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During field work in the summer of 1990 oily material was discovered in mineralised breccias in Germania Land, North-East Greenland. Organic geochemical data (TOC, Rock Eval pyrolysis, extraction, GC, py-GC, GC-MS) show that the material is composed of degraded crude oil. Thermal alteration has been limited whereas biodegradation, probably during migration or earlier trapping, has been severe.

The oil was apparently generated from a source rock dominated by terrestrially derived organic matter, probably deposited in a lacustrine environment. Several geochemical parameters suggest saline rather than freshwater conditions in the lakes. Based on tectonic setting and knowledge on existing sediments in the region several possibilities for the source exist. The most likely source rock seems to be of Late Palaeozoic age.

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Introduction

In the summer of 1990, during GGU's regional geological mapping of the area between 75° and 78° (Henriksen, 1989, 1991) oily material was discovered by H. Stendal (Copenhagen University) and S. M. Jensen (GGU) in pyrite-mineralised breccias in metamorphic basement on Germania Land (Fig. 1). The breccia zones are situated along NNW-SSE orientated faults which cut the basement. The fault zone appears to continue towards the north where it probably forms the border of an offshore sedimentary basin between Île de France and Gamma Ø (Fig. 1). The existence of this basin has been suggested on the basis of aeromagnetic data by Larsen (1984, 1990) and has been confirmed further north by seismic data acquired by the German research vessel "Polarstern" in 1988 (unpublished). Erosional remnants of this and other sedimentary basins are exposed on Depotnæsset, along the eastern part of Fladebugt, north of Kulhøj and on Store Koldewey (Fig. 1) (Stemmerik & Piasecki, 1990; Piasecki & Stemmerik, in prep.).

The discovery of the oil relics in the metamorphic basement in Germania Land is encouraging because it most likely involves long-distance migration along the fault zone from deeply buried oil-generating units or accumulations of oil. The implications are especially important in relation to the offshore basins which are being studied seismically as part of the ongoing KANUMAS programme (Larsen & Pulvertaft, 1990).

Geological setting

Germania Land is situated north of the areas of exposed sedimentary basins in East and North-East Greenland. Outliers of Carboniferous and Mesozoic sediments, however, indicate that a thick sedimentary succession once covered most of the region. This has been removed during

later, Tertiary (?) uplift (Piasecki & Stemmerik, in prep.). The exposed basement rocks thus once formed the floor of sedimentary basins like those exposed further to the south.

The oil relics were found in steep, NNW-SSE (~160°) striking breccia zones located along a 75 m high coast-parallel cliff at the southeastern corner of Fladebugt (Fig. 2). Pyrite-mineralised breccias are exposed in several segments along the cliff. They appear to constitute three parallel zones with a combined exposed length of about 800 m. The main breccia zone, furthest to the west, is 5-10 m wide, whereas the two subordinate zones are 1-5 m wide. The exposed segments of the main breccia zone are 100-150 m long.

The country rock is a grey amphibolitic gneiss with swarms of subparallel pegmatite sheets. The gneissic banding and pegmatite sheets, which generally strike 140-150° and dip 70-80°W, are cut at a very low angle by the subvertical fault breccia zones. The pegmatite sheets in the gneiss are 0.2-2 m wide and in places along the cliff they occur in swarms that constitute more than half of the exposed rock. They consist of red alkali feldspar and quartz, with varying amounts of accessory magnetite and biotite.

Within the breccia zones four types of superimposed structures are recognised. In chronological order, from the margins of breccia zones towards the centres, they have the following characteristics:

- 1 Brecciation of gneiss and pegmatites. Fragments are pervasively silicified and the breccia cement consists of light grey, microcrystalline quartz. Pyrite occurs in tiny fractures in rock fragments and finely disseminated in the cement.
- 2 Massive irregular pyrite veins cutting central parts of the silicified breccia zones. Large pyrite aggregates often show zonation typical of open space precipitation.
- 3 A second generation of microcrystalline quartz breccia cement precipitated contemporaneously with – or later than – the massive pyrite veins. The cement is darker grey and more fine

grained than that of the earlier breccia, and contains abundant coarse grained pyrite and accessory calcite and baryte.

- 4 A few late calcite veins, up to 0.5 m wide, with accessory pyrite, baryte and fluorite cut all the above structures. The calcite veins are generally orientated 020/60 E.

The magnetite observed in fresh pegmatite sheets is not preserved where these are brecciated and silicified. Released iron gives the cliff striking orange-red weathering colours. The altered magnetite may have been an important source of iron in the formation of pyrite and only silica and sulphur need to have been introduced in appreciable quantities from sources outside the breccia zones. Introduction of relatively little metal into the breccia zones is also suggested by generally low concentrations of metals other than iron in stream sediment samples and pyrite-mineralised rock samples.

Oily material is not confined to any one structure and has been observed in all exposed segments of the breccia zones. It seems to be present in largest quantity in small cavities and late fractures in the youngest quartz breccia cement. In some places, just below the breccia zone, oily relics form a thin (1-2 mm) coating on the loose, rusty-weathering fine-grained soily material. Migration of oil through the breccia zone probably took place late during, or after, the formation of the fault breccia.

Material and methods

Three breccia samples containing oil relics (GGU 365085 = B163/B164, GGU 365085-3 = B165 and GGU 365091-2 = B166) have been analysed geochemically. The analytical programme includes total organic carbon (TOC) determination in a LECO furnace, Rock Eval pyrolysis, extraction in a Soxtech apparatus with subsequent deasphalting and column separation into

saturated and aromatic hydrocarbons and NSO compounds. The saturated hydrocarbons were analysed by gas chromatography (GC), and pyrolysis-gas chromatography was carried out on the asphaltenes (py-GC). All these analyses were carried out by the source rock laboratory of the Geological Survey of Denmark. The analytical procedures follow the description given by Christiansen *et al.* (1989) and Bojesen-Koefoed (1989).

GC-MS analyses of the saturated hydrocarbons were carried out by the Organic Geochemistry Section of Institutt for Kontinentalsokkelundersøkelser og Petroleumsteknologi, Trondheim (Kaarstad, 1991) and by the Geological Survey of Denmark.

Five plugs of samples 365085 and 365091 have been studied at the Core Analysis Laboratory of the Geological Survey of Denmark (by Pernille Skejnæs). The porosity (calculated from caliper measurements), and the grain density were measured both before and after cleaning with toluene (Table 1), whereas the air permeability was only measured on cleaned samples.

Results

Porosity/permeability

The five plugs from samples GGU 365085 and GGU 365091 all display a distinct vuggy porosity. The vugs are often elongated with typical lengths between 5 and 10 mm and a width of a few millimetres. Both the porosity and permeability show a considerable variation with some patches showing an almost good reservoir potential (Table 1). The average porosity is 7.29% and 9.77% before and after cleaning with toluene respectively. This difference is comparable to the measured extractabilities (see later) and gives an average saturation of oil + oil relics of 25.3%. This value is rather high considering that a considerable amount of hydrocarbons has been lost by *in situ* leakage, and during sampling and preparation. The permeability varies from almost negligible to fair which is typical for this type of reservoir where the vugs often are poorly

connected. The average grain density is high (2.847 for cleaned samples) compared to normal sandstones which typically show values between 2.62 and 2.66. This increase in grain density is due to the pyrite content.

LECO and Rock Eval pyrolysis

One sample, GGU 365025 was analysed by LECO and Rock Eval pyrolysis before being split into the two subsamples B163 and B164; the TOC was remeasured for each of the two subsamples in order to test reproducibility. The TOC values are high (0.5-0.6%) compared to typical values for basement rocks (Table 2).

The Rock Eval data show an anomalously low T_{\max} value and a high S_1/S_2 ratio which is typical for oil stained rocks (Table 2) (Clementz, 1979; Christiansen *et al.*, 1989). Compared to migrated material from elsewhere in North-East Greenland and North Greenland, the very low T_{\max} , and the high S_1+S_2/TOC point towards a limited thermal alteration (see Christiansen, in review).

Extraction

All organic material in the studied samples is extractable which is a peculiar feature known only from certain types of clean reservoir rocks (Table 3). The extracts contain approximately equal amounts of hydrocarbons (saturated + aromatic) and non-hydrocarbons (NSO + asphaltenes) (Table 3, Fig. 3). For comparison it should be noted that typical crude oil contains 10-15% non-hydrocarbons, typical heavy crude oil 40-50% non-hydrocarbons, whereas seep or impregnation material from North Greenland contains 60-80% non-hydrocarbons (Tissot & Welte, 1984; Cornelius, 1987; Christiansen, in review). Thus it seems that the non-hydrocarbons in the present material have been only moderately concentrated during degradation.

The ratio of saturated to aromatic hydrocarbons is relatively high compared to most normal crude oils and to degraded oils. Such high ratios are typical for oils generated from non-marine source rocks.

Gas chromatography (GC)

All three samples show rather similar gas chromatograms which typically display two unresolved humps (Fig. 4). The humps tend to peak at a retention time corresponding to C₁₆-C₁₈ and C₂₇-C₃₀, respectively. The *n*-alkanes, which normally are the most abundant compounds in the saturated fraction, seem to have been lost during degradation. This is also the case for the isoprenoids. The few prominent peaks above the humps have not been identified.

Pyrolysis-gas chromatography (Py-GC)

All three samples show homogeneous patterns and there is only little variation in calculated compound ratios from Py-GC of asphaltenes (Table 4). Based on the assumption that the composition of pyrolysates of asphaltenes is rather similar to pyrolysates of the kerogen in the source rock, it is possible to classify the primary composition of the oil once generated. The pyrolysates have relative low contents of aromatic compounds (Figs 5 and 6) compared to published examples from various source rocks (e.g. Horsfield, 1989), and in terms of petroleum composition the pyrolysates are classified as a mixed oil with naphthenic tendencies (Fig. 6). However, asphaltene pyrolysates tend to be enriched in aromatics and depleted in *n*-alkanes relative to kerogen pyrolysates (see J. Bojesen-Koefoed *et al.*, in prep.). Thus the classification should probably be changed to a mixed oil with paraffinic tendencies.

The pyrolysate is dominated by short chain compounds (Fig. 7). Compared to Horsfield's classification, it should be noted that the *n*-C₅ compound is usually insufficiently resolved to allow

a positive identification, and all compounds of chain length less than C₆ are thus included as gases. Asphaltene pyrolysates often are enriched in gases compared to kerogen pyrolysates, and therefore the seep pyrolysate probably classifies as a condensate or as a mixed oil.

Biomarker distribution

The distribution of sterane, terpane and other biomarkers has been studied using especially the m/z 71 (alkane), 217 (sterane) and m/z 191 (terpane) mass fragmentograms with additional information from the m/z 163, 177, 205, 281, 231 and 259 mass fragmentograms and the total ion chromatograms (TIC).

In contrast to the GC data it has been possible to resolve and identify *n*-alkanes and isoprenoids using the GC-MS technique (TIC and m/z 71) (Fig. 8). The relative content of these compounds (height above 'naphthenic hump') seems to vary from extract to extract with very low values in B 164 and relatively higher values in B 165 and B 166.

The *n*-alkanes peak at *n*-C₁₇ and an even over odd predominance is noted between *n*-C₂₂ and *n*-C₂₈. This pattern and the relative low Pr/Ph values (Table 5, Fig. 8) could be indicative of a carbonate or saline lacustrine environment of the source rock (ten Haven *et al.*, 1988).

These data should, however, be carefully interpreted since many other parameters suggest such a severe biodegradation that *n*-alkanes are not likely to have been preserved. The existence of limited amounts of *n*-alkanes could be due to contamination or mixing of a severely degraded crude oil with less altered material.

The three analysed samples contain very low contents of steranes and individual compounds cannot be resolved. The terpanes are completely dominated by the tricyclics, whereas the pentacyclic terpanes (hopanes) only occur in low concentrations and identification of the compounds are difficult (Fig. 9). The low concentration or even lack of steranes and hopanes

points again towards a severe alteration of the seep material (e.g. Seifert & Moldowan, 1979; Connan, 1984).

The relative enrichment of tricyclic to pentacyclic terpanes is probably due to degradation since the tricyclics are very resistant both to biodegradation (e.g. Connan, 1984; Lin *et al.*, 1989; Waples & Machihara, 1990) and to thermal alteration (Kruege, 1986; Christiansen *et al.*, 1989; van Graas, 1990).

A high tricyclic to pentacyclic ratio may, however, also be controlled by the depositional environment of the source rock and has been well documented in saline lake deposits and carbonates (Zumberge, 1984; Palacas *et al.*, 1984; Mello *et al.*, 1988a,b; Buchardt *et al.*, 1989; Waples & Machihara, 1990; Kruege *et al.*, 1990; Christiansen *et al.*, in review). In these cases the distribution of the individual tricyclic compounds is often distinct with a notable dominance of T₂₃. In the present material a peculiar distribution is noted. The following compounds have almost uniform abundance T₂₀ and T₂₁, T₂₃ and T₂₄, T₂₅ and T₂₆, T₂₈ and T₂₉, with the following order of abundance of the pairs : $T_{23+24} \geq T_{20+21} > T_{25+26} \geq T_{28+29}$ (Table 6, Fig. 9). This contrasts with for example the Upper Permian Ravnefjeld Formation from East Greenland where $T_{23} > T_{24}$, $T_{21} > T_{20}$ and $T_{25} > T_{26}$, with the following relative abundance of pairs: $T_{23+24} > T_{25+26} > T_{20+21} \geq T_{28+29}$ (Christiansen *et al.*, in review; see also Christiansen, in review). The only known example of a similar distribution of tricyclics in Greenland samples is in Middle Devonian bitumens which probably were derived from a saline lacustrine source rock (Christiansen *et al.*, 1990; Christiansen, in review).

The distribution of tricyclic compounds in freshwater source rocks from East Greenland is poorly known due to the relatively low concentration, this is the case for example in the Upper Devonian lacustrine source rock, the Upper Carboniferous lacustrine source rocks, the Lower

Jurassic Kap Stewart Formation, and the Lower Jurassic Sortehat Member, (Christiansen *et al.*, 1991; Krabbe, in prep.; Krabbe *et al.*, in prep. plus unpublished GGU data).

Discussion

The geochemical data obtained provide a good background for discussion of thermal alteration and biodegradation of the migrated hydrocarbons and also provide finger printing for possible correlation to a source rock, or at least indications of the depositional environment of the source rock.

Although most of the analytical data seems to support the same model of degradation, some differences are noted between results from the various methods. This is also the case for parameters applicable in oil-source rock correlation due to the relatively intense degradation.

Thermal alteration. Due to lack of steranes and hopanes normal molecular maturity indicators cannot be applied. The distribution observed in chromatograms with maxima at relatively short carbon numbers, the py-GC classification, and the lack of some biomarkers indicate a relatively high thermal maturity. Either the source rock or the accumulated crude oil has been thermally altered under conditions corresponding to the deeper part of the oil-window but without reaching the condensate zone. The lack of highly coalified solid bitumen in the vein suggests that the *in situ* thermal alteration has been limited.

Biodegradation. All the applicable analytical data suggest that biodegradation has taken place, but estimates of intensity vary from moderate to severe depending on arguments. The distribution of hydrocarbons:non-hydrocarbons, the shape of the chromatograms and the minor, but significant, presence of *n*-alkanes and isoprenoids suggest only a moderate biodegradation. In contrast the

complete lack of steranes and hopanes with only the tricyclic terpanes retained in significant amounts point towards a much stronger or severe degradation. The relatively homogeneous composition from sample to sample suggests that the biodegradation is not a recent near-surface process, but probably took place during accumulation before the final leakage through the vein. Some of the differences may be explained due to mixing of crude oils with a varying degradation history.

Source rock correlation. Due to lack of exposed organic-rich units in the surrounding area, the source rock correlation study concentrates on defining parameters which provide information on the depositional environment of the source rock. The lack of steranes and hopanes due to degradation limits such possibilities. The gross composition of the extracts and the py-GC parameters are in favour of a non-marine source rock, whereas the possible *n*-alkane and isoprenoid distribution (which could reflect some sort of contamination), the high tricyclic terpene content and the distinct distribution of tricyclics suggest a saline lacustrine source rock. These data however show negative correlation with the following lacustrine possibilities in East Greenland:

- the Upper Devonian freshwater lacustrine source rock (Christiansen *et al.* 1990; Christiansen *et al.*, 1991; unpublished data)
- the Upper Carboniferous freshwater lacustrine source rock (Christiansen *et al.* 1990; Christiansen *et al.*, 1991; unpublished data)
- the Lower Jurassic Kap Stewart Formation (freshwater lacustrine) (Dam & Christiansen, 1990; Christiansen *et al.*, in press; Krabbe, in prep.)
- the Lower Jurassic Sortehat Member (brackish) (Krabbe *et al.*, in review)
- the Lower Jurassic algal coal (Bojesen-Koefoed *et al.*, in prep.).

The following marine source rocks that have been studied have different parameters:

- the Upper Permian Ravnefjeld Formation (carbonate influenced) (Christiansen *et al.*, 1991; Christiansen *et al.*, in review)
- the Upper Jurassic Hareelv and Bernbjerg Formations (Requejo *et al.*, 1989; Christiansen *et al.*, in press).

The only material analysed from Greenland that has a rather similar *n*-alkane, isoprenoid, and tricyclic terpane distribution is some Middle Devonian solid bitumens from Gauss Halvø in North-East Greenland (Christiansen *et al.*, 1990; Christiansen, in review; unpublished data). Although the detailed information on the source rock for these bitumens is limited due to a high thermal maturity, a saline lacustrine source rock of Middle Devonian age has been suggested (Christiansen *et al.*, 1990).

It is not likely that the oil is sourced by Devonian shales because the oldest sediments seen in the Germania Land area are of mid to late Carboniferous age. Rather the geochemical parameters indicate that the source rock was deposited in a period with a hot and arid climate. By comparison to the succession in North-East Greenland (Stemmerik *et al.*, in press) it is most likely that the source rock is of Carboniferous or Early Permian age.

Conclusion

Based on the analytical data, the tectonic setting, and knowledge of existing sediments in both on- and offshore areas of the region, the following conclusions may be drawn:

- The oily material analysed is a relict crude oil.
- The oil has been severely biodegraded, during either migration or earlier trapping in a reservoir in the subsurface.

- The oil seems to have been generated from a source rock dominated by terrestrially derived organic matter, e.g. a lacustrine deposit.
- Lacustrine organic-rich shales are known elsewhere in East and North-East Greenland from the Devonian, the Upper Carboniferous and Lower Jurassic successions.
- The few significant biomarker distributions point towards a saline lacustrine deposit as source rock, and a Late Palaeozoic age is the most likely.
- The discovery of the oil seep is encouraging for further studies in the region and offshore exploration in the future.

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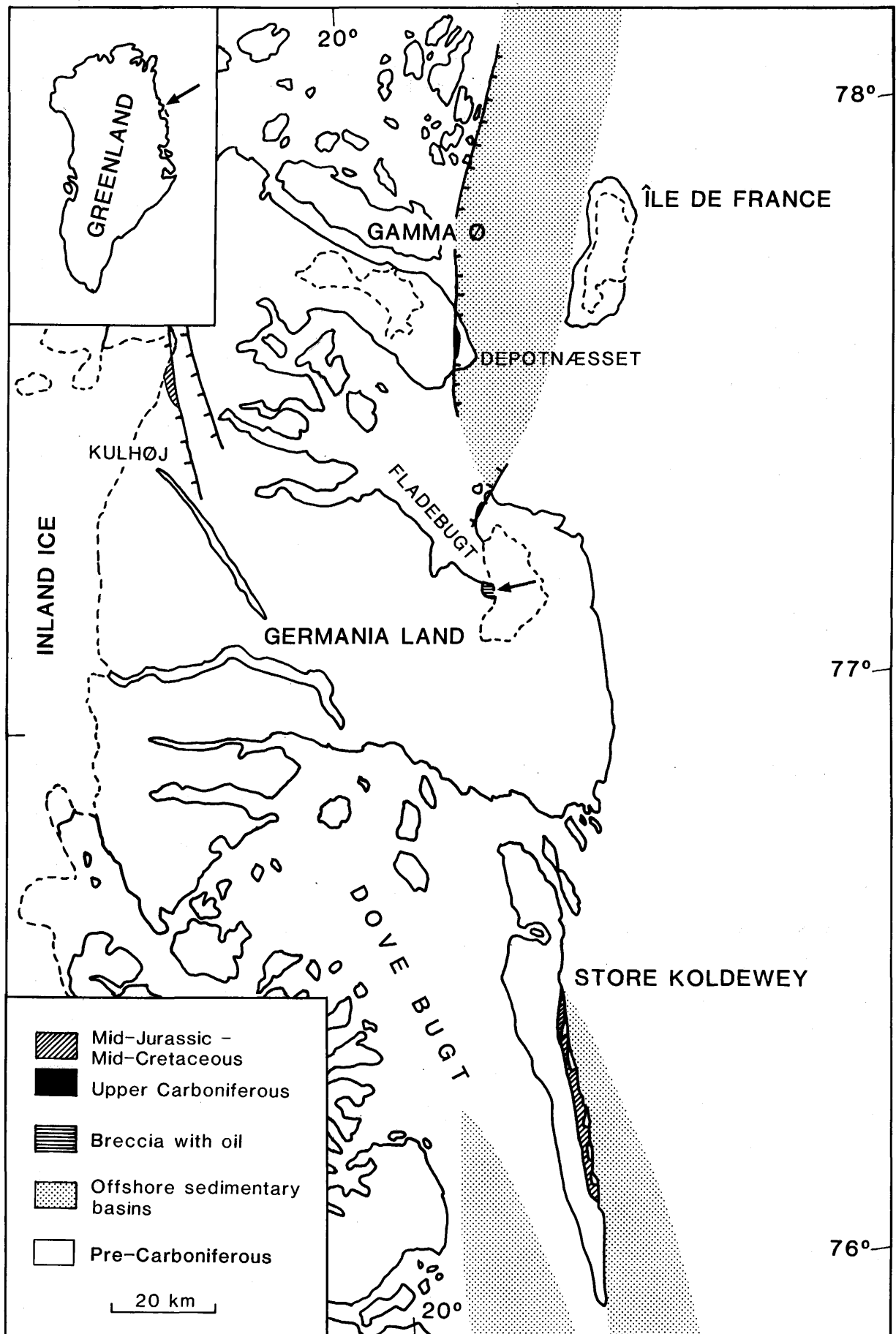


Fig. 1. Map showing the location of the Germaniella oil seep (arrow). The distribution of exposed sediments is based on Stemmerik & Piasecki (1990) and Henriksen (1991); the outline of the offshore sedimentary basins is based on Larsen (1990).

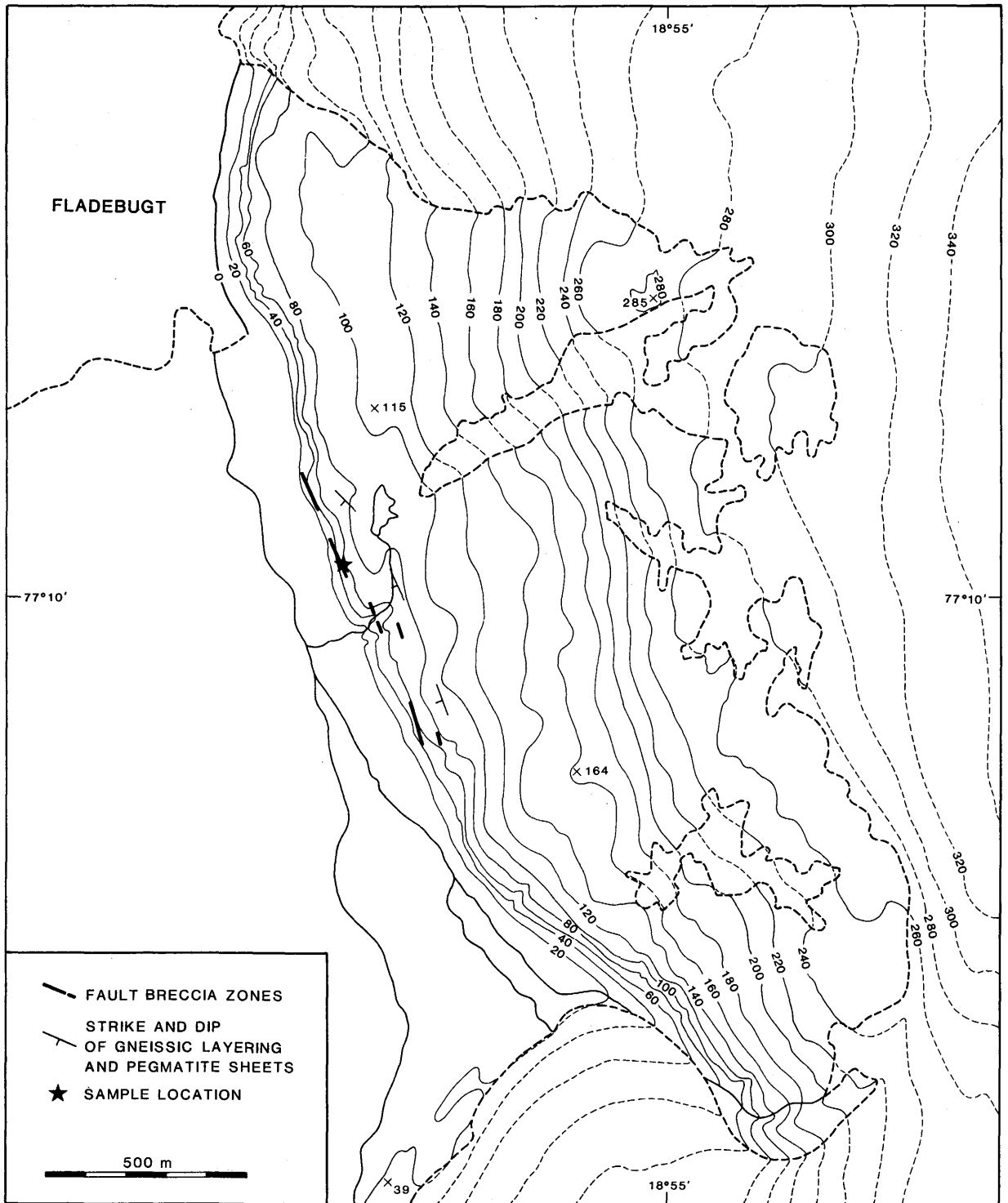


Fig. 2. Map of coastal cliff at the southwestern corner of Fladebugt, north Germania Land. Oil remnants have been observed in all exposed parts of the fault breccia zones.

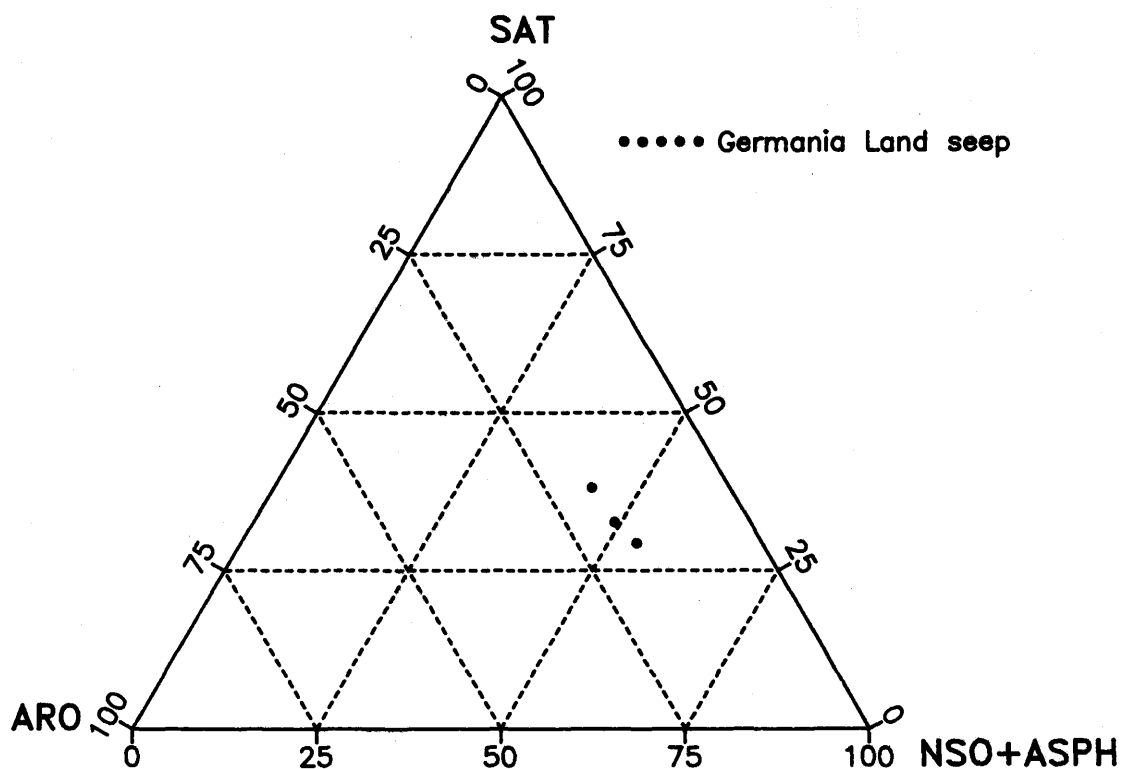


Fig. 3. Triangular diagram showing relative extract composition of saturated hydrocarbons, aromatic hydrocarbons, and NSO compounds + asphaltenes.

365085
(B 163)

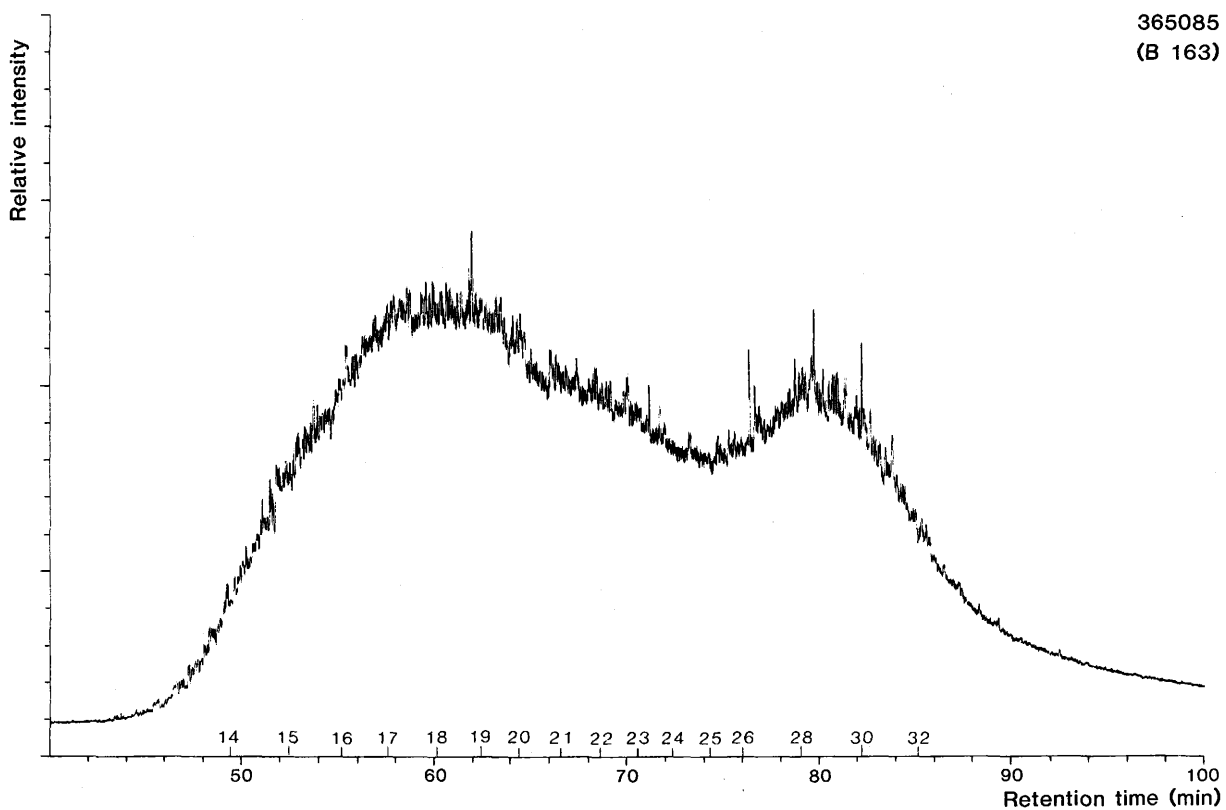


Fig. 4. Gas chromatogram of saturated hydrocarbons in GGU 365085 (B 163). Numbers correspond to approximate number of carbons.

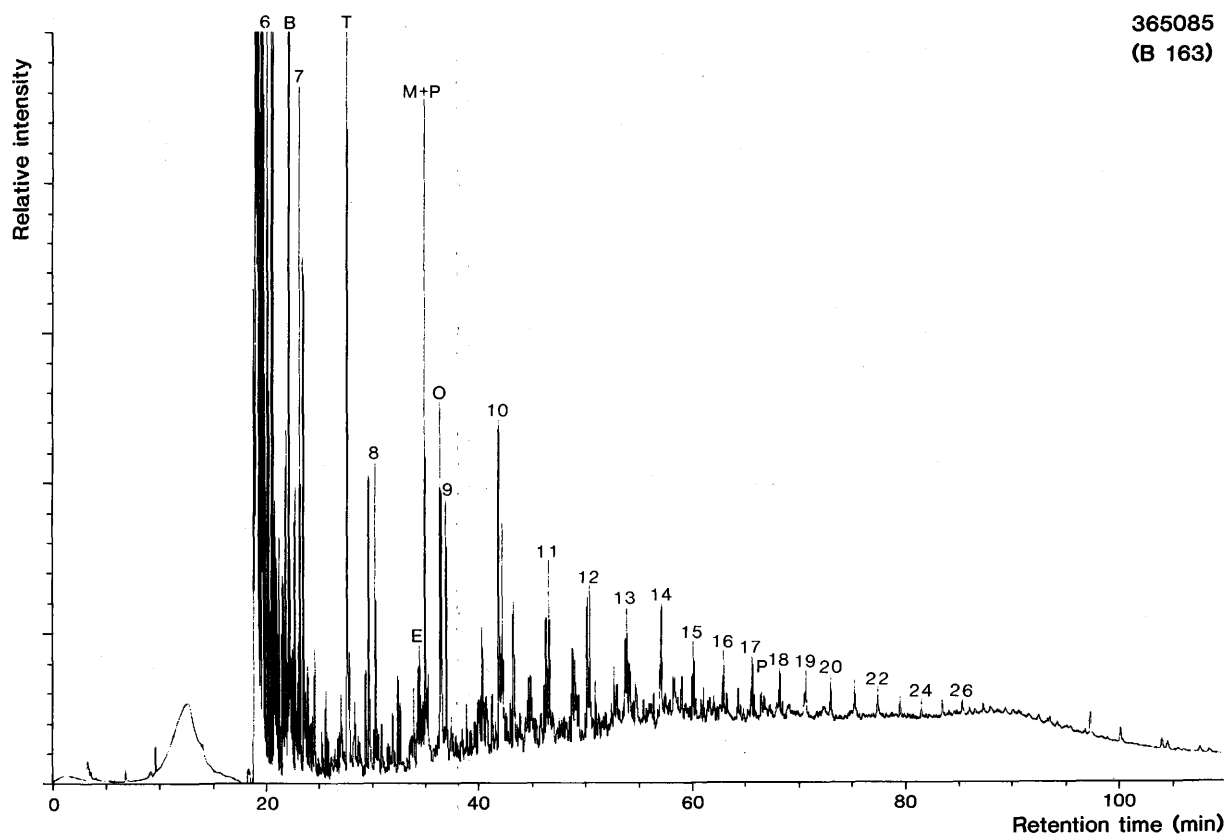


Fig. 5. Pyrolysis-gas chromatogram of asphaltenes in GGU 365085 (B 163). Numbers correspond to carbon numbers in pairs of alkanes/alkenes. B = benzene, T = toluene, E = ethylbenzene, M+P = m+p- xylene, O = O-xylene, P = prist-1-ene.

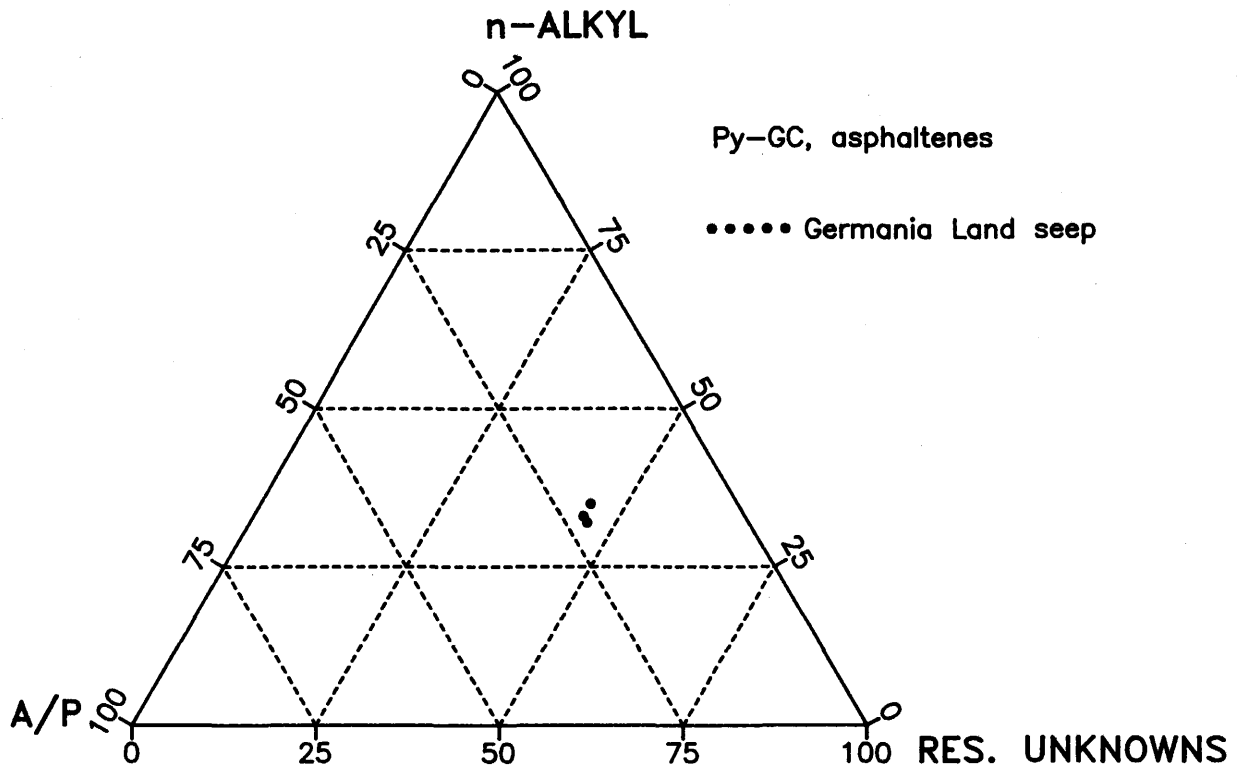


Fig. 6. Triangular diagram showing composition of pyrolysate according to proportion on C_{6+} alkyl moieties, aromatic moieties and resolved unknowns. The inferred petroleum composition and kerogen types are adopted from Horsfield (1989).

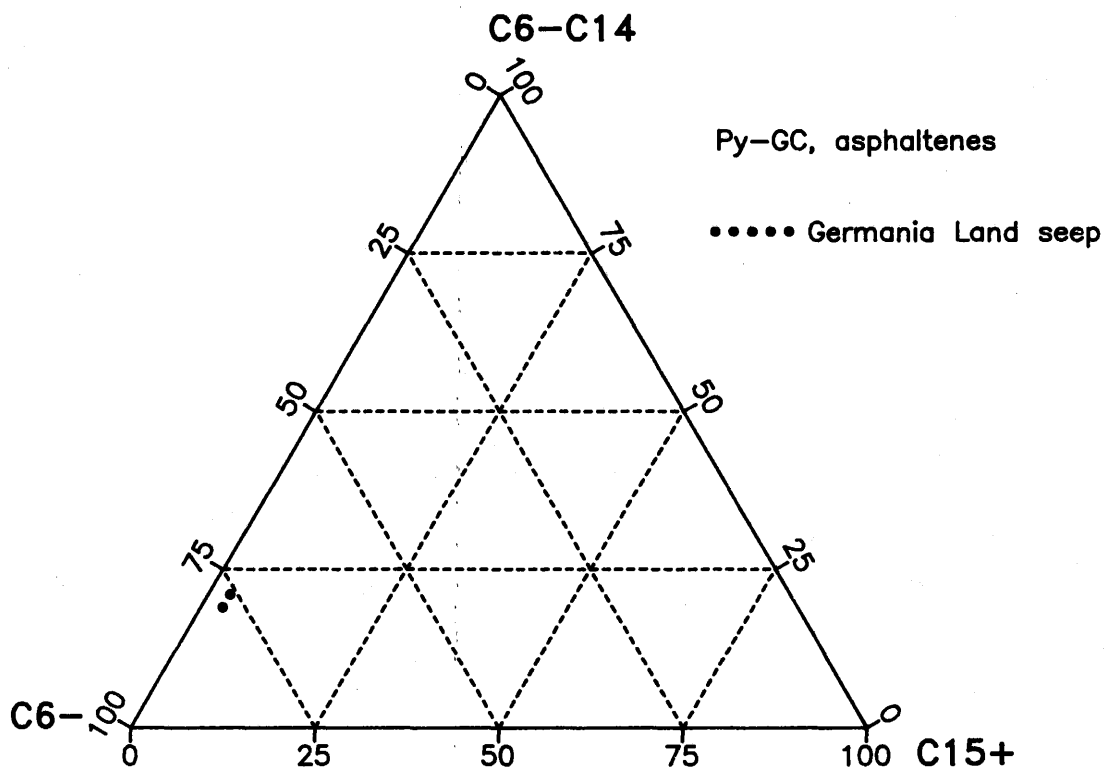


Fig. 7. Triangular diagram showing composition of pyrolysate according to chain length distribution of total C₁-C₆ resolved pyrolysate, C₆-C₁₄ alkyl-moieties and C₁₅₊ alkyl moieties. Adopted from Horsfield (1989).

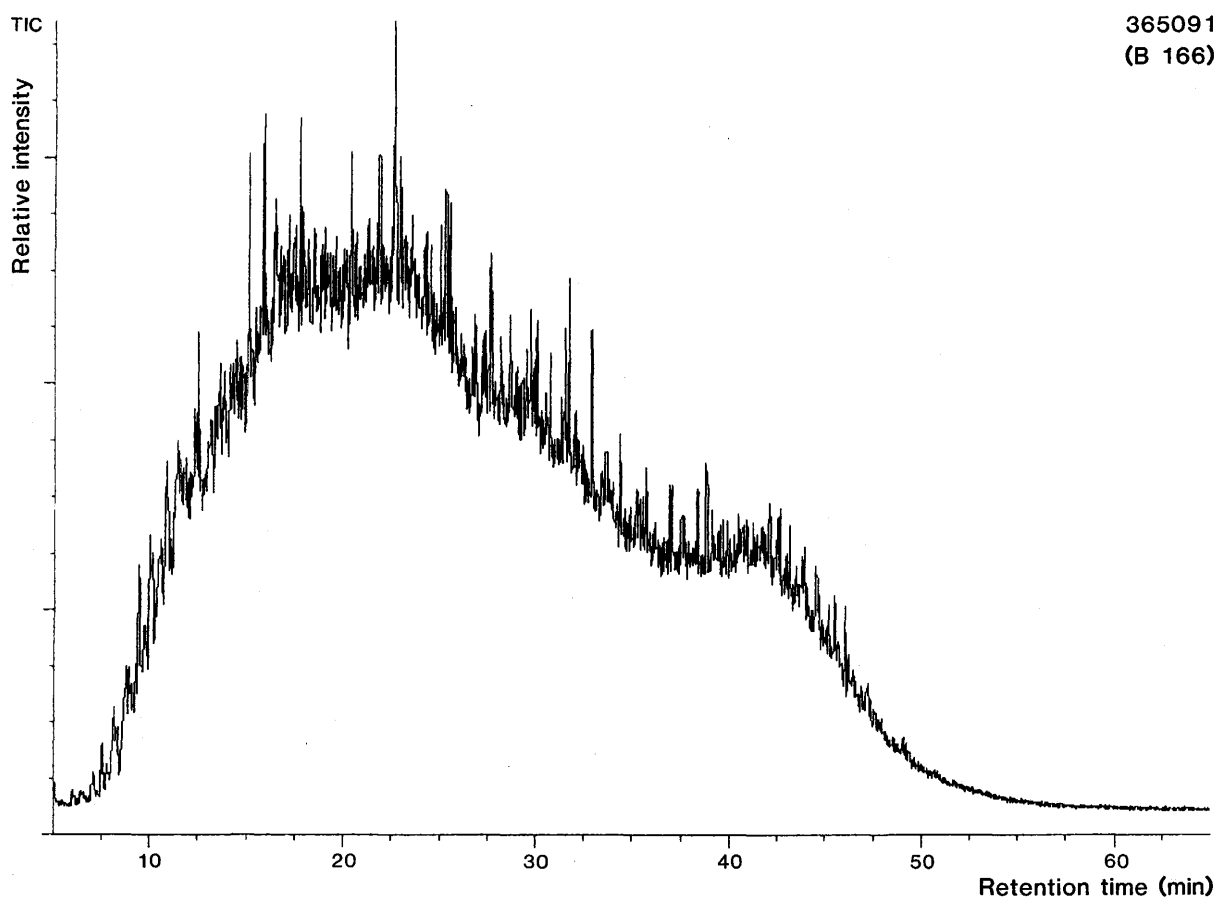
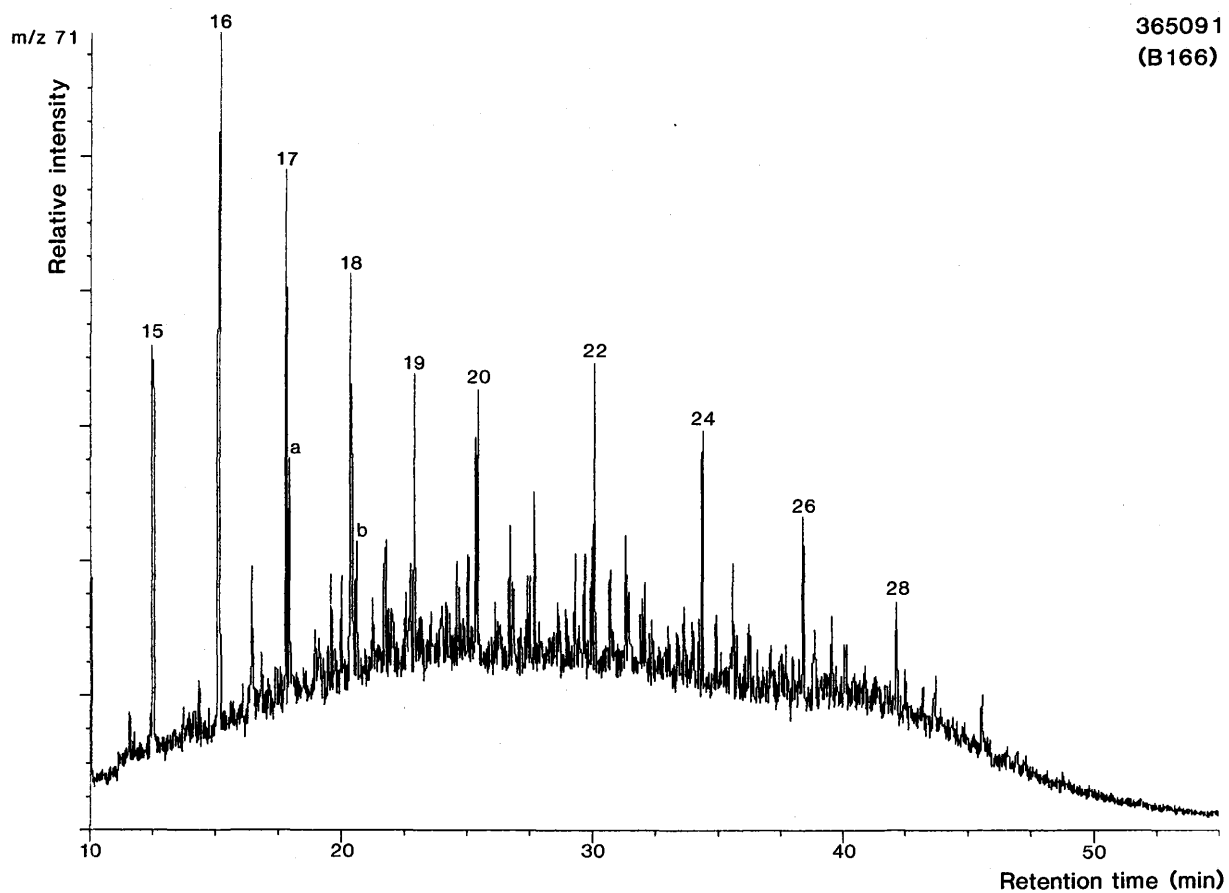


Fig. 8. Mass chromatogram (m/z 71) and total ion chromatogram (TIC) of the saturated hydrocarbons in GGU 365091-2 (B 166) showing distribution of *n*-alkanes (carbon-numbers indicated) and isoprenoids. (a: pristane, b: phytane).

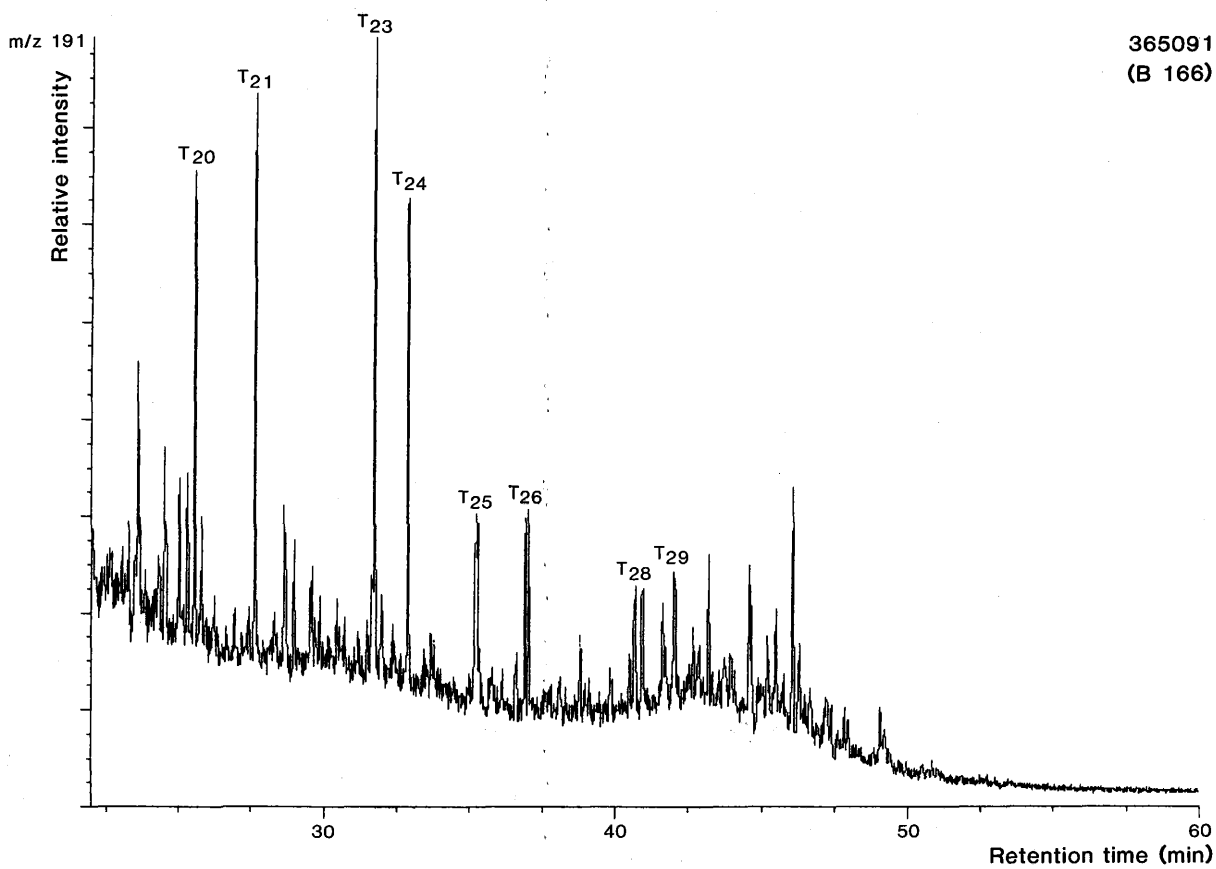


Fig. 9. Mass chromatogram (m/z 191) of the saturated hydrocarbons in GGU 365091-2 (B 166) showing distribution of terpanes. T_{20} to T_{29} are all tricyclic terpanes.

Table 1 Porosity and permeability data (A before cleaning with toluene; B after cleaning with toluene)

	Porosity A(%)	Porosity B(%)	Permeability (mD)	Grain dens. A (g/cm ³)	Grain dens. B (g/cm ³)
365085-1	3.43	3.93	0.012	2.699	2.707
365085-2	9.20	9.85	2.313	3.127	3.141
365085-3	11.79	15.44	(fractured)	2.762	2.831
365091-1	4.77	7.96	0.409	2.696	2.766
365091-2	7.28	11.65	3.047	2.700	2.791 (0.86)
Average:	7.29±3.36	9.77±4.27	-	2.821±0.206	2.847±0.170

Average saturation: $\frac{9.77-7.29}{9.77} \times 100(\%) = 25.3(\%)$ (+ loss during sampling + prep.)

Table 2 Leco and Rock Eval data

	TOC(%)	Tmax(°C)	(mg HC/g rock)		(100xS2/TOC)	(100xS1+S2/TOC)
			S1	S2	HI	HI'
365085 (bulk)	0.55	411	4.88	1.18	216	1101

Table 3 Extraction data

		<u>Extraction</u>			<u>Deasphalting</u>			
		TOC(%)	Amount (g)	SOM(g)	Amount (mg)	Asph. (mg)	HC+NSO(mg)	Loss (mg)
365085	B 163	0.529	20.0	0.1946	86.4	16.6	66.9	(2.9)
365085	B 164	0.563	20.0	0.2014	84.2	16.0	68.4	(-0.2)
365085-3	B 165	0.372	20.0	0.0559	55.9	6.2	52.6	(-2.9)
365091-2	B 166	1.680	20.0	0.4138	77.0	10.8	70.1	(-3.9)

		Sat. (%)	Aro. (%)	NSO(%)	Asph. (%)	HC:non-HC	Sat:Aro
365085	B 163	32	19	30	19	1.0	1.7
365085	B 164	32	18	31	19	1.0	1.8
365085-3	B 165	32	18	39	11	1.0	1.8
365091-2	B 166	38	18	41	3	1.3	2.1

Table 4 Pyrolysis - gas chromatography (py-GC) data

			C1-C5 norm%	C6-C14 n-alkyl norm%	C15+ n-alkyl norm%	C6+ n-alkyl norm%	C6+ arom+phenol norm%	C6+ unknowns norm%
365085	B 164	ASPH	76	21	3	35	20	45
365085-3	B 165	ASPH	78	19	3	33	22	45
365091-2	B 166	ASPH	78	19	3	32	22	46

Table 5 Distribution of n-alkanes and isoprenoids calculated from the (m/z 71) mass chromatograms

	Resolved	Max	Pr/Ph	Pr/nC ₁₇	Ph/nC ₁₈	Dominance
B 164	poor	17-20	1.27	1.07	1.08	(20)
B 165	good	16-17	1.42	0.38	0.32	(22)-24-26-28
B 166	good	16-17	1.05	0.45	0.54	22-24-26-28

Table 6 Relative distribution of tricyclic terpanes calculated from the (m/z 191) mass chromatograms

		T ₂₀	T ₂₁	T ₂₃	T ₂₄	T ₂₅	T ₂₆	T ₂₈	T ₂₉	T ₂₃	T ₂₄	T ₂₅
B 164	(IKU)	13	13	21	18	9	9	8	9	45	37	18
	(DGU)	14	18	24	19	7	8	5	5	49	38	13
B 165	(IKU)	17	12	16	16	8	10	10	11	41	40	19
	(DGU)	23	13	18	16	7	8	7	8	42	40	18
B 166	(IKU)	14	17	21	19	7	9	6	7	44	40	16
	(DGU)	<u>16</u>	<u>20</u>	<u>23</u>	<u>17</u>	<u>7</u>	<u>7</u>	<u>5</u>	<u>5</u>	<u>48</u>	<u>37</u>	<u>15</u>
Average		16	16	21	18	7	8	7	7	45	39	16

