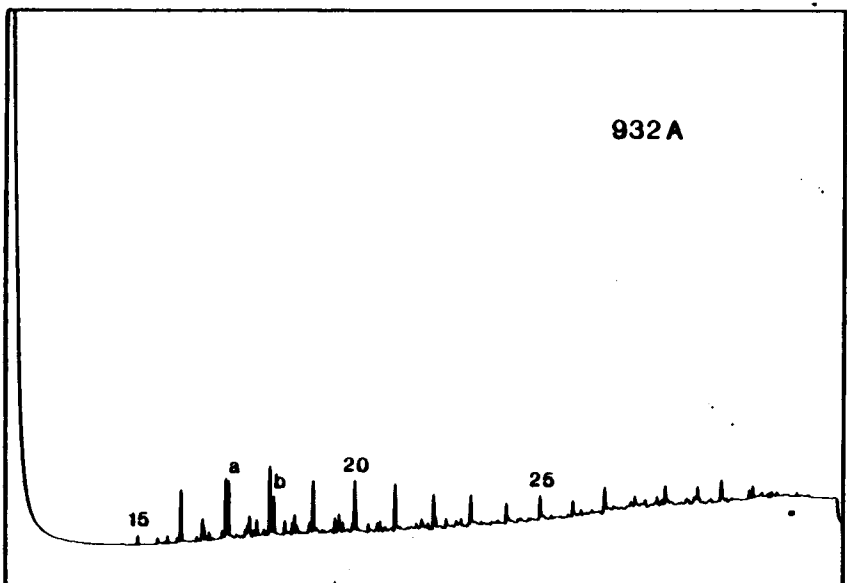
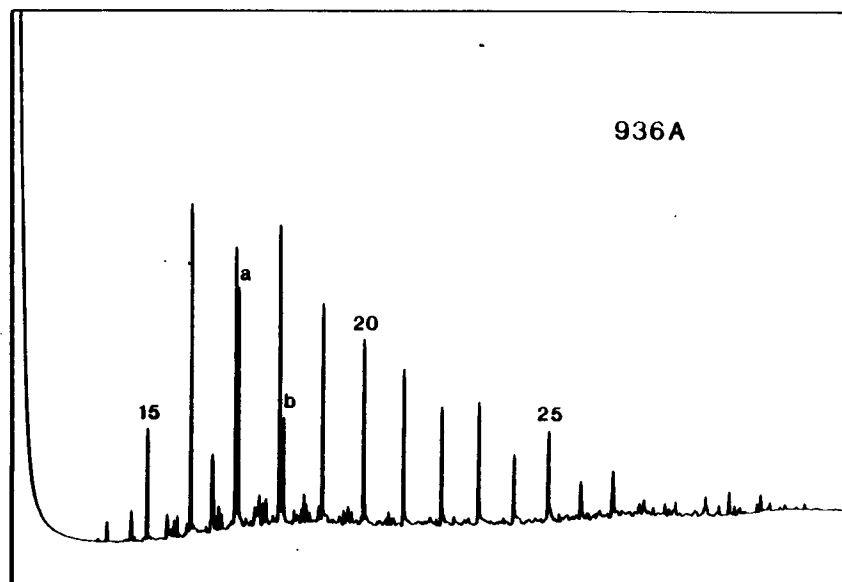
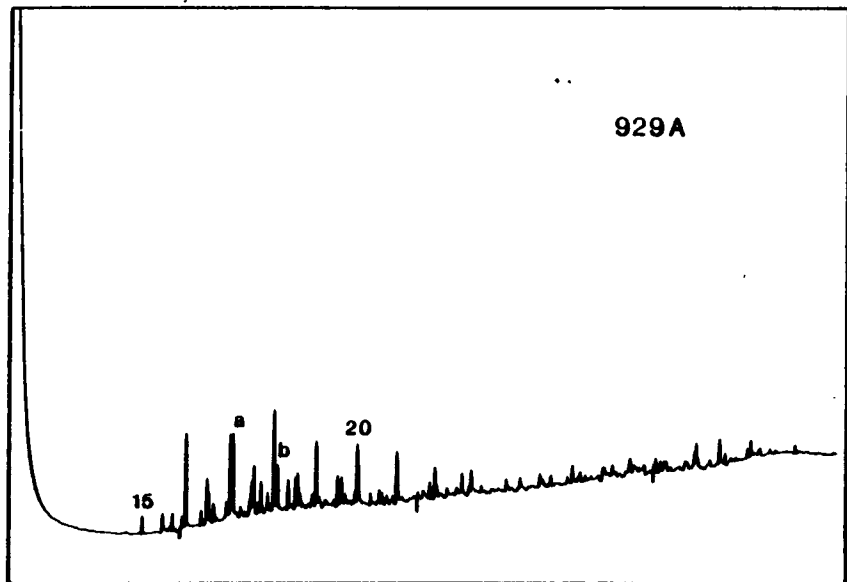


Fig. 9 c.

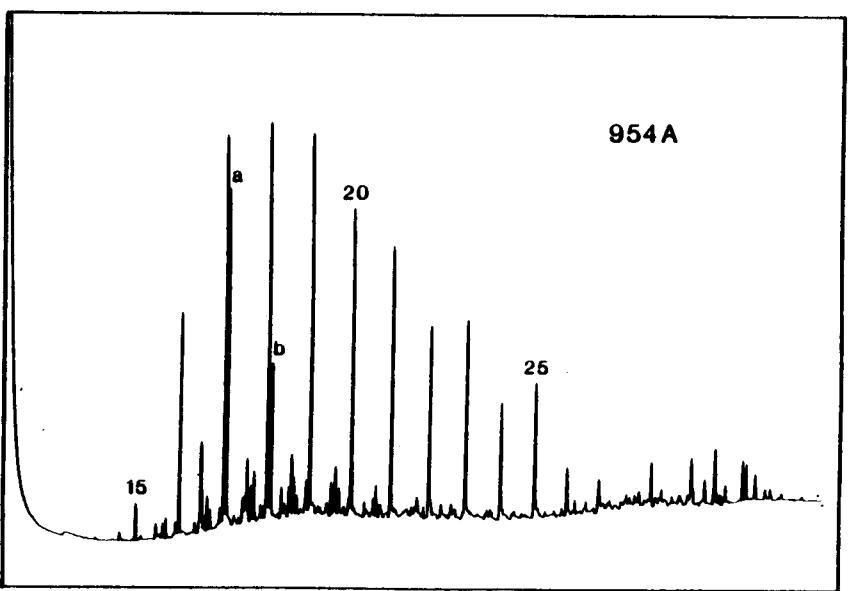
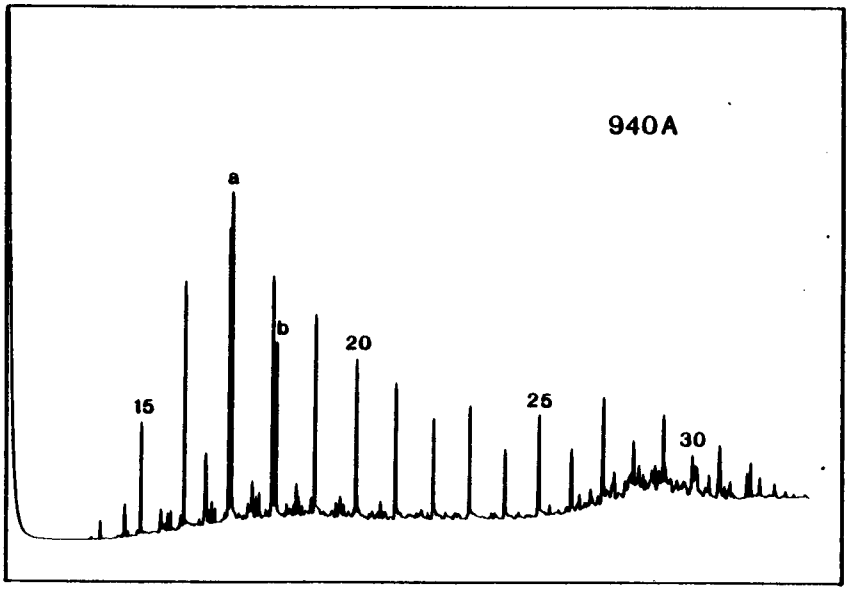
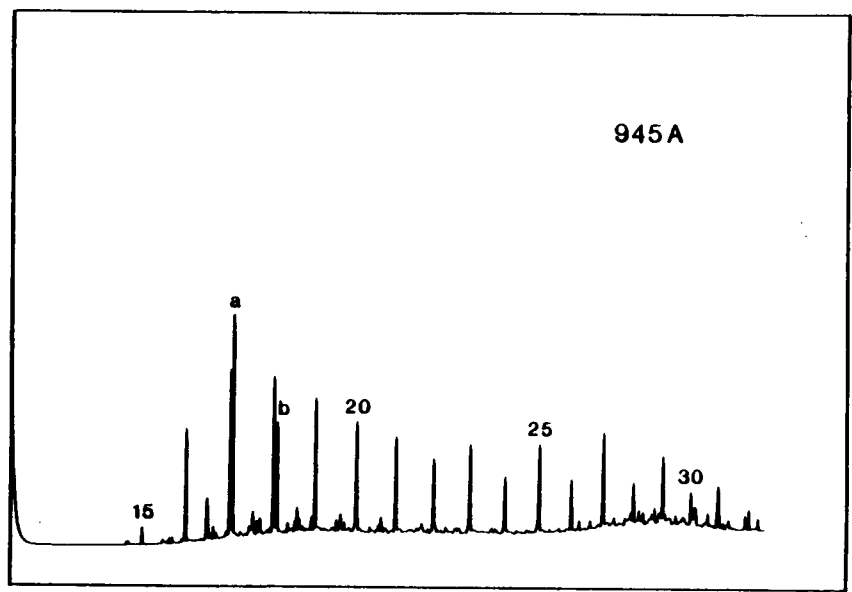
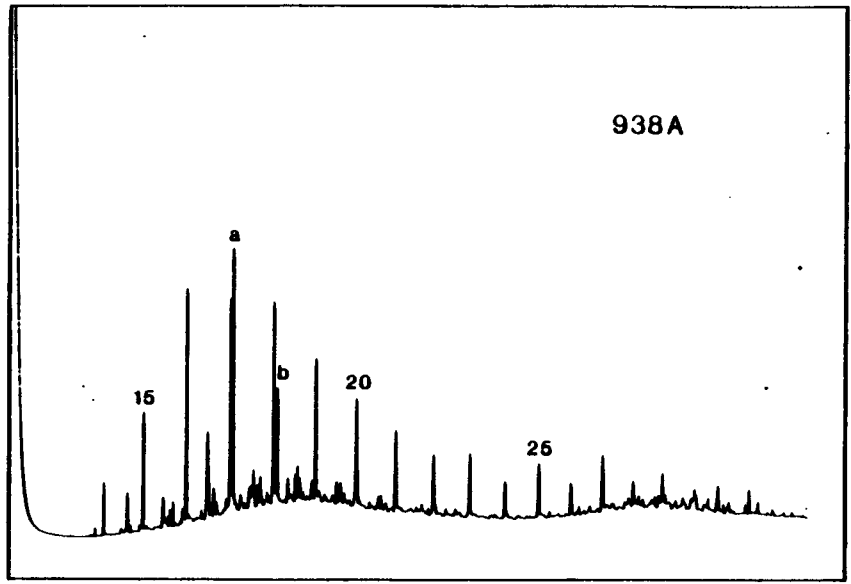
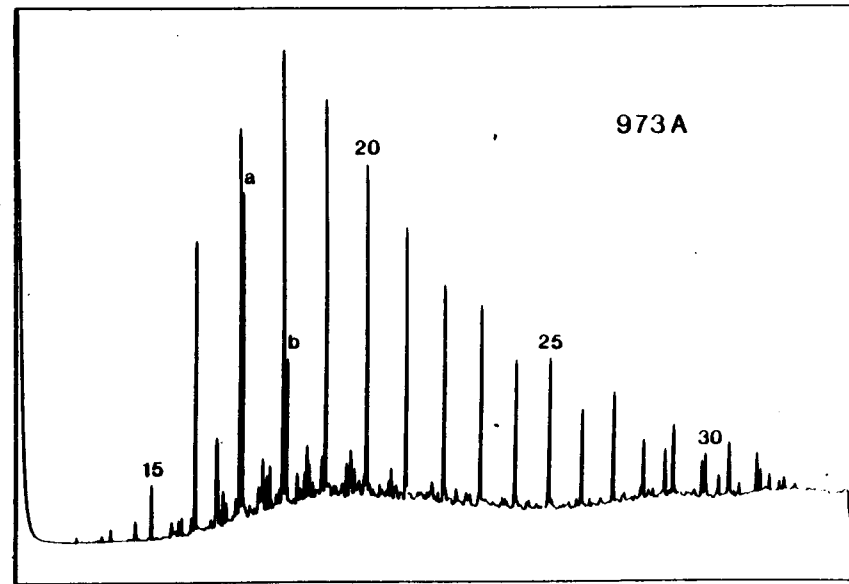
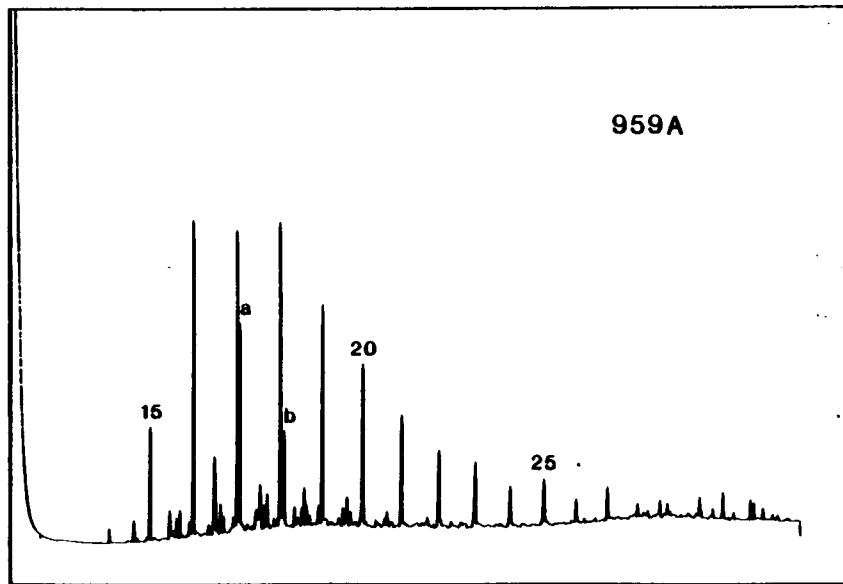
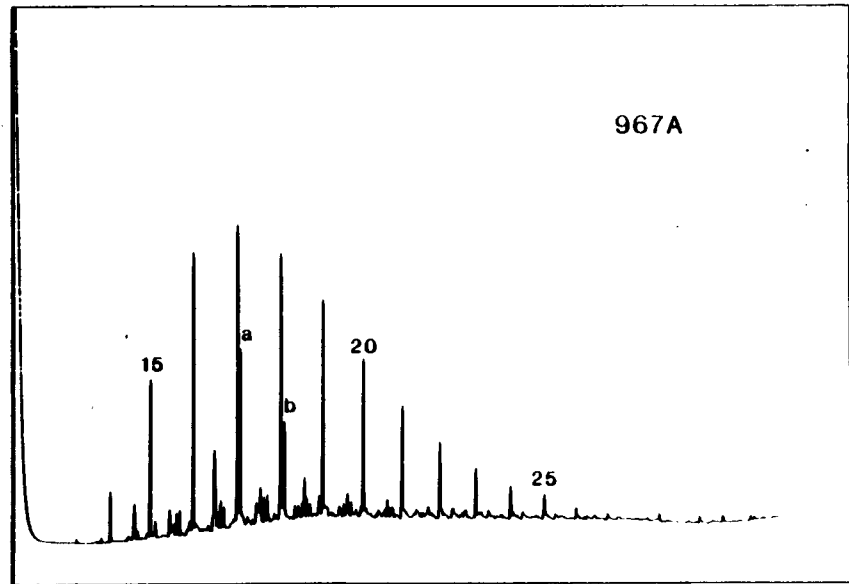
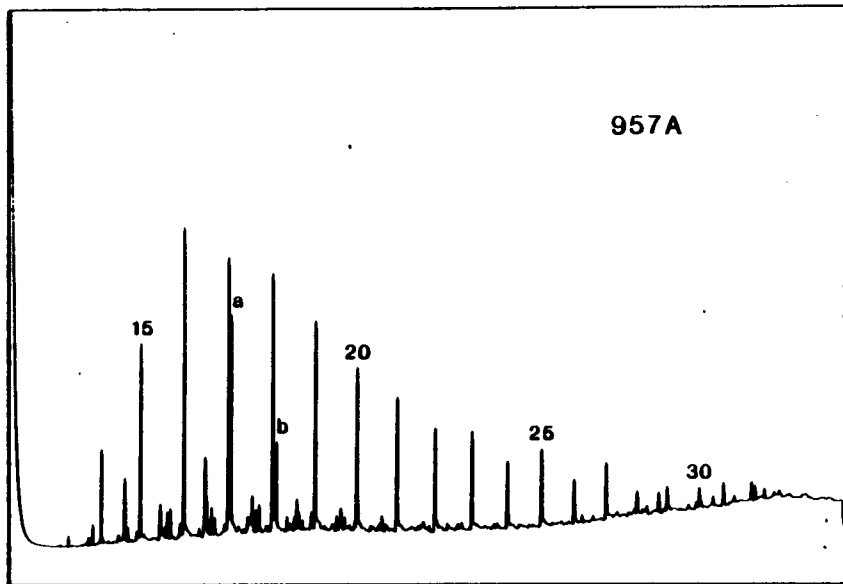


Fig. 9 d.



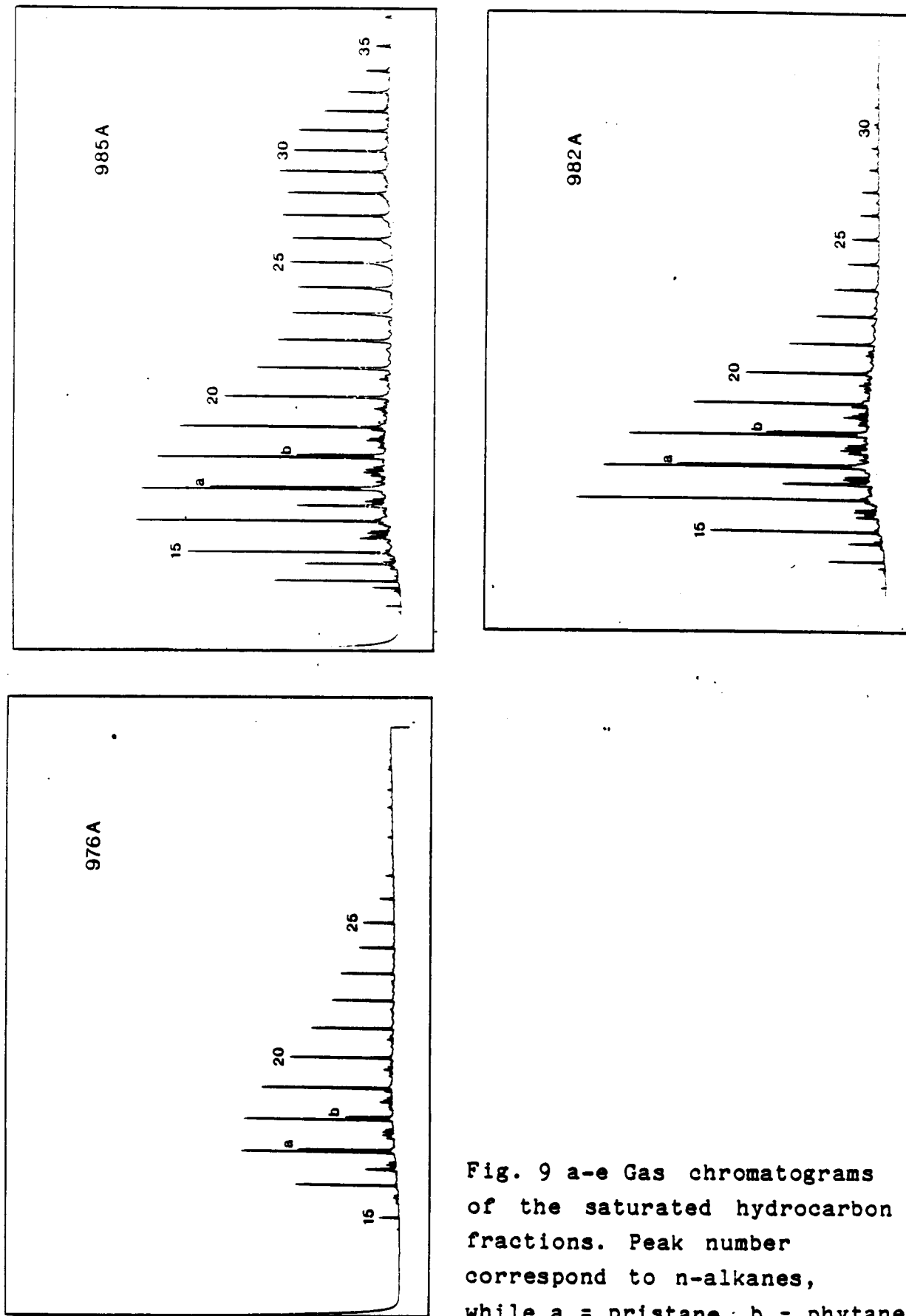


Fig. 9 a-e Gas chromatograms of the saturated hydrocarbon fractions. Peak number correspond to n-alkanes, while a = pristane, b = phytane.

Fig. 9 e.

discussed previously. In these gas chromatograms the n-alkanes in the low molecular weight range are dominant with a maximum at n-C<sub>17</sub> typical of marine algal material. Only a few small peaks in the higher molecular range > n-C<sub>23</sub> are seen (indicative of terrestrial input). This is in good accordance with the marine type of sediments and the visual appearance of 915A (dinoflagellates can be identified among the alginites) while in 913A the terrestrial input appears to be more prominent.

The Bream Formation is represented by gas chromatograms of samples 929A and 932, which only show small peak intensities because of the small amounts of hydrocarbon extracts available. In 929A only short chain alkanes were detected with n-C<sub>17</sub> predominant, indicative of marine phytoplankton, whilst in 932A longer chain n-alkanes with a prominent n-C<sub>27</sub> peak were also detected, indicating some mixing with organic matter of terrestrial origin. This terrestrial input is not confirmed by the microscopic observations. In both samples a dominance of marine algal material is seen under the microscope (some dinoflagellates are observed) in accordance with the marine-dominated nature of the sediments.

The Haldager Formation: No samples of this formation have been analysed.

The Fjerritslev Formation is represented by samples 936A, 937A, 938A, 940A, 945A, 954A, 957A, 959A and 967A.

In the gas chromatograms of 936A (transition from the Haldager Formation to F-4 Member) n-alkanes in the low molecular range with a n-C<sub>17</sub> maximum are dominant, indicating a high contribution of marine algal material. Under the microscope, the terrestrial contribution seems larger due to some large angular huminite/vitrinite particles. These may be flooded into the marine environment. The gas chromatograms from the other samples from the F-4 and F-3 Member (937-945) are rather similar. They show a slightly bimodal n-alkane distribution with a high maximum at n-C<sub>17</sub> and a

smaller one at n-C<sub>27</sub>, indicating varying proportions of a dual algal-higher plant source for the organic matter. In the gas chromatograms from the deeper samples 940A and 945A a slightly higher input of terrestrial material is observed compared to the upper two samples 937A and 938A. Under the microscope these samples from the F-4 and F-3 Member also look very similar as they are dominated by liptinite composed mainly of bituminite and some recognizable alginite and sporinite. Alginite (a few algae of Tasmanites type) is seen in all samples and is probably responsible for the n-C<sub>17</sub> maximum. The bituminite content increases downwards in these samples and is the main organic component in the lowermost two samples. As there is only a minor contribution of huminite/vitrinite in these samples, it is assumed that at least some of the bituminite is of terrestrial origin, providing the high molecular weight range n-alkanes.

The gas chromatograms from samples 954A-967A (F-2 and F-1 Member) are relatively similar. In all samples short chain alkanes are clearly dominant, with prominent n-C<sub>17</sub> peaks. Only a few small peaks were detected beyond n-C<sub>25</sub> (especially seen in 957A and 959A). The prominent n-C<sub>17</sub> peaks (some evaporation is seen in 954A) might indicate a considerable marine phytoplankton origin for the organic matter, which is consistent with the marine sediment type. Under the microscope most of the organic matter seems to be of terrestrial mainly reworked which thus may not have contributed to the extract. Slightly increased maturity is seen in 967A (no CPI-calculation was possible) compared with the others.

The Gassum Formation is represented by samples 973A and 976A. Like the preceding gas chromatograms, 973A and 976A are dominated by n-alkanes in the low molecular weight range. However 973A is also associated with several small peaks of longer chain n-alkanes beyond n-C<sub>25</sub> (normally derived from higher land plant material). 973A is notable for its content of a few microscopically identifiable coal particles. But the terrestrial input is not particularly significant in the gaschromatogram. This may be due to the

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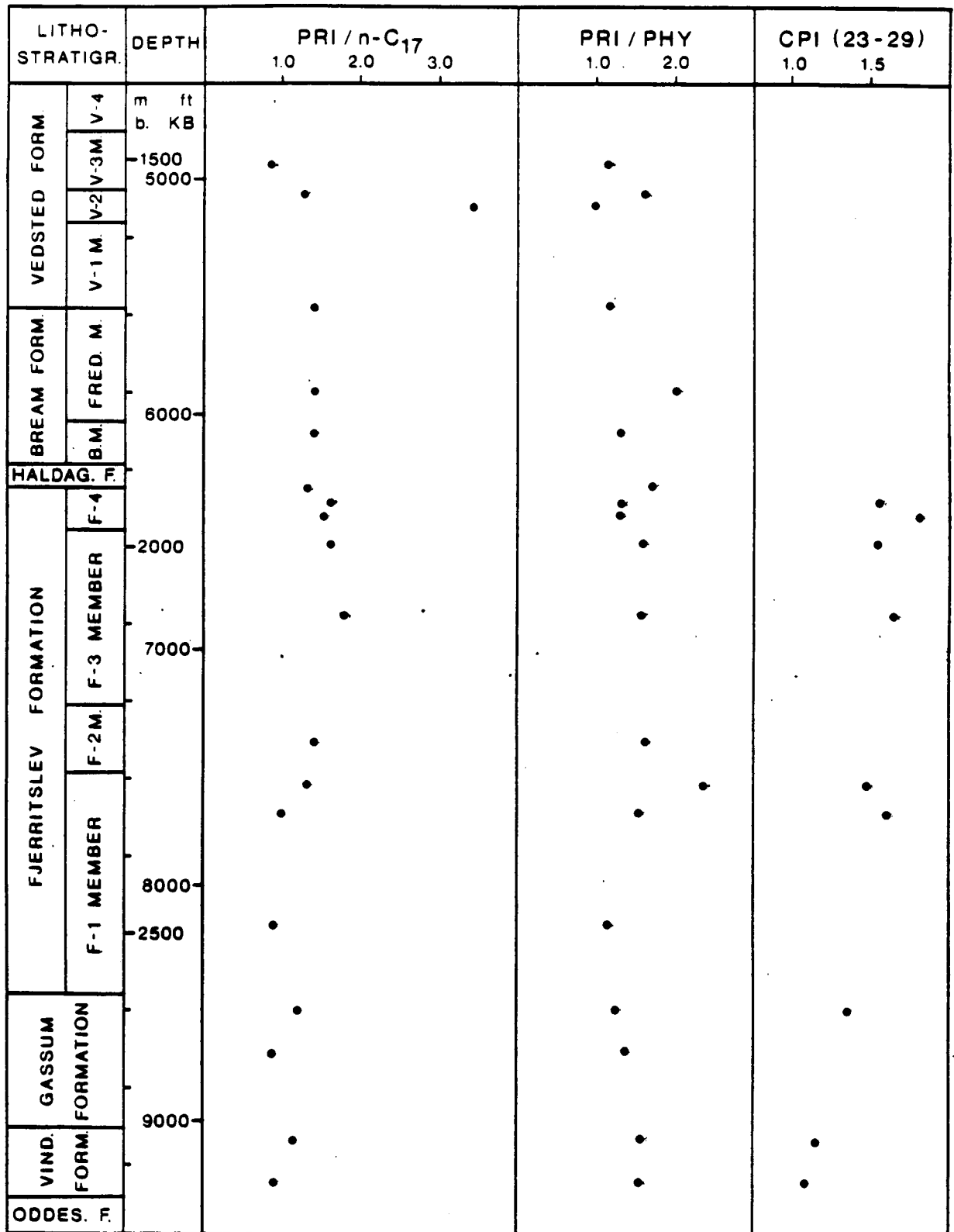


Fig. 10 Calculated maturity indices obtained from quantified peak areas in the corresponding gas chromatograms.

fact that only very small amounts of the organic matter may have been extracted from the terrestrial material and that the terrestrial influence has been overestimated optically. It may also be caused by the small amount of extract available and thus not optimal analysis. The gaschromatogram of sample 976A resembles the former but with a more smooth n-alkane distribution curve with an only slightly discernible odd-predominance. The CPI cannot be calculated.

The Vinding Formation is represented by gas chromatograms 982A and 985A.

982A closely resembles 976A as it is mostly dominated by shorter chain n-alkanes, though small peaks of longer chain n-alkanes can be detected up to n-C<sub>31</sub> (indicating at least some input of terrestrial higher plant material). Under the microscope, a rather mixed organic matter composition is seen. In the gas chromatogram the dominance of the n-alkanes in the low molecular range and the almost non-existent odd carbon number predominance must reflect increased maturity which restricts further differentiation of the organic composition.

The gas chromatogram from 985A is very different from all the others in showing a clear bimodal distribution of the n-alkanes with a very high proportion of the long chained n-alkanes up to n-C<sub>36</sub>. In the low molecular range the n-C<sub>19</sub> peak is (according to the integrated peak areas) slightly predominant, indicating fresh water plant material (Hutton et al. 1980). In the high molecular range the prominent n-C<sub>29</sub> peak normally indicates a higher land plant material. It can alternatively be derived from the fresh-brackish water algae *Botryococcus* (Hutton et al. 1980). This source is confirmed as under the microscope a liptinite dominance is seen, mainly composed of *Botryococcus*-like algae. The lowered CPI-value close to 1 reflects a mature sediment.

The Oddesund Formation: No samples from deposits belonging to this formation have been analysed.



## Maturity indices:

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The CPI-index was originally proposed by Bray & Evans (1961) who calculated it as the ratio of the sum of the percentages of C<sub>25</sub>-C<sub>33</sub> odd-carbon n-alkanes to the sum of C<sub>26</sub>-C<sub>34</sub> even-carbon n-alkanes. In this study only a few gas chromatograms showed long chain n-alkanes in excess of n-C<sub>30</sub>, and as the reliability is decreased in the peak area calculation of smaller peaks, the CPI indices were calculated for C<sub>23</sub>-C<sub>29</sub>:

$$\text{CPI} = \frac{\frac{1}{2} C_{23} + C_{25} + C_{27} + \frac{1}{2} C_{29}}{C_{24} + C_{26} + C_{28}}$$

CPI-indices have not been calculated for all samples, since longer chain n-alkanes derived from terrestrial material are not always present.

In the gas chromatograms an increased maturity can be observed from the base of the Fjerritslev Formation downwards, indicated by the smoothing of the n-alkane distribution curves. This is also reflected in the CPI-values.

The data show a clear lowering in the odd-even carbon number predominance of the higher n-alkanes with a decreasing trend in CPI-values from about 1.6 at the top of the Fjerritslev Formation and approaching 1.1 in the deeper samples of the well. CPI-calculations were not possible for the relatively mature-looking gas chromatograms from samples 967A and 976A.

According to Tissot & Welte (1984) CPI-values above 1.5 always refer to relative immature sediments, Waples (1980) sets a value of 1.2. The Fjerritslev and Gassum Formation values seem to reflect immature sediments whilst an increased maturity is indicated from the values of the Vinding Formation sediments. These results are in good agreement with the maturity estimations from extraction data, but in slight contradiction with the vitrinite reflectance analysis, if 0,55-0,60%R<sup>o</sup> is used as threshold for the onset of the oil generation. Maturity may be reached at a greater depth than indicated from the reflectance measurements. This discrepancy may be due to the fact that the CPI-indices, as well as the amount and composition of the extracts, is related to the original composition of the organic material

while reflectance is only related to the change of vitrinite.

In this study there are only small differences in the pristane/ n-C<sub>17</sub> ratio. This ratio increases slightly in the upper part of the well, followed by a slight decrease of the ratio in the lower part of the well.

Thus the index seems not to be useful as no clear indications can be obtained either for environmental conditions or for maturity.

From the pristane/phytane ratios in this study no clear evidence is generally obtained to indicate the palaeoenvironmental conditions during deposition of the sequence. No pronounced high or low values are detected. Most samples have values around 1.5, indicating non-oxidized to mildly oxidized material (typical for a "normal" marine environment). Only a few samples (929A (Bream Formation) and 957A (Fjerritslev Formation, F-1 Member)) have values of 2 or higher, indicating more oxidized organic content, but this does not generally correspond with other observations and data. Some of the pristane/phytane ratios may be too low, such as for sample 937A. This may be caused by laboratory evaporation of the lighter component pristane, which is more volatile than phytane, or it may be caused by imperfect integration of the peak areas of the gas chromatograms.

#### Hydrocarbon potential;

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Most of the gas chromatograms seem to indicate sediments with a main potential for oil as they show a high proportion of n-alkanes in the lower molecular weight range with maxima at n-C<sub>17</sub>, typical for phytoplankton material. On the other hand, under the microscope most of these samples seem to be mainly composed of terrestrial material, often with a more or less reworked appearance ("pseudovitrinite") with only a minor liptinite contribution (as is especially seen in the Vedsted Formation and the lower part of the Fjerritslev

Formation F-1, F-2 and deeper F-3 Member sediments). This seeming discrepancy may be attributed to the fact that much of the terrestrial organic matter is practically inert and yields little or no extract. The gas chromatograms therefore mainly represent the liptinite component. The hydrocarbon potential in these sediments must therefore be downgraded. Alternatively, the small liptinite fragments may have been underestimated during optical description, as they are sometimes hardly visible under the microscope (submicroscopic).

The gas chromatograms of the upper Fjerritslev Formation (F-4 and upper F-3 Member) seem to indicate a potential mainly for oil (also indicated by the relatively high microscopic liptinite content of bituminite and alginite), although especially in the F-3 Member some terrestrial organic material, representing the secondary maximum in the high molecular weight range of the n-alkanes (presumably derived from some of the bituminite seen under the microscope) may show a lower potential for liquid hydrocarbons, maybe for a high-waxy oil or even ?gas.

Thus the potential for liquid hydrocarbons seems to decrease downwards in the sediments of the Fjerritslev Formation. In the deeper part of the well (approximately from the base of the Fjerritslev Formation and downwards) an increasing maturity of the extracts can be observed in the gas chromatograms although, according to the amount and composition of the extracts and the CPI-values, clear evidence for mature sediments is first present in samples from the Vinding Formation. The gas chromatograms tend to become similar, and differentiation of the origin of the organic matter and assessment of the hydrocarbon potential becomes increasingly difficult. This seems particularly valid for the Gassum Formation sediments where the terrestrial input seen under the microscope can not be identified with certainty in the corresponding gas chromatograms.

A bimodal n-alkane profile, with maxima centered at n-C<sub>19</sub> and n-C<sub>29</sub>, occurs in the gas chromatograms, for one particular sample (985A) from the Vinding Formation. Although the

$C_{23}$  n-alkanes are usually considered to be derived from higher plant lipids, this is not true for the present sample as microscopical observation reveals a fresh/brackish water algae (Botryococcus-like) origin which is oil prone, giving a high waxy type oil (McKirdy et al. 1986). Thus, according to the type of organic matter, in the Vinding Formation some horizons seem to possess a good potential for oil, though the potential may be reduced because of the low organic content.

#### Gas chromatography - mass spectrometry of biomarkers

Since all the samples selected for analysis were rich in hopanes, most information was obtained on this particular type. (The steranes were present in too low concentration for analysis). Gaschromatographic-mass spectrometric analyses were concentrated on determination of the mass fragments for  $m/z$  191 and the results are shown in fig. 11a-b.

The relative abundance of the  $17\beta(H)21\beta(H)$  hopanes (=  $\beta\beta$  hopanes) is shown in table 6. The average ratios  $22S/S+22R$  of the C-22 diastereoisomers in the range of  $C_{31}$ - $C_{34}$  of the  $17\alpha(H)21\beta(H)$ -hopanes (=  $\alpha\beta$  hopanes) were calculated and are listed in table 6. For quantification of the epimerisation of the C-22 position in the  $\alpha\beta$  hopanes, ideally the  $C_{32}$  hopane diastereoisomers should be used. The  $C_{31}$  isomers should be avoided as coevolution of other triterpanes with the 22R isomer can occur under certain conditions (Mackenzie 1984). Besides this, the different isomer pairs all have slightly different equilibrium constants.

As in this study the  $C_{31}$  isomers are the most abundant components, while the other isomer pairs only occur in smaller quantities, the four stereoisomer pairs of  $C_{31}$ - $C_{34}$  were used for calculation of an average ratio. In a few of the upper samples (913A, 915A and 940A) only the isomer pairs of the  $C_{31}$  and  $C_{32}$  were detected in sufficient quantities to be used for calculations. In a few samples (913A,

916A, 932A) either very small amounts were generally available for calculation, or coevolution occurred so that the results were unreliable.

In the m/e 191 fragmentograms not all the possible hopane configurations ( $17\beta(H)$ ,  $21\beta(H)$  and  $17\beta(H)$ ,  $21\alpha(H)$  and  $17\alpha(H)$ ,  $21\beta(H)$ ) were detected in all samples.

The hopane distribution pattern clearly changes downwards from 915A to 967A:

High proportions of  $\beta\beta$ -hopanes are still present in 913A and 915A but the content decreases until 940A, and no  $\beta\beta$ -hopanes are detected at greater depths.

The  $\alpha\beta$ -hopanes are present in all samples, clearly showing increased epimerisation downwards of the 22R-isomer to the 22S-isomer in the C<sub>31</sub>-C<sub>34</sub> range.

The 22S/22S+22R ratio increases from 0.30 in 915A to 0.59 in 967A. In the upper samples (913A-940A) the 22R configuration is still dominant, but in 959A the 22S configuration is approaching equilibrium (=equal amount) with the 22R configuration, and from 967A down to 985A the 22S configuration is dominant. In these samples from the deeper part of the well the final mixture of the 22S and 22R isomers seems to be reached with an isomerisation of 55-59%, indicating that equilibrium has been reached (Mackenzie & Maxwell 1981). Thus from 967A downwards no further differences in the maturity of the sediments can be detected. In this area other chemical maturity parameters should be used, such as the isomerisation of the steranes, but they occur in too low concentrations to be taken into consideration. The  $\beta\alpha$ -hopanes were also detected between the  $\alpha\beta$ -hopanes, but they were not quantified.

Compared with the results of Mackenzie (1984) (fig. A-4) it is surprising that the least stable hopanes, the  $\beta\beta$ -hopanes, are still present in the upper sequence of the well. According to Mackenzie (loc. cit.) the  $\beta\beta$ -hopanes (analysed from the Paris Basin) disappear at an earlier stage of the isomerisation of the  $\alpha\beta$ -hopanes than that which seems to have been passed in this study, according to the reflectance

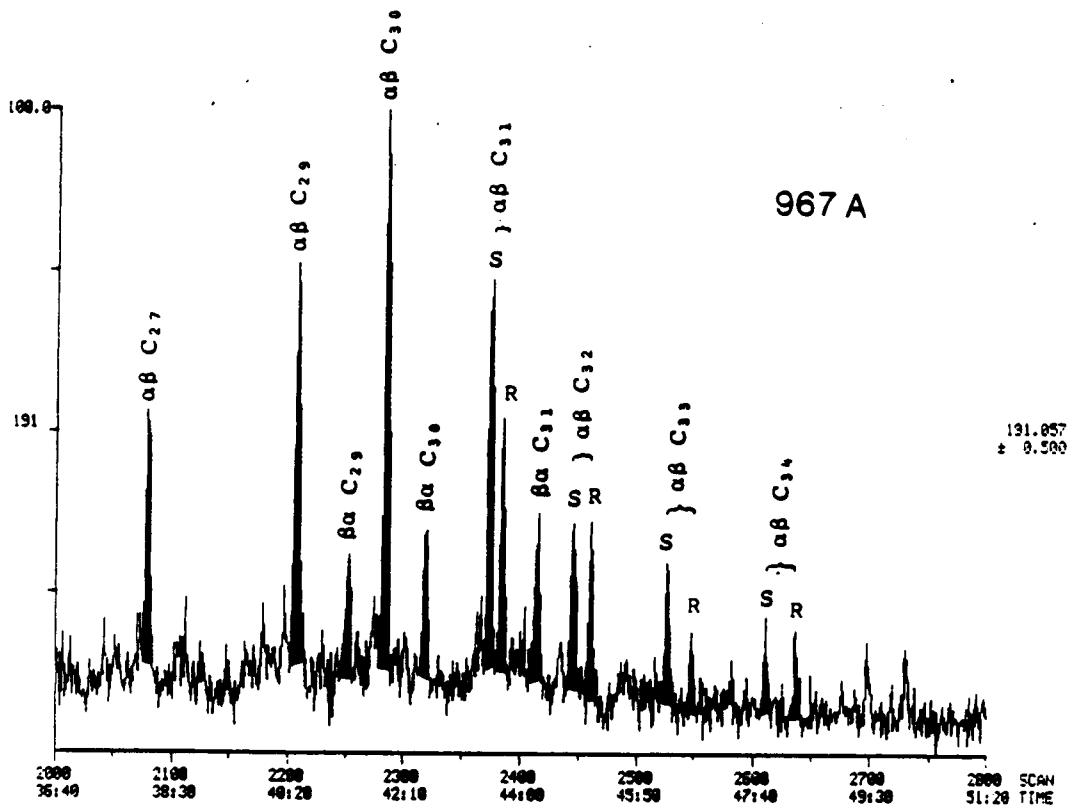
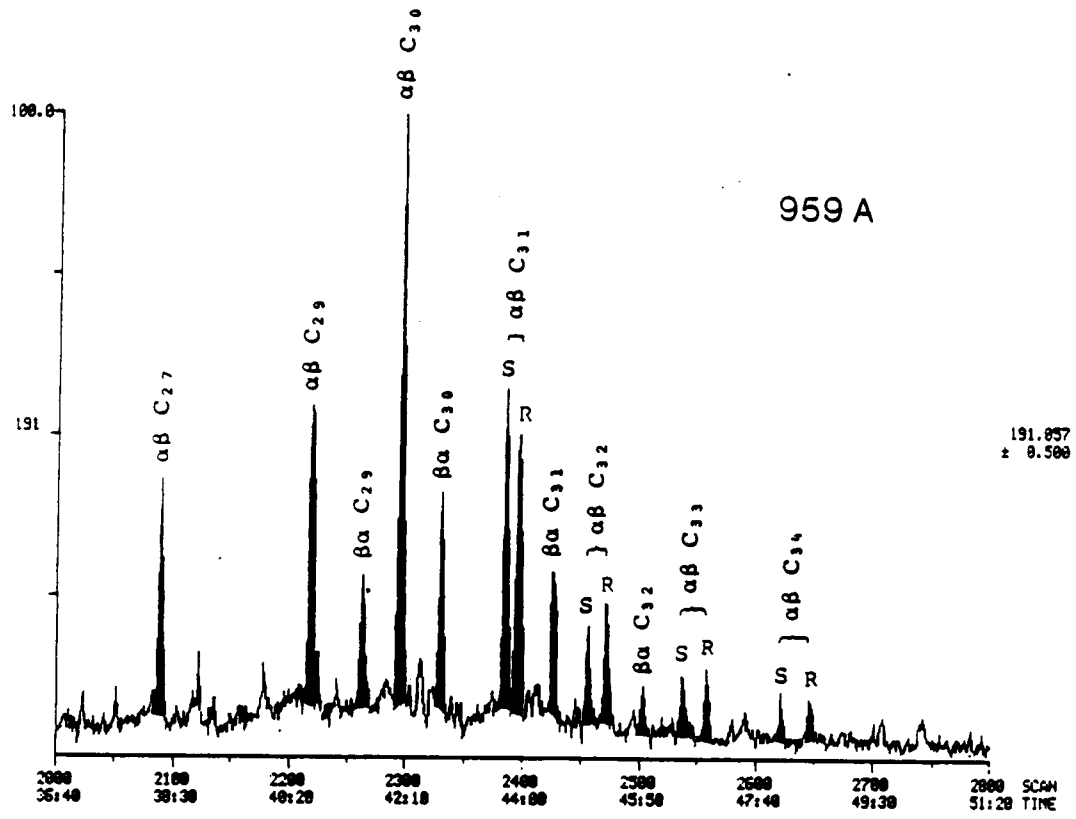


Fig. 11 a Examples of hopane fragmentograms (m/z = 191).

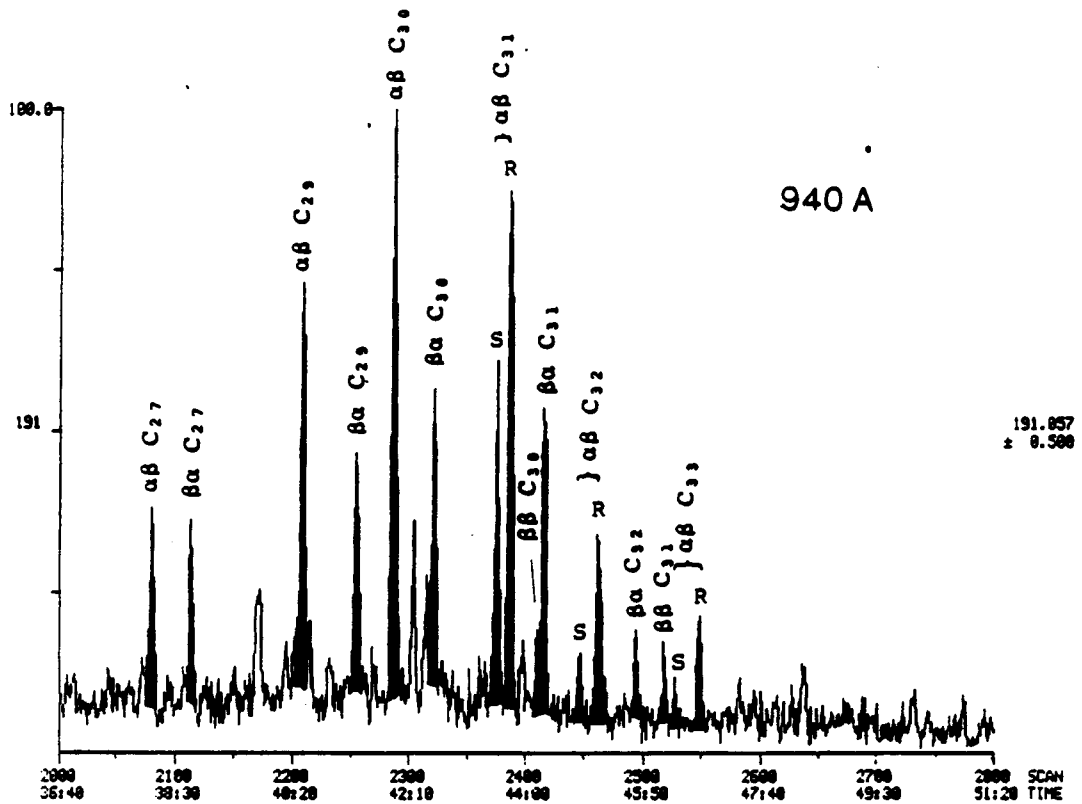
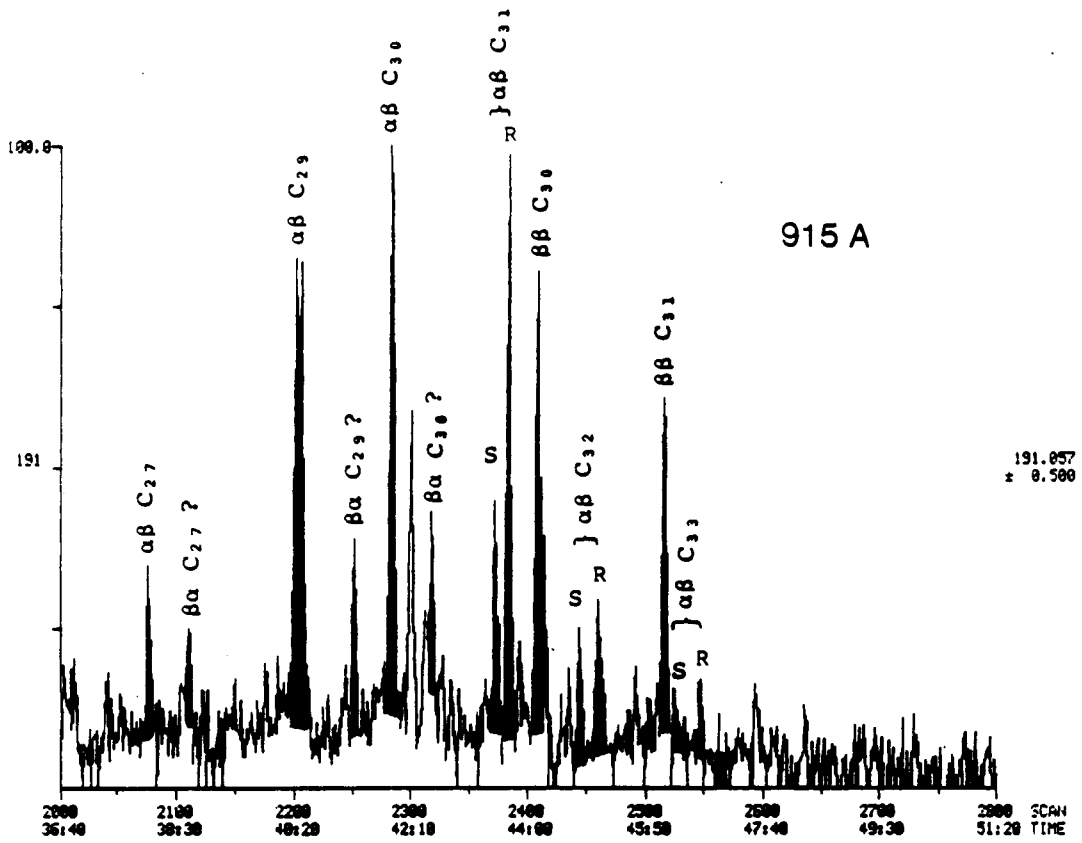


Fig. 11 b Examples of hopane fragmentograms ( $m/z = 191$ ).

measurements of the corresponding samples. The isomerisation ratios of the  $\alpha\beta$ -hopanes, on the other hand, seem to be in good agreement with the reflectance measurements. The equilibrium mixture of the S and R isomers is reached at approximately 8200' depth, equivalent to a vitrinite reflectance value of about 0.57 %R which indicates that the zone of hydrocarbon generation has not been reached. This is in good accordance with Seifert & Moldowan (1980) and Mackenzie & Maxwell (1981) as isomerisation may be completed before the zone of significant hydrocarbon generation is attained.



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## CONCLUSION

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The various methods used to assess the source rock potential and maturity of the investigated Mesozoic sediments indicate, that maturity seems to be reached at different depths.

The vitrinite reflectance measurement is the most widely used standard method always used for correlation of maturity parameters, though vitrinitic organic material itself is not oil prone. Whilst the other organic chemical methods mainly reveal the maturity of the oil prone organic matter. Thus discrepancies arise from the results of the different used techniques. An important factor is that relatively few data were available in this study. Biomarker analyses may be useful although isomerization of the hopanes did not help resolving maturity problems as the maturity of the sediments had passed the level where the final equilibrium mixture of the C-22 diastereoisomers of the  $\alpha\beta$ -hopanes was reached. Analyses of the steranes would be useful to elucidate the maturity conditions at this level, but the concentration of the steranes was too low for further investigations.

The type of the organic matter and its hydrocarbon potential were estimated by optical characterization of the organic composition and gas chromatographic analysis of the extracted bitumen combined with TOC-analyses. A combination of these methods helped in the recognition of reworked/oxidized, contaminated, biodegraded material, and to resolve seeming discrepancies in the gas chromatographic and microscopic results.

The composition and amount of organic matter indicate some sequences with potential source rock characteristics in the investigated Mesozoic sediments. These sediments may act as source rocks if sufficient maturity is attained.

Sediments with the most promising source rock charac-

teristics occur in the F-4 and upper F-3 Member of the Fjerritslev Formation. The organic content of these lagoonal and deep water marine sediments is relatively high in some horizons where it is mainly composed of liptinite (bituminite and alginite) normally regarded as good source material for oil. But according to the gas chromatograms some of the bituminite seen under the microscope (especially in the F-3 Member) may be of terrestrial origin which may lower the hydrocarbon potential.

Most of the other sediments (especially from the Vedsted and the lower part of the Fjerritslev Formation) are relatively unfavourable with respect to hydrocarbon generation, though the gas chromatograms show good quality material with respect to oil. However, under the microscope the main part of the organic matter is seen to be composed of reworked/oxidized terrestrial material with very limited hydrocarbon potential. Thus the potential for liquid hydrocarbons decreases in the sediments from the F-4 to the F-3 Member and downwards in the Fjerritslev Formation.

In the Vedsted Formation, only one sample with a high organic content composed of bacterial reworked organic matter may indicate an anoxic event with deposition of sediments with an increased hydrocarbon potential.

The principal source rocks of the North Sea graben structures are organic-rich "black" shales from the Kimmeridge Clay Formation and its lateral equivalents the Børglum Member of the Bream Formation and the J-4 unit (Barnard & Cooper 1981, Michelsen 1982, Cornford 1984). The equivalent Upper Jurassic sediments of the Bream Formation in this well do not show similar prominent source rock characteristics, though a higher proportion of liptinite (algal material) is observed.

Most of these sediments may be excluded as actual source rocks as maturity, according to the vitrinite reflectance measurements and assuming 0.60 %R as the threshold value for the top of the "oil window" is first reached at 8500', approximately at the top of the Gassum Formation. The base of the "oil window" at approximately 1.3 %R may, by extra-

polation of the reflectance trend, be predicted at 17,400' (presumably in Triassic redbeds). Thus the mature sediment sequences with respect to oil generation may be thinner and at slightly shallower depth in the central part of the Danish Subbasin than in the marginal areas (Lindgreen & Thomsen 1982). This is attributed to a slightly higher geothermal gradient and higher heat flow in the central part of the basin compared to the areas outside the basinal depocenter. While maturity seems to be reached in the deeper part of the well, no commercial accumulations of hydrocarbons have so far been encountered. The reasons for this may be numerous. The upper threshold values of the oil window should perhaps be set at higher  $R_o$ -values, as according to the extraction data (amount and composition of the organic matter) maturity may first be reached in the Vinding Formation. Alternatively, the terrestrial-influenced type of organic matter in the Gassum Formation has an unfavourable potential for oil, and/or the organic content is too low. However, generation and possible migration of gaseous hydrocarbons cannot be excluded here or elsewhere in the basin, as in the coal-measure sequences the TOC-values fluctuate considerably and organic rich sequences are likely (as they are known from other wells).

In the sediments of the Vinding Formation the type of organic matter seems to show a good potential for oil in some algal-rich horizons, but it is doubtful whether oil generation can reach commercial levels as the low organic content may be critically close to the lower limit for potential source rocks. Alternatively, hydrocarbon saturation and expulsion have not been reached and the maturity threshold value may be set at higher  $R_o$ -values. The situation may only be slightly different off-structure in this area as the F-4 and F-3 Member sequence according to seismic sections is found at approximately the same depth. But the thickness of and the depth to the Fjerritslev Formation is increasing towards the south east into the rimsyncline of the salt domes nearby.

The deeper part of the "oil window" seems to fall within

the continental redbed sequences and evaporites of the Triassic (Bertelsen 1980) which are unlikely source rocks.

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**ACKNOWLEDGEMENTS**

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In particular I wish to thank the staff of "Lehrstuhl für Erdöl und Kohle" (Aachen Technical University, F.R.G.), Prof. M. Wolf, H.W. Hagemann and W. Püttmann for their assistance and discussion of the results. E.Thomsen and P. Østfeldt Geological Survey of Denmark are thanked for their comments on a draft version of the text, J.R. Wilson (Geological Institute, University of Aarhus) kindly improved the English text.

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## APPENDIX

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### Lithological description

The lithologies of the cutting samples were examined and the main lithology of each sample (washed and dried cuttings) was estimated.

Observation of the glauconite and pyrite content was done in the polished specimens under the microscope.

### Optical coal petrographic methods

#### Vitrinite reflectance measurements

##### Introduction

In reflected light the maturity/rank of the organic matter in the sediments can be determined by reflectance measurements on the dispersed organic particles belonging to the maceral group of huminite/vitrinite. Huminite/vitrinite particles are used as they are the main maceral group in humic coals and also occur widely in sediments of different lithologies and facies; they also mature regularly. Care must be taken in the choice of particles for measurements (Bostick 1974, 1979). The dispersed organic matter in the sediments usually consists of a mixture of various types of autochthonous and reworked, or in other ways altered, products with a large reflectance range. Only the least altered particles represent the indigenous maturity of the rock and must be selected for measurements. In the huminite/vitrinite group many higher reflecting particles represent transitions

to the inertinite group (counted here as pseudovitrinites) giving too high reflectance values, while other very low reflecting particles belong to the bituminous or "A"-types of huminites giving too low reflectance values. As many low reflecting huminite/vitrinite grains as possible must be recorded in each sample (Stach et al. 1982, Lindgreen & Thomsen 1982). The measurements are presented in histograms and only the lower reflecting huminite/vitrinite are chosen as representing the autochthonous material.

Here some samples with coaly particles, whose reflectance results are unequivocal, are used as marker horizons to determine the realistic reflectance range.

Though huminite/vitrinite generally is not oil prone material, the huminite/vitrinite reflectance is now the most widely used optical technique in the petroleum industry for determining the maturity of sediments (Hunt 1979, Tissot & Welte 1984). Reflectance increases with increasing maturity, and the following stages can be distinguished (Tissot & Welte, 1984):

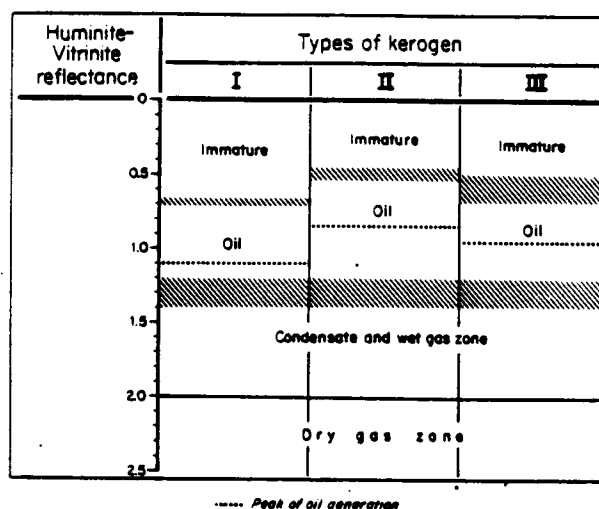
- a)  $\bar{R}_o < 0.5$  to  $0.7 \%$  diagenesis stage; source rock immature; Hydrocarbons scarce.
- b)  $0.5$  to  $0.7 \%$   $< \bar{R}_o$   $< \text{ca.} 1.3\%$  catagenesis stage; source rock mature; main zone of oil generation ("oil window").
- c)  $\text{ca.} 1.3 \%$   $< \bar{R}_o < 2 \%$  catagenesis stage; source rock post mature with respect to oil generation; zone of wet gas and condensate.
- d)  $\bar{R}_o > 2 \%$  metagenesis stage; methane remains as the only hydrocarbon; dry gas zone.

The boundaries of the oil and gas zones change slightly

according to time-temperature relationships and according to the composition (type) of organic matter (fig. A-1).

#### Sample preparation

Small cutting samples were sent to the Coal Petrographic



1. Fig. A-1: Approximate boundaries of the oil and gas zones in terms of vitrinite reflectance. Boundaries may change slightly according to the time-temperature relationship, and also to the various types of organic matter. Reprinted from Tissot & Welte (1984), with permission.

Laboratory, Danish Geological Survey, Copenhagen, for preparation of the specimens for microscopy.

The samples were imbedded in a cold setting synthetic resin, followed by grinding and polishing using 1/4 micron diamond powder for the final polish. All grinding and subsequent polishing stages were carried out using DP Lubricant Blue (Struers), since water causes swelling and disintegration of the clay content of some samples.

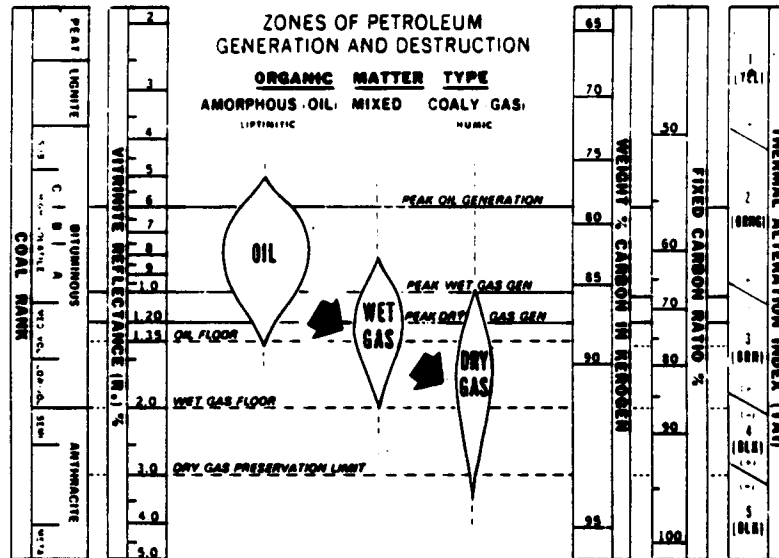


### Instruments

All polished sections were studied in reflected light by means of a "Zeiss Photomicroscope" equipped with a 40x "Epipol" oil immersion objective, an adjustable lamp current stabilizing transformer with a 100 W halogen lamp, filters for green light (546 nm) and a MP03 photomultiplier with digital read-out, connected to a BBC computer. The photomultiplier was calibrated on the microscopy by measurements on a polished optical glass standard of constant known reflectance. The measured field was about 2 microns in diameter.

### Qualitative description of the organic matter

The type of organic matter influences the quality and quantity of the hydrocarbons which may be generated if sufficient maturity is reached. Liptinitic plant material (especially of phytoplankton origin) shows a good potential for oil



2. Fig. A-2: Correlation scheme of vitrinite reflectance and other maturity parameters with the main stages in the generation of oil and gas.

The importance of the type of organic matter for the resulting hydrocarbon product is illustrated for liptinitic, mixed and humic type of organic matter. Reprinted from Dow (1977a), with permission.

while humic material (huminite/vitrinite) derived from terrestrial plants generally has a main potential for gas. Reworked/oxidized material (inertinite, pseudovitrinite) shows a very limited or no potential for gas. This is illustrated in fig. A-2 (Dow 1977a) (p.37).

The type of organic matter is rated qualitatively by reflected light microscopy, supplemented with observations on blue-light induced fluorescence performed on the same polished mounts as used for reflectance analysis. In the description of the organic matter (see tabel 7) special attention was given to the composition and fluorescence colours of the liptinite group.

The nomenclature is in accordance with Stach et al. (1982) though in this study "huminite" and "vitrinite" are used parallel, as precise distinction between brown or bituminous coal rank particles is impossible in sediments.

The organic particles show much larger scattering in reflectance in sediments than in coals. In sediments a continuous reflectance and morphological sequence exists between particles of the huminite/vitrinite group due to extensive reworking and oxidation (Bostick 1974).

These particles with somewhat higher reflectance than true huminite/vitrinite are termed pseudovitrinite in this study, although in a coal petrographic sense most of them might belong to the inertinite group.

## Spectral fluorescence measurements

### Introduction

If reflectance measurements on huminite/vitrinite particles are impossible (absent, rare or mainly represented by reworked particles) the fluorescence properties of the liptinite group can be used as a maturity/rank parameter. Liptinite

components show a characteristic change in fluorescence properties with increasing rank/maturity which causes the colour spectrum to change progressively: The wavelength range of the fluorescence light moves from green-yellow towards orange and red simultaneously with decreasing fluorescence intensity. The visible fluorescence of liptinites disappears as a maturity corresponding to the end of the oil generation zone is reached (van Gijssel 1981, Ottenjann et al. 1974, Teichmüller & Ottenjann 1980, 981/1982, Teichmüller & Durand 1983). The fluorescence spectra can be quantified and several parameters are used as rank/maturity indicators.

Spectral fluorescence measurements are usually performed on liptinites from polished surfaces of whole rock samples. As a new analytical method, fluorescence measurements have been carried out on extracted bitumens (i.e. the soluble part of the organic matter) since the fluorescence properties of the extracts change in a similar way to the macerals of the liptinite group (Hagemann & Hollerbach 1980, 1981a, 1981b).

In this study this new method has been applied to some selected samples. The mean spectra (average of 10 spectral measurements) were quantified and the following parameters were used as rank/maturity indicators:

- 1) The red/green ratio, i.e. the ratio between the relative intensities at 650 nm and at 500 nm (the so-called Q-values) were calculated.

- 2) The position of the maximum of the spectrum ( $\lambda_{\max}$ ) was determined.

- 3) The quantitative coefficients of the corresponding fluorescence colours, the so called trichromatic coordinates (Chromaticity coefficients) x,y of the ICI system (International Commission of Illumination) were computed.

Very small differences in colour are detected by this calculation as intensities over the entire spectrum (420-700 nm wavelength) account for the trichromatic coordinates.

The trichromatic coordinates are plotted graphically in a two dimensional diagram (DIN 6164 for the illumination D65) see page 20 for an explanation.

### Sample preparation

The fluorescence analyses were carried out at the "Lehrstuhl für Erdöl und Kohle", Aachen Technical University, West Germany.

The spectral fluorescence measurements were performed on small amounts of the extracted bitumens (Soxhlet extracted with  $\text{CH}_2\text{Cl}_2$ ) from the same samples as used for the chemical analysis.

In order to get comparable results by quantification of the spectral curves, a precise concentration of extract was adsorbed on small glass slides covered with Kieselguhr G (Merck).

Small amounts of the extract were dissolved in 20  $\mu\text{l}$ . toluene/methanol (3:1) per milligram extract.

Kieselguhr G has a very low blank fluorescence value and there is no chromatographic effect for the selection of any component from the extract solution, with the exception of high asphaltic compounds. After careful evaporation of the solvent mixture under vacuum, the glass slides with the extracts are ready for the fluorescence measurements (Hagemann & Hollerbach 1981).

Measurements were started after adjusting the optical system of the microscope and the photoelectric assembly and were performed on small uniform fluorescing diatoms. In this study the alteration effect (alteration = change of fluorescence during irradiation) has not been taken into account. To achieve the minimum alteration effect, focussing is done as quickly as possible. Ten spectral curves are measured for each sample and the mean spectrum calculated, which was used for quantification of the maturity parameter.

### Instruments

The Zeiss microscope was equipped with a MPN 01K photometer, a reflected-light condenser III-RS with excitation filter G 365, chromatic beam splitter FT 395 and barrier filter LP 397, a Neofluar 16/0.40 T objective, stabilized superpres-

sure discharge lamp HBO 100 as light source for fluorescence excitation and a grating monochromator registering the spectral range from 420 to 700 nm in single steps of 5 nm. The measured field was 0.030 mm in diameter. The microscope was attached to and controlled by a HP-85 computer which also recorded the spectra. In order to obtain the real spectrum the registered fluorescence intensities were corrected for several distorting effects caused by the photometric equipment (i.e. all optical and electrical parts). In this study a stabilized tungsten-filament lamp with a flat coil 60 W, 10.1 V, colour temperature 2850°K was used as comparison lamp to obtain the correction factors. The correction factors for the spectral curves were calculated based on Planck's law of radiation (Piller 1978).

#### Organic geochemical methods

##### Total organic carbon (TOC)

The amount of organic matter is estimated by organic carbon analysis.

A minimum content of organic matter is required for a sediment (under otherwise favourable conditions) to be a source rock. Generally 0.5 % organic carbon (TOC) has been accepted for shales (0.3 % for carbonates) to be the critical lower limit, though in many laboratories the limits are set at higher values (Tissot & Welte 1984) as the hydrocarbon potential is only affected by the percentage of carbon which is hydrocarbon prone.

The analytical laboratory technique involves combustion of the organic carbon to CO<sub>2</sub> in an oxidizing atmosphere in a Leco induction furnace, followed by quantitative detection of the CO<sub>2</sub> formed and calculation of weight percent total organic carbon.

The analyses were performed on all finely ground rock samples (ca. 2 g) which had been treated with 10 % HCl and

washed with distilled water to remove any carbonates present (Tissot & Welte 1984).

## Extractable organic matter (EOM) and chromatographic separation

### Introduction

Chemical analyses were performed on the extractable part of the organic matter (EOM) - also called bitumen.

The amount of bitumen in the organic matter depends on the maturity as bitumen is generated from the kerogen during thermal degradation. During maturation the composition of the bitumen changes. The amount of saturated hydrocarbons in the bitumen increases as, to a lesser degree, does the amount of aromatic hydrocarbons, while heterocompounds (N, S, O compounds) decrease in concentration.

Thus the amount of the extractable organic matter (mg EOM/g TOC) and the hydrocarbon ratios (mg HC/g TOC) can be used as maturity indices.

Maturity is indicated by a pronounced increase in the bitumen and hydrocarbon content, but variations in the amounts of extracts and their composition are also influenced by the type of the primeval plant material. Continental plant material generally generates smaller amounts of bitumens and hydrocarbons than marine or limnic predecessors.

Extractability values may also be influenced by contaminating materials from processing the well and handling the cutting materials. Pronounced high values may indicate migrated hydrocarbon materials.

### Extraction

Samples were selected for extraction on the basis of the TOC determinations and the qualitative description of the microscopic organic matter. About 50 g finely ground material

was Soxhlet extracted with dichloromethane ( $\text{CH}_2\text{Cl}_2$ ) for 48 h at 45 °C.

Sulphur was removed by activated Cu-sheets in the flasks. The solvent was distilled before use; all extraction thimbles and filter papers were pre-extracted with pure solvents to remove any soluble component. After extraction the solvent was removed at reduced pressure and temperature (not exceeding 30°C) on a Rotavapor rotary evaporator. The extract was transferred, with small amounts of solvent, to small bottles. The amount of the extractable organic matter (EOM) was determined after the solvent had been completely evaporated at room temperature.

#### Chromatographic separation

The extractable organic matter (EOM) was fractionated by column chromatography. The column was packed with solvent washed and preactivated (1 h at 110°C) silica gel (Merck Kieselgel 60), and the upper 1/3 was packed with alumina. Small portions of EOM (ca. 5 mg) were dissolved in a few drops of  $\text{CH}_2\text{Cl}_2$ , adsorbed on alumina by gentle evaporation and transferred to the top of the column. Fractions of increasing polarity were eluted by pouring increasingly polar solvents through the column. About 5 ml of each solvent was used. The eluants were collected in small bottles and the various fractions, the saturated hydrocarbons (eluted with n-hexane), the aromatic hydrocarbons (eluted with dichloromethane), and the NSO (polar nitrogen-, sulphur-, and oxygen containing) compounds (eluted with methanol) were determined after evaporation of the solvents at room temperature. The remaining part of the extract includes asphaltenes (retained on the column) and evaporative losses during handling. The weights of the different fractions were expressed in percent.

## Gas chromatography

### Introduction

The extractable organic matter includes biomarker molecules which carry information about the type and origin of the organic matter. (For the importance of the type of organic matter with respect to the quality and quantity of the hydrocarbons generated during catagenesis, see p. 64). These can be detected by gas chromatographic analyses of the saturated hydrocarbon fraction of the bitumens. Thermally induced changes in the organic matter are reflected in the gas chromatograms and some indices can be used to assess the maturity of the organic matter.

In the gas chromatograms from immature extracts, differences in n-alkane distribution patterns can be attributed to the origin of the organic matter as there are fundamental differences between the chemical composition of marine planktonic algae and terrestrial higher plants. Long chained n-alkanes are derived from lipids (especially cuticular waxes) of higher plants. They show a strong predominance of odd carbon numbered over even carbon numbered n-alkanes, especially apparent from n-C<sub>23</sub> to n-C<sub>35</sub> with a strong predominance of n-C<sub>27</sub>, n-C<sub>29</sub> and/or n-C<sub>31</sub><sup>23</sup> (Hunt 1968, Tissot & Welte 1984). Shorter chained n-alkanes in the low-medium molecular weight range are derived from marine algae, with only slight predominance of odd-numbered n-alkanes and especially prominent n-C<sub>15</sub> and/or n-C<sub>17</sub>. With increasing thermal maturation n-alkanes of decreasing chain lengths are formed, principally within the oil generation zone. Especially in terrestrial organic matter a heavy-end bias gradually gives way to a light-end bias in corresponding gas chromatograms, so that the odd n-alkanes of biogenic origin become progressively diluted by thermally new generated n-alkanes without carbon number predominances. The odd-dominance is thereby lowered and the chromatograms tend to become alike in well mature sediments. This odd-dominance can be quantified by the so called Carbon Preference-Index CPI (which



stands for the ratio of odd- to even chain length n-alkanes in a specific high molecular weight range) and used as a maturity index (Bray & Evans 1961). In recent sediments the CPI-index is about 2-2.5 or even higher. With increased maturity the CPI-index is lowered and gradually approaches 1.0 when peak oil generation is reached (Tissot & Welte 1984).

The pristane/n-C<sub>17</sub> ratio has also been used as a maturity indicator (Tissot et al. 1971) as well as an environmental indicator (Lijmbach 1975).

As a function of n-alkane generation by thermal processes the pristane/n-C<sub>17</sub> ratio decreases with increasing maturity (Tissot et al. 1971). The ratio is also highly dependent on the original input of organic matter and the redox potential of the depositional environment, and sensible to biodegradation. Marine algal material with a prominent n-C<sub>17</sub> peak will lower the pristane/n-C<sub>17</sub> ratio, while in a highly oxygenated environment pristane generation will be favoured and thus increase the pristane/n-C<sub>17</sub> ratio. Lijmbach (1975) proposed a high pristane/n-C<sub>17</sub> ratio in peat swamp environments and a low ratio in aquatic environments.

The acyclic isoprenoids pristane and phytane can be used as environmental indicators. Pristane and phytane are considered to be end products of the diagenesis of the phytol side chain of chlorophyll, their relative abundance depending on the local environment (Powell & McKirdy 1975, Brooks 1969). In oxidizing environments phytol is mainly transformed to pristane while reducing environments favour the generation of phytane. Thus differences in the pristane/phytane ratios (typically > 3) indicate oxidizing environments of land derived organic matter or organic matter which has passed through an oxygenated phase during decomposition while low pristane/phytane ratios (typically < 1.5) indicate reducing environments with anaerobic decay.

The pristane/phytane ratio is influenced by thermal processes as pristane is progressively generated during the late stages of diagenesis and early stages of catagenesis. As a result the pristane/phytane ratio increases slightly

during maturation until the cracking zone is reached then it decreases (Tissot & Welte 1984). Maturation processes do not readily affect the pristane/phytane ratio to such an extent that original palaeoenvironmental information is obscured (Didyk et al. 1978).

### Instruments

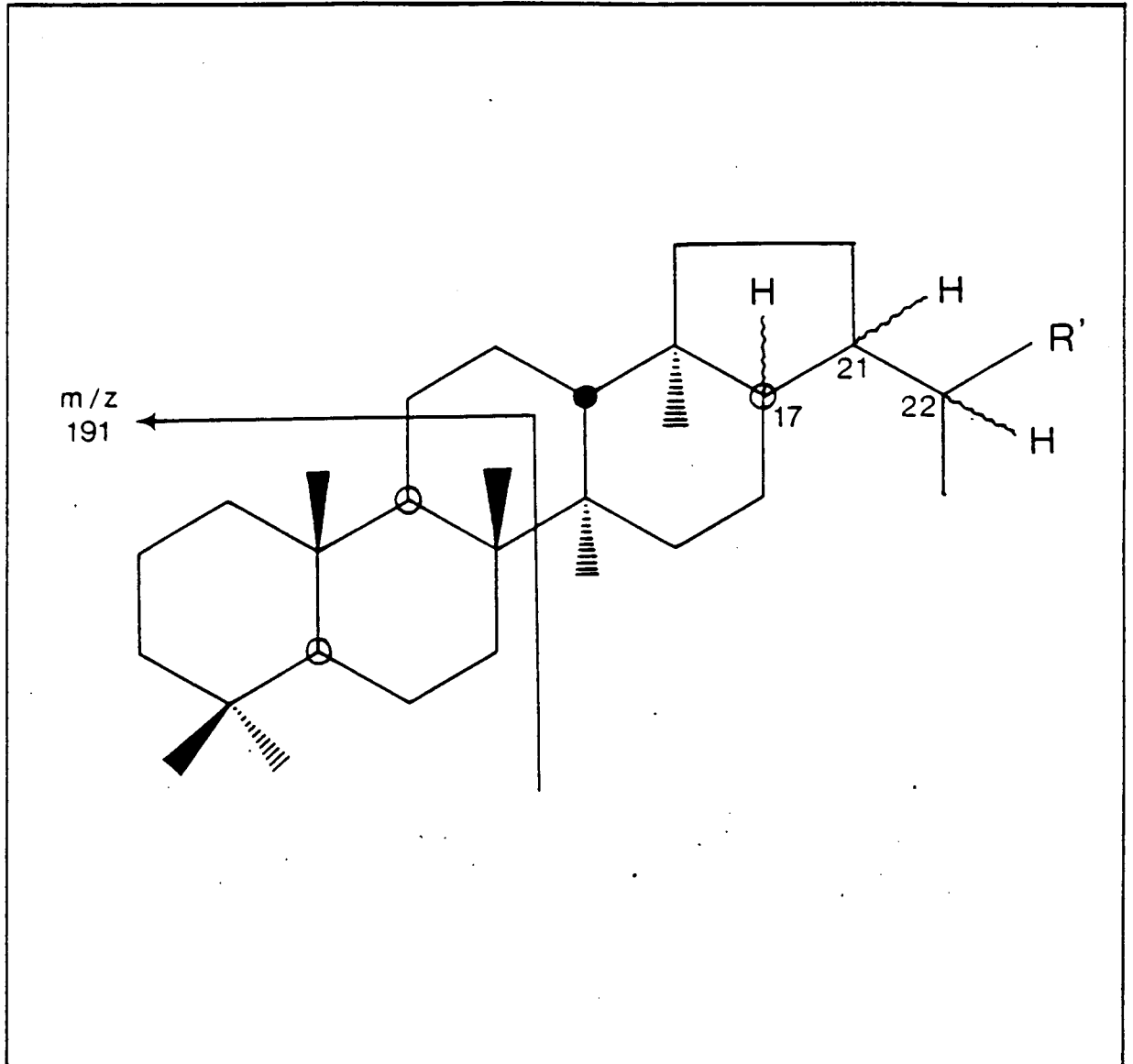
Gas chromatographic analysis of the saturated fractions (and for control also some aromatic hydrocarbon fractions) were performed on a HRGC 5160 Mega Series (Carlo Erba) instrument equipped with a 50 m glass-capillary SE 54 Column with H<sub>2</sub> as carrier gas using the split injection technique. The effluent from the column was detected with a Flame Ionisation detector (FID). The column temperature was increased from 100° to 300°C at a rate of 4°C/min and subsequently maintained at 300°C until the analysis was completed. Relative concentrations of the n-alkanes and the isoprenoids pristane and phytane a.o. were found by integrating the peak areas on the gas chromatograms.

### Gas chromatography - Mass spectrometry (GC-MS)

#### Introduction

Some samples (i.e. the saturated hydrocarbon fraction) were selected on the basis of the gas chromatograms for further investigation of the biomarkers with computerized GC-MS.

Biomarkers are any organic compounds detected in the geosphere whose carbon skeletons have survived in a recognizable form through the process of early sediment diagenesis (Mackenzie 1984). Thus they can be linked to some major structural types known from biological molecules. In their structure they often contain several chiral centers i.e. atoms around which alternative spatial arrangements of attached groups result in different stereoisomers. These



3. Fig. A-3: General hopane structure ( $c \geq 29$ ) with the key fragment  $m/z = 191$  of all hopanes indicated.  $R'$  means the alkyl side chain  $R' = C_n H_{2n+1}$  or  $R' = H$ .

A solid/open dot represents a carbon-hydrogen bond pointing out/into the plane of paper. A solid/dotted triangle represents a carbon-carbon bond above/below the plane of paper and a sinuous line means that both the latter possibilities exist.

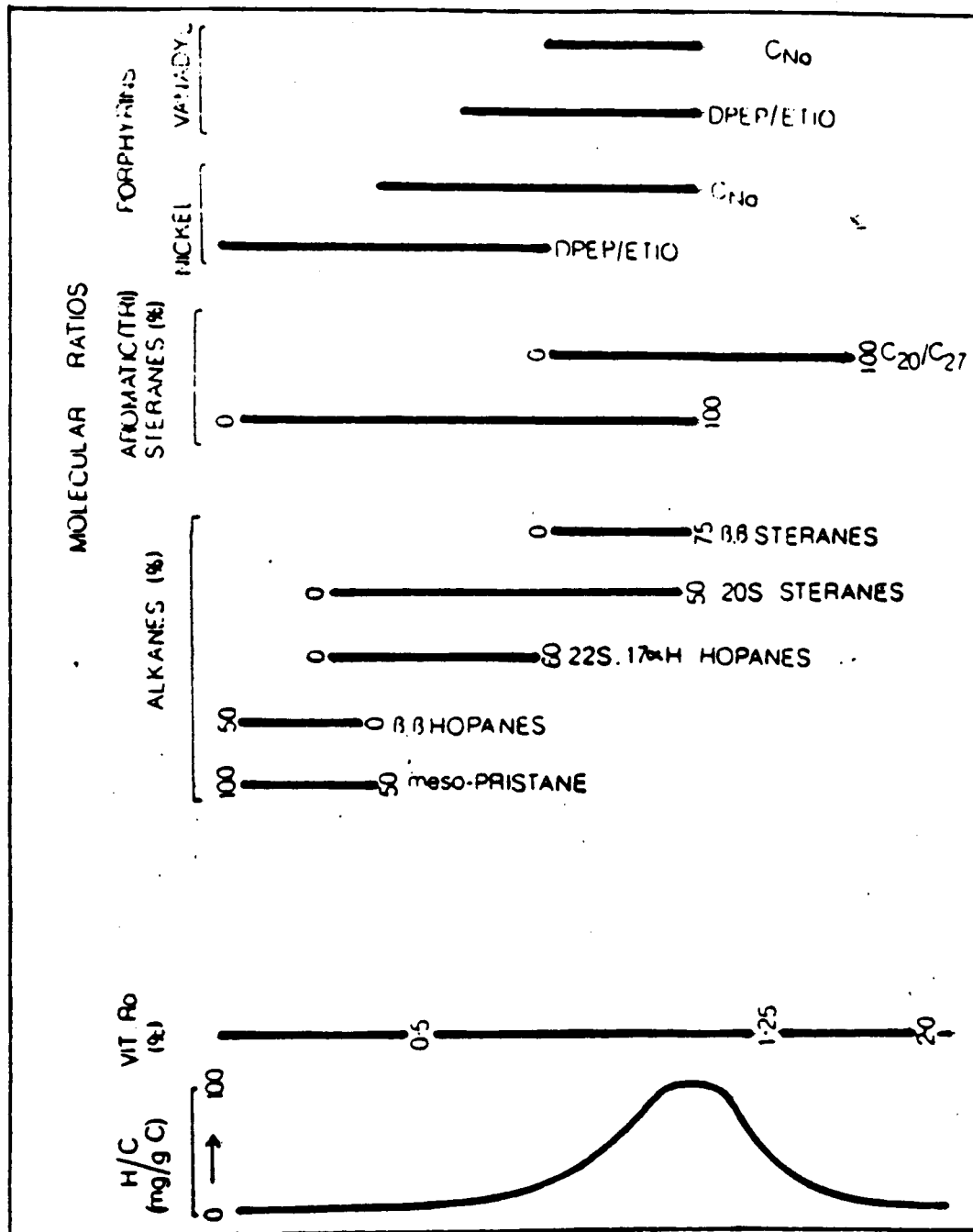
stereoisomers have lightly different chemical and physical properties and can be distinguished in GC-MS by their different mass spectra and/or retention times. During sediment burial many of the biological stereoisomers become thermally unstable and adjust to more stable structures. These thermally induced changes in the stereochemistry of some biomarkers (e.g. hopanes, steranes, meso-pristane) can be quantified and used as maturity indices.

Selective ion monitoring was used for investigation of the biomarkers.

In this study most information was obtained from the GC-MS investigation of the hopane group (pentacyclic triterpenoids). The hopanes are one of the most abundant molecular groups of the biomarkers, reflecting their bacterial origin (Ourisson et al. 1979, 1984). Thus hopanes are found in almost all sediments (and petroleum) which are sufficient immature to have allowed preservation. All hopanes possess the same pentacyclic triterpenoid structure which only vary in the length of their alkyl side chains and stereochemistry: the molecular structure include possibilities for  $\alpha$  or  $\beta$  H atoms at C-17 and C-21 and R or S configuration at C-22. The key fragment  $m/z$  191 of all hopanes is indicated in fig. A-3.

It has been shown (Seifert & Moldowan 1980) that the hopanes in living organisms and immature sediments are typically 17  $\beta$ H, 21  $\beta$ H hopanes occurring as a single epimer at C-22 (22R). The process of thermal maturation affects conversion to some 17  $\beta$ H, 21  $\alpha$ H compounds (moretanes) but mainly 17  $\alpha$ H, 21  $\beta$ H hopanes (which are more stable) while the 17  $\beta$ H, 21  $\beta$ H hopanes disappear (Ensminger & Albrecht 1978). At higher levels of maturity the 17  $\beta$ H, 21  $\alpha$ H moretanes are also converted into the thermodynamically most stable 17  $\alpha$ H, 21  $\beta$ H configuration with the two C-22 diastereoisomeric pairs (22R and 22S) in the extended hopane members  $\geq C_{31}$  (Seifert & Moldowan 1980) (See fig. A-4).

The epimerisation at the C-22 position takes place at a



4. Fig. A-4: Ranges of individual molecular measurements for thermal maturation, plotted against the down-hole hydrocarbon generation curve and vitrinite reflectance-values ( $R_0$ ). (Toarcian shale, Paris Basin and Pliensbachian shale, N.W. Germany). Reprinted from Mackenzie (1984), with permission.

slower rate than conversions to 17 $\alpha$ H, 21 $\beta$ H hopanes. The 22S isomer is thermodynamically slightly more stable than the 22R isomer and a final 60:40 mixture of S and R is achieved before the onset of intense hydrocarbon generation, as has been shown for the Paris Basin (Mackenzie & Maxwell 1981). The hopane distributions in mature sediments and petroleum are thus characterized by high relative abundance of the stable 17 $\alpha$ H, 21 $\beta$ H configuration with mixtures of the C-22 isomers in members  $\geq C_{31}$  (e.g. van Dorsselaer et al. 1974).

The ratio of the C-22 diastereoisomers in the 17 $\alpha$ H, 21 $\beta$ H hopane series was calculated by measuring the appropriate peak area for each component over the range  $C_{31}$ - $C_{34}$  in mass fragmentograms of m/z 191. The ratio, expressed as an average of the ratios for each carbon number in the range (Mackenzie 1981), has been used as maturity index.

#### Instruments

The samples were analysed on a Varian 3700 gas chromatograph fitted with a quartz capillary column (20 m x 0.25 mm i.d.) with SE 54 as stationary phase. Helium was used as a carrier gas (head pressure 0.5 bar). The temperature was programmed from 100-300°C at 4°C/min and held at 300°C for 15 minutes.

The gas chromatograph was interfaced directly (open interface) with a Finnigan Mat 8200 mass spectrometer (electron energy 70 eV, filament current 1 mA). The acquired data were registered in an Incos-system. The mass spectra were recorded cyclically with 1.1 sec. pr. scan.

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**TABELS**

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Table 1 Stratigraphy, lithology and total organic carbon (TOC)

Sample no.	Lithostratigraphy	Depth below KB	Main cutting lithology	TOC %
909A	Vedsted Formation (V-4 Mb.)	4700'	light-medium grey siltstone	0.55
910A	- - -	4760'	- -	0.19
911A	- - (V-3 Mb.)	4820'	- -	0.48
912A	- - -	4880'	dark-grey-black silty claystone	1.06
913A	- - -	4940'	- -	1.19
914A	- - -	5000'	dark grey shaly claystone	1.33
915A	- - (V-2 Mb.)	5060'	- -	1.13
916A	- - -	5120'	- -	2.71
917A	- - -	5180'	- -	1.01
918A	- - (V-1 Mb.)	5240'	- -	1.08
919A	- - -	5300'	- -	1.33
920A	- - -	5360'	- -	1.14
921A	- - -	5420'	- -	1.09
922A	- - -	5480'	- -	0.88
923A	- - -	5540'	medium grey claystone	1.28
924A	Bream Formation (Fred. Mb.)	5600'	- -	1.19
925A	- - -	5660'	- -	0.92
926A	- - -	5720'	- -	0.75
927A	- - -	5780'	brownish grey silt/sandstone	0.77
928A	- - -	5840'	- -	1.32
929A	- - -	5900'	- -	0.88
930A	- - -	5960'	- -	0.73
931A	- - -	6020'	medium grey silty claystone	0.56
932A	- - (Børgl. Mb.)	6080'	medium grey claystone	0.80
933A	- - -	6140'	- -	0.95
934A	- - -	6200'	- -	1.16
935A	Haldager Formation (Flyvbj. Mb.)	6250'	gray claystone and light sandstone, coaly particles	0.86



Table 1 Stratigraphy, lithology and total organic carbon (TOC) - continued

Sample no.	Lithostratigraphy	Depth below KB	Main cutting lithology	TOC %
936A	Fjerritslev Formation (F-4 Mb.)	6310'	light sandstone with coaly particles	0.83
937A	- - -	6370'	dark grey-black fissile shale	1.95
938A	- - -	6430'	- -	1.20
939A	(F-3 Mb.)	6490'	- -	1.17
940A	- - -	6550'	- -	1.52
941A	- - -	6610'	- -	1.21
942A	- - -	6670'	- -	1.18
943A	- - -	6730'	- -	0.91
944A	- - -	6790'	- -	1.07
945A	- - -	6850'	- -	1.35
946A	- - -	6910'	grey and brown shaly claystone	1.17
947A	- - -	6970'	- -	1.02
948A	- - -	7030'	- -	0.96
949A	- - -	7090'	- -	1.01
950A	- - -	7150'	- -	1.07
951A	- - -	7210'	grey shaly claystone	1.33
952A	(F-2 Mb.)	7270'	- -	1.22
953A	- - -	7330'	- -	1.17
954A	- - -	7390'	- -	1.14
955A	- - -	7450'	- -	1.15
956A	- - -	7510'	- -	÷
957A	(F-1 Mb.)	7570'	dark grey shale	1.06
958A	- - -	7630'	- -	1.08
959A	- - -	7690'	- -	1.05
960A	- - -	7750'	- -	1.06
961A	- - -	7810'	- -	0.92
962A	- - -	7880'	- -	0.97
963A	- - -	7930'	- -	1.00
964A	- - -	7990'	- -	1.02
965A	- - -	8050'	- -	1.25
966A	- - -	8110'	- -	1.02

Table 1 Stratigraphy, lithology and total organic carbon (TOC) - continued

Sample no.	Lithostratigraphy	Depth below KB	Main cutting lithology	TOC %
967A	Fjerritslev Formation (F-1 Mb.)	8170'	dark grey shale with some coarse quartz grains	1.13
968A	- - -	8230'	dark grey shale with some coarse quartz grains and dark fine laminated sand/siltstone with coaly stringers	÷
969A	- - -	8290'	- -	0.85
970A	- - -	8350'	dark laminated sand/siltstone with coaly stringers and dark shale	0.89
971A	- - -	8410'	- -	0.89
972A	Gassum Formation	8470'	- -	1.01
973A	- -	8530'	grey shale, coarse grained sandstone with coaly particles	1.22
974A	- -	8590'	- -	1.09
975A	- -	8650'	- - besides some carbonate cutting	0.92
976A	- -	8710'	dark grey shale and yellowish fine grained sandstone with coaly particles	1.11
977A	- -	8770'	- -	0.98
978A	- -	8830'	light sandstone, red-green mottled claystone, dark claystone	0.71
979A	- -	8890'	- -	0.55
980A	- -	8950'	light coarse grained sand and siltstone, dark shale, some mottled red-green claystone	0.42
981A	- -	9020'	- -	0.68
982A	Vinding Formation	9080'	dark shale, some sand/siltstone with coaly stringers, carbonates	0.82

Table 1 Stratigraphy, lithology and total organic carbon (TOC) - continued

Sample no.	Lithostratigraphy	Depth below KB	Main cutting lithology	TOC %
983A	Vinding Formation	9140'	dark grey and brownish shale, dark mottled carbonates	0.73
984A	- -	9200'	- -	0.75
985A	- -	9260'	dark grey-black brownish shale, dark mottled carbonates	0.56
986A	- -	9320'	- -	0.76
987A	Oddesund Formation	9380'	light grey claystone and siltstone, few carbonates	0.70
988A	- -	9440'	- -	0.54
989A	- -	9460'	- -	0.67

Table 2 Vitrinite reflectance measurements

Sample no.	Depth below KB	% $\bar{R}_O$	Number of measurements
909A	4700'	0.45	18
910A	4760'	0.48	9
911A	4820'	0.44	31
912A	4880'	0.46	49
913A	4940'	0.45	56
914A	5000'	0.45	32
915A	5060'	0.47	19
916A	5120'	0.48	44
917A	5180'	0.46	11
918A	5240'	0.47	9
919A	5300'	0.45	14
920A	5360'	0.46	16
921A	5420'	0.47	11
922A	5480'	0.45	16
923A	5540'	0.46	26
924A	5600'	0.47	17
925A	5660'	0.47	13
926A	5720'	0.48	20
927A	5780'	0.42	16
928A	5840'	0.47	20
929A	5900'	0.44	25
930A	5960'	0.45	17
931A	6020'	0.47	28
932A	6080'	0.46	19
933A	6140'	0.50	33
934A	6200'	0.50	27
935A	6250'	0.50	37
936A	6310'	0.49	78
937A	6370'	0.49	55
938A	6430'	0.49	46
939A	6490'	0.49	47
940A	6550'	0.50	35
941A	6610'	0.52	23
942A	6670'	0.48	29
943A	6730'	0.51	46

Table 2 Vitrinite reflectance measurements - continued

Sample no.	Depth below KB	% $\bar{R}_O$	Number of measurements
944A	6790'	0.48	27
945A	6850'	0.51	37
946A	6910'	0.51	25
947A	6970'	0.50	17
948A	7030'	0.53	56
949A	7090'	0.54	58
950A	7150'	0.53	30
951A	7210'	0.54	42
952A	7270'	0.52	54
953A	7330'	0.54	52
954A	7390'	0.54	68
955A	7450'	0.54	36
956A	7510'	0.55	44
957A	7570'	0.56	24
958A	7630'	0.57	12
959A	7690'	0.57	37
960A	7750'	0.56	37
961A	7810'	0.58	47
962A	7870'	0.58	56
963A	7930'	0.58	62
964A	7990'	0.60	54
965A	8050'	0.55	82
966A	8110'	0.57	56
967A	8170'	0.57	31
968A	8230'	0.61	27
969A	8290'	0.59	49
970A	8350'	0.60	62
971A	8410'	0.60	63
972A	8470'	0.63	46
973A	8530'	0.63	81
974A	8590'	0.62	21
975A	8650'	0.62	74
976A	8710'	0.63	65
977A	8770'	0.62	53
978A	8830'	0.63	20

Table 2 Vitrinite reflectance measurements - continued

Sample no.	Depth below KB	% $\bar{R}_O$	Number of measurements
979A	8890'	0.62	23
980A	8950'	0.67	33
981A	9020'	0.62	45
982A	9080'	0.63	66
983A	9140'	÷	÷
984A	9200'	0.63	16
985A	9260'	0.67	33
986A	9320'	÷	÷
987A	9380'	0.62	27
988A	9440'	0.64	18
989A	9460'	0.66	41

Table 3 Data of spectral fluorescence measurements on the organic extracts and corresponding reflectance values.

Sample no.	red/green ratio (Q-value)	$\lambda_{\max}$ (nm)	trichromatic coordinates		$\bar{R}_O$
			x	y	
913A	0.86	565	0.3694	0.4424	0.45
915A	0.44	540	0.3161	0.4192	0.47
916A	0.53	505	0.3019	0.3739	0.48
923A	0.31	520	0.2853	0.3858	0.46
929A	0.52	545	0.3318	0.4318	0.44
932A	0.66	550	0.3526	0.4461	0.46
936A	0.67	545	0.3449	0.4325	0.49
937A	1.20	580	0.3861	0.4378	0.49
938A	1.06	570	0.3797	0.4390	0.49
940A	1.22	575	0.3911	0.4460	0.50
945A	1.46	580	0.4090	0.4548	0.51
954A	1.66	590	0.4203	0.4565	0.54
957A	0.99	565	0.3861	0.4641	0.56
959A	1.03	575	0.3910	0.4645	0.57
967A	1.23	580	0.4010	0.4624	0.57
973A	1.73	590	0.4241	0.4629	0.63
976A	1.44	585	0.4092	0.4587	0.63
982A	1.06	575	0.3864	0.4491	0.63
985A	1.14	570	0.3994	0.4666	0.67

Table 4 Extractable organic matter (EOM) and composition of chromatographic fractions.

Sample no.	Sediment extracted (g)	EOM ppm	mg EOM /g TOC	mg HC /g TOC	sat HC %	arom HC %	total HC %	non HC %
913A	47.21	180	15.1	6.6	19	25	44	56
915A	55.37	220	19.5	9.4	24	24	48	52
916A	59.32	912	33.7	9.1	7	20	27	73
923A	49.87	900	70.3	41.5	34	25	59	41
929A	41.22	197	22.3	5.8	15	11	26	74
932A	39.05	213	26.6	11.4	27	16	43	57
936A	49.52	214	25.8	15.2	26	33	59	41
937A	22.34	663	34.0	12.6	16	21	37	63
938A	39.62	558	46.5	8.8	7	12	19	81
940A	46.58	554	36.4	16.0	22	22	44	56
945A	44.31	413	30.6	6.7	4	18	22	78
954A	51.06	313	27.5	11.0	10	30	40	60
957A	48.97	292	27.5	13.8	15	35	50	50
959A	54.84	254	24.1	7.2	13	17	30	70
967A	44.30	348	30.8	14.8	16	32	48	52
973A	46.89	346	28.3	10.2	7	29	36	64
976A	43.47	368	33.2	11.6	9	26	35	65
982A	51.96	579	70.6	28.9	22	19	41	59
985A	49.79	593	105.8	60.3	45	12	57	43



Table 5 Ratios determined by gas chromatographic analysis.

Sample no.	pri/n-C <sub>17</sub>	phy/n-C <sub>18</sub>	pri/phy	CPI (C <sub>23</sub> -C <sub>29</sub> )
913A	0.86	0.44	1.13	-
915A	1.28	0.80	1.62	-
916A	3.43	2.65	0.98	-
923A	1.40	0.38	1.17	-
929A	1.41	0.55	2.00	-
932A	1.40	0.75	1.31	-
936A	1.33	0.61	1.71	-
937A	1.61	0.93	1.30	1.54
938A	1.54	1.01	1.32	1.79
940A	1.62	1.10	1.58	1.53
945A	1.78	1.06	1.56	1.64
954A	1.42	0.74	1.62	-
957A	1.32	0.56	2.34	1.48
959A	0.99	0.58	1.54	1.60
967A	0.89	0.75	1.15	-
973A	1.22	0.72	1.24	1.36
976A	0.87	0.62	1.36	-
982A	1.14	0.83	1.55	1.16
985A	0.91	0.54	1.54	1.08

Table 6 Average diastereoisomer ratio of the C<sub>31</sub>-C<sub>34</sub> 17 $\alpha$ (H)21 $\beta$ (H)-hopanes and relative content of the 17 $\beta$ (H)21 $\beta$ (H)-hopanes.

Sample	Depth below KB	$\Sigma$ 22S( $\alpha\beta$ ) / $\Sigma$ 22S+22R( $\alpha\beta$ )	$\beta\beta$
913A	4940'	(0.37) <sup>†</sup>	+++
915A	5060'	0.30 <sup>†</sup>	+++
916A	5120'	(0.45)	++
932A	6080'	( - )	+
940A	6550'	0.33 <sup>†</sup>	+
959A	7690'	0.50	-
967A	8170'	0.59	-
973A	8530'	0.56	-
982A	9080'	0.56	-
985A	9260'	0.55	-

<sup>†</sup> : only estimated in the range C<sub>31</sub>-C<sub>32</sub>

( ) : low concentrations

Table 7 Microscopic description of the cuttings and composition of the organic matter

- 909A 4700' Vedsted Formation (V-4 Member)
- Light-medium grey siltstone, glauconite very frequent (some chalk-cavings).
- The sample has a moderate-low organic content of fine grained particles, dominated by pseudovitrinite and only a few small true huminite/vitrinite particles for reflectance measurements. Very few wisps of liptinite and inertinite. Pseudovitrinite particles often look reworked: high reflecting rounded particles with oxidation rims and shrinkage cracks. Blue light induces a weak brownish-orange fluorescence from few sporinite and few liptodetrinites.
- 910A 4760' Vedsted Formation (V-4 Member)
- Like 909A, but difficult to estimate because of extensive contamination (cavings and additives) - many chalk cavings.
- 911A 4820' Vedsted Formation (V-3 Member)
- Like 909A
- 912A 4880' Vedsted Formation (V-3 Member).
- Dark grey-black silty claystone, large content of small pyrite framboids, glauconite very rare.
- The sample shows a moderate content of organic matter, most pseudovitrinite and large angular huminite/vitrinite particles (often ulminites with cellular structure), few liptinites.
- Blue light induces dark orange fluorescence from few sporinite and liptodetrinite, low liptinite content.
- 913A 4940' Vedsted Formation (V-3 Member)
- Like 912A, but some contamination.
- 914A 5000' Vedsted Formation (V-3 Member)
- Dark grey shaly claystone, often pyrite framboids, some glauconite. The sample has a moderate content of organic matter, most pseudovitrinite (some show oxidation rims and shrinkage cracks) together with liptinite, only few small huminite/vitrinite particles.
- In blue light some alginite (dinoflagellates) are seen with yellow fluorescence, few sporinite (yellow-orange fluorescence), but most liptodetrinites (dark orange fluorescence); moderate liptinite content.
- 915A 5060' Vedsted Formation (V-2 Member)
- Like 914A, but some more alginite (dinoflagellates).

- 916A 5120' Vedsted Formation (V-2 Member)  
 Dark grey shaly claystone (some chalk-cuttings), glauconite is rare, but small pyrite framboids are very frequent.  
 The sample has a very high content of fine grained organic matter, most consisting of liptinite and smaller amounts of pseudovitrinite and huminite/vitrinite, but relatively high content of inertinite particles (tiny fusinite fragments and some micrinite). True huminite/vitrinite particles are small and angular, some of them are relatively weak reflecting types.  
 In blue light a high liptinite content is seen, most as many fine stringers of bituminite with dull orange fluorescing inclusions, besides some small lipodetrinites. Few greenish-yellow fluorescing alginites (dinoflagellates).
- 917A 5180' Vedsted Formation (V-2 Member)  
 Dark grey shaly claystone, pyrite and glauconite occur.  
 The sample has a moderate content of organic matter with approximately equal amounts of pseudovitrinite (many reworked) and liptinite, few wisps of huminite/vitrinite and rare inertinite.  
 In blue light some alginite (dinoflagellates/ ar-critarchs) are seen with yellow fluorescence, some few sporinites and moderate liptinite content.
- 918A 5240' Vedsted Formation (V-1 Member)  
 Like 917A.
- 919A 5300' Vedsted Formation (V-1 Member)  
 Like 917A, besides a few stringers of bituminite.
- 920A 5360' Vedsted Formation (V-1 Member)  
 Like 919A.
- 921A 5420' Vedsted Formation (V-1 Member)  
 Like 919A.
- 922A 5480' Vedsted Formation (V-1 Member)  
 Like 919A.
- 923A 5540' Vedsted Formation (V-1 Member)  
 Medium grey claystone, with a large content of pyrite, glauconite occurs.  
 The sample has a varying (low-high) content of organic matter, only a few cuttings show a high content of very fine grained particles, mostly liptinite and pseudovitrinite, less huminite/vitrinite.  
 In blue light in a few cuttings many very small and some larger lipodetrinites are seen together with some alginite (dinoflagellates with greenish-yellow fluorescence) and few sporinites (yellow fluorescence), few bituminite stringers.

- 924A 5600' Bream Formation (Frederikshavn Member)  
Like 923A, but some less liptinite and less organic matter.
- 925A 5660' Bream Formation (Frederikshavn Member)  
Like 924A.
- 926A 5720' Bream Formation (Frederikshavn Member)  
Like 924A.
- 927A 5780' Bream Formation (Frederikshavn Member)  
Brownish grey silt/sandstone, very rich in glauconite, some pyrite.  
The sample has a moderate-low content of relatively coarse grained organic matter, most liptinite and less pseudovitrinite. Very few huminite/vitrinite (some lower reflecting types) and inertinite.  
In blue light some large structured liptodetrinites are seen together with some alginite (dinoflagellates, greenish-yellow fluorescence) and sporinite, few streaks of bituminite are also seen.
- 928A 5840' Bream Formation (Frederikshavn Member)  
Like 927, but with a higher organic content.
- 929A 5900' Bream Formation (Frederikshavn Member)  
Brownish grey silt/sandstone, rich in glauconite in the coarser cuttings, frequently pyrite.  
The sample has a moderate content of organic matter, higher in the more coarse grained cuttings. Especially in the coarse grained parts liptinite is dominant, some pseudovitrinite and inertinite fragments (fusinite and sclerotinite), a few huminite/vitrinite.  
In blue light much liptinite is seen as large detrinites (and some dinoflagellates) with yellow fluorescence and stringers of bituminite, a few recognizable alginite and sporinite, resinite observed.
- 930A 5960' Bream Formation (Frederikshavn Member)  
Like 929A, but lower organic content and many small liptodetrinites.
- 931A 6020' Bream Formation (Frederikshavn Member)  
Medium grey silty claystone, pyrite and glauconite occur.  
The sample has a moderate-low content of organic matter, like in 929A and 930A mainly consisting of liptinite and only small amounts of pseudovitrinite and huminite/vitrinite.  
In blue light the liptinite (yellow-orange fluorescence) is mainly seen as very small tiny detrinites (< 5  $\mu$ ) of alginites together with a few recognizable alginites (dinoflagellates) and sporinites.

- 932A 6080' Bream Formation (Børglum Member)  
Medium grey claystone with pyrite and glauconite.  
The sample has a moderate content of organic matter with a composition like 931A, besides some stringers of bituminite.
- 933A 6140' Bream Formation (Børglum Member)  
Like 932A.
- 934A 6200' Bream Formation (Børglum Member)  
Like 932A.
- 935A 6250' Haldager Formation (Flyvbjerg Member)  
Grey claystone and light sandstone, coaly particles and mica, pyrite occurs, glauconite very rare.  
The sample has a moderate content of organic matter, but somewhat varying amount and composition. Approximately equal proportions of liptinite and pseudovitrinite together with some particles of huminite/vitrinite and a few inertinites.  
In blue light the liptinite (yellow-orange fluorescence) especially is seen as tiny liptodetrinites and sporinites, some bituminite (cutinite observed).
- 936A 6310' Fjerritslev Formation (F-4 Member)  
Light sandstone with coaly particles and mica, pyrite framboids, glauconite absent.  
The sample has a moderate content of organic matter, about equal amounts of liptinite and pseudovitrinite and huminite/vitrinite. Many small angular particles of pseudovitrinite and huminite/vitrinite occur, but a few large (up to 1 mm) huminite/vitrinite particles (sometimes with cellular structure) 936A cont.  
also occur. Here also some few inertinites (fusinites and sclerotinites) are seen.  
Blue light shows a moderate liptinite content. Orange fluorescence from some streaky liptodetrinites and sporinite and orange-brown bituminite. Alginite and ?cutinite are observed.
- 937A 6370' Fjerritslev Formation (F-4 Member)  
Dark grey-black fissile shale, very rich in framboids of pyrite, glauconite absent.  
The sample has a very high content of organic matter, most liptinite, but also large amounts of pseudovitrinite and huminite/vitrinite, few inertinites (micrinite).  
In blue light much fine-grained liptinite is seen. Many streaks of bituminite (dull brown-orange fluorescence and greenish inclusions), some sporinite (yellow-orange fluorescence), several alginites (Tasmanites with greenish-yellow fluorescence) and alginite fragments.

- 938A 6430' Fjerritslev Formation (F-4 Member)  
Dark grey-black fissile shale, very rich in framboids of pyrite, glauconite absent.  
The sample has a moderate-high content of organic matter, most liptinite, only small amounts of pseudovitrinite and huminite/vitrinite and inertinite. In blue light much very fine grained liptinite is seen (like in 937A), most bituminite (brownish-orange fluorescence with greenish fluorescing inclusions), some sporinite (orange fluorescence) and few alginites (Tasmanites with greenish-yellow fluorescence).
- 939A 6490' Fjerritslev Formation (F-3 Member)  
Like 938A.
- 940A 6550' Fjerritslev Formation (F-3 Member)  
Like 938A.
- 941A 6610' Fjerritslev Formation (F-3 Member)  
Like 938A, but moderate organic content, liptinite (bituminite) predominant, few very fine grained huminite/vitrinite particles, some are lower reflecting ulminite types.
- 942A 6670' Fjerritslev Formation (F-3 Member)  
Like 941A.
- 943A 6730' Fjerritslev Formation (F-3 Member)  
Like 941A.
- 944A 6790' Fjerritslev Formation (F-3 Member)  
Like 941A.
- 945A 6850' Fjerritslev Formation (F-3 Member)  
Like 941A.
- 946A 6910' Fjerritslev Formation (F-3 Member)  
Grey and brown shaly claystone, pyrite frequent, no glauconite.  
The sample has a moderate content of relatively coarse grained organic particles (some long ulminites up to 200  $\mu$ m). The composition of the organic matter like 941A, but somewhat more pseudovitrinite and huminite/vitrinite.
- 947A 6970' Fjerritslev Formation (F-3 Member)  
Like 946A.
- 948A 7030' Fjerritslev Formation (F-3 Member)  
Grey-brown shaly claystone, glauconite absent, pyrite (small stems) rare.  
The sample has a moderate content of organic matter, most pseudovitrinite and huminite/vitrinite, less liptinite.

Blue light shows a low liptinite content (dull brown-orange) or no fluorescence, some bituminite and sporinite.

- 949A 7090' Fjerritslev Formation (F-3 Member)  
Like 948A, besides some large huminite/vitrinite with cellular structure (some telinites contain resinite), a few alginites (yellow fluorescence).
- 950A 7150' Fjerritslev Formation (F-3 Member)  
Like 949A.
- 951A 7210' Fjerritslev Formation (F-3 Member)  
Grey shaly claystone, glauconite rare, pyrite rare. The sample shows a moderate content of organic matter, most consisting of pseudovitrinite and some liptinite, less true huminite/vitrinite (often lower reflecting types, can be coarse grained). Blue light shows a moderate-low liptinite content, most unidentifiable liptodetrinites some sporinites (orange fluorescence) and bituminite.
- 952A 7270' Fjerritslev Formation (F-2 Member)  
Like 951A, but pseudovitrinite dominant. Some very coarse grained cuttings (? cavings).
- 953A 7330' Fjerritslev Formation (F-2 Member)  
Like 952A.
- 954A 7390' Fjerritslev Formation (F-2 Member)  
Grey shaly claystone, probably like 915A, but also some different coarse grained cuttings and coaly particles (? cavings). The sample shows a moderate content of organic matter, dominated by pseudovitrinite and almost equal amounts of huminite/vitrinite, a few inertinites, almost no liptinite. Blue light shows a very low liptinite content, besides some liptodetrinites only a few sporinites and bituminites can be recognized.
- 955A 7450' Fjerritslev Formation (F-2 Member)  
Like 954A.
- 956A 7510' Fjerritslev Formation (F-2 Member)  
Like 954A.
- 957A 7570' Fjerritslev Formation (F-1 Member)  
Dark grey shale, glauconite absent, framboids of pyrite. The sample has a moderate-low content of organic matter, most pseudovitrinite (often showing oxidation rims and shrinkage cracks, probably due to reworking) and some liptinite. Only a few small whisps of true huminite/vitrinite and some small inertinite fragments.



Blue light shows some liptinite with varying fluorescence, most small liptodetrinites (yellow-orange fluorescence), a few sporinite (orange fluorescence) and streaks of bituminite. A few alginites (Tasmanites, yellow fluorescence) probably in cavings.

- 958A 7530' Fjerritslev Formation (F-1 Member)  
Like 957A.
- 959A 7690' Fjerritslev Formation (F-1 Member)  
Dark grey shale, glauconite rare, pyrite "tissues". The sample has a moderate-low content of fine grained organic particles, highly dominated by pseudovitrinite (some showing reworking, oxidation rims and shrinkage cracks), only few liptinite and huminite/vitrinite particles and a few inertinites (sclerotinite and fusinite).  
Blue light shows a low liptinite content of small liptodetrinites (yellow-orange fluorescence), a few sporinite (dull orange-brown fluorescence) and very few streaks of bituminite.
- 960A 7750' Fjerritslev Formation (F-1 Member)  
Like 959A.
- 961A 7810' Fjerritslev Formation (F-1 Member)  
Like 959A.
- 962A 7870' Fjerritslev Formation (F-1 Member)  
Like 959A.
- 963A 7930' Fjerritslev Formation (F-1 Member)  
Like 959A.
- 964A 7990' Fjerritslev Formation (F-1 Member)  
Like 959A.
- 965A 8050' Fjerritslev Formation (F-1 Member)  
Dark grey shale, glauconite and pyrite (rhombic) rare.  
The sample has a moderate content of organic matter, most pseudovitrinite (often showing reworking, shrinkage cracks and oxidation rims) and some angular huminite/vitrinite, few inertinites (sclerotinite) and liptinite.  
Blue light shows a low content of liptinite, most structured liptodetrinites and a little bituminite, few alginites and sporinites.
- 966A 8110' Fjerritslev Formation (F-1 Member)  
Like 965A.
- 967A 8170' Fjerritslev Formation (F-1 Member)  
Like 965A.

- 968A 8230' Fjerritslev Formation (F-1 Member)  
Dark grey shale with some coarse quartz-grains and dark finely laminated sand/siltstone with coaly stringers.  
The sample has a moderate-low content of organic matter, with a composition like 965A, but the coarse grained cuttings more like 970A.
- 969A 8290' Fjerritslev Formation (F-1 Member)  
Like 968A.
- 970A 8350' Fjerritslev Formation (F-1 Member)  
Dark laminated sand/siltstone with coaly stringers and dark grey shale, glauconite rare, pyrite common. The sample has a moderate content of relatively coarse grained angular and gnarled organic particles, most consisting of pseudovitrinite and huminite/vitrinite (often bituminous), very little liptinite and inertinite.  
Blue light shows a very low content of liptinite, very few of sporinite (brownish-orange fluorescence), resinite, bituminite and some liptodetrinites
- 971A 8410' Fjerritslev Formation (F-1 Member)  
Like 970A.
- 972A 8470' Gassum Formation  
Like 970A.
- 973A 8530' Gassum Formation  
Different lithologies, grey shale and coarse grained sandstone with coal particles, feldspar, kaolinite, white and brown mica. Gauconite rare pyrite common. The sample has a moderate-high very coarse grained organic content with a composition like 970A and besides a few large coal grains (up to 1/2 mm in diameter) (most desmocolinite with sporinite, cor-pocollinite).  
Blue light shows a rather low but varied content of liptinite, especially of sporinite (orange fluorescence) besides liptodetrinites and bituminite, a few resinites, cutinite and ?alginite.
- 974A 8590' Gassum Formation  
Like 973A, but more finegrained.
- 975A 8650' Gassum Formation  
Same cutting lithology as 973A (very coarse grained sandstone), besides some carbonate cuttings.  
The sample shows a varied content of organic matter, like 973A, besides in the carbonate cuttings: many angular huminite/vitrinite and pseudovitrinite, some inertinite and liptinite.  
Blue light shows a moderate content of liptinite, most bituminite.

- 976A 8710' Gassum Formation  
 Dark grey shale and yellowish fine grained sandstone with coaly particles, glauconite rare, pyrite common.  
 The sample has a moderate content of coarse grained organic matter. Many angular huminite/vitrinite and pseudovitrinite but almost equal amount of liptinite.  
 Blue light shows a moderate content of liptinite, much sporinite (orange fluorescence) and bituminite, a few resinites and cutinite (?) besides some liptodetrinites.
- 977A 8770' Gassum Formation  
 Like 976A, but lower organic content (some "clea" coarse grained sandstone cuttings without organic material).
- 978A 8830' Gassum Formation  
 Many very different cuttings, light sandstone, red-green mottled claystone, dark claystone, white carbonates (? cavings).  
 The sample has in average a low organic content, but composition is difficult to estimate.
- 979A 8890' Gassum Formation  
 Like 978A.
- 980A 8950' Gassum Formation  
 Different lithologies, light coarse grained sand and siltstone (with feldspar and kaolinite) dark shale and some mottled red-green claystone. Glauconite absent, pyrite rare.  
 The sample has a varying but generally rather low content of organic matter, largely dominated by pseudovitrinite and few huminite/vitrinite and very few liptinites.  
 Blue light shows almost no liptinite, a few recognizable sporinites (dark orange-red fluorescence) besides a few liptodetrinites.
- 981A 9020' Gassum Formation  
 Like 980A.
- 982A 9080' Vinding Formation  
 Dark shale, some sand/siltstone with coaly stringers and carbonates. Pyrite and glauconite rare.  
 The sample has a moderate-low organic content, most fine-grained angular huminite/vitrinite and pseudovitrinite, but also some liptinite.  
 Blue light shows a low liptinite content, sporinite (orange-red fluorescence), small liptodetrinites and few alginites (yellow fluorescence).
- 983A 9140' Vinding Formation  
 Missing

- 984A 9200' Vinding Formation  
 Dark grey and brownish shale, dark mottled carbonates. Glauconite absent, pyrite common.  
 The sample has a low organic content, some liptinite often associated with small pyrites and a few huminite/vitrinite and pseudovitrinite.  
 Blue light shows a low liptinite content, most small liptodetrinites and alginites (yellow-orange fluorescence), few sporinites.
- 985A 9260' Vinding Formation  
 Dark grey-black and brownish shale, dark mottled carbonates. Glauconite absent, pyrite common.  
 The sample generally has a moderate-low organic content, dominated by liptinite and only a few pseudovitrinite and huminite/vitrinite.  
 Blue light shows a low liptinite content, most alginite (tousled Botryococcuslike with yellow-orange fluorescence) and some liptodetrinites. In a few cuttings the liptinite content is very high consisting of large Botryococcus like and alginite fragments with bright yellow fluorescence.
- 986A 9320' Vinding Formation  
 Missing
- 987A 9380' Oddesund Formation  
 Light gre claystone and siltstone, few carbonates, some cavings. Glauconite absent, pyrite common.  
 The sample has a very low content of very small (usually < 5 m) organic particles, some liptinite and some pseudovitrinite and huminite/vitrinite (many lower reflecting, ? cavings).  
 Blue light shows a few liptinites, small liptodetrinites and sporinite (orange-red fluorescence).
- 988A 9440' Oddesund Formation  
 Like 987A.
- 989A 9460' Oddesund Formation  
 Like 987A, and a few alginites (Botryococcus-like).

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The source rock potential of the Mesozoic sediments in the Hyllebjerg-1 well is assessed in this paper.

The methods used encompass vitrinite reflectance measurements, spectral fluorescence measurements, Gas Chromatography and mass spectrometry of Biomarkers.

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