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Organic compounds from the Rhaetic-Liassic coals of Scoresby Sund, East Greenland

by

K. Raunsgaard Pedersen and Jørgen Lam

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Abstract

A large number of aromatic and hydroaromatic hydrocarbons were detected by gas chromatography and mass spectrometry from the bituminous coals. A minor part consists of saturated aliphatic hydrocarbons in which the distribution of normal alkanes in the range C_{24} to C_{32} shows a similarity to that of recent plant material with an odd/even distribution rate higher than one. A relatively high amount of pristane is present which is not known in higher plants. A heterocyclic compound, dibenzofuran, was isolated and characterized, and a methyl dibenzofuran seems to be present. Monoterpenoid compounds and minor amounts of acids were detected; myristic and palmitic acids were characterized as methyl esters.

The coal is derived from the Kap Stewart Formation which bears a well known fossil flora dominated by gymnospermous plants and without angiosperms. Some of the extracted organic compounds are present in recent gymnosperms. In recent material dibenzofuran and derivatives are only known in lichens and one angiospermous species.

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GEOLOGY, FOSSIL FLORA
AND MATERIAL

The Kap Stewart Formation outcrops in south-western Liverpool Land, in the eastern part of Jameson Land along Hurry Inlet, and further north in Klitdal and northern Jameson Land (fig. 1; Surlyk & Birkelund, 1972; Surlyk *et al.*, 1973). In the southern area the Kap Stewart Formation is about 150 m thick and consists mainly of arkosic sandstones. In the upper part of the formation dark carbonaceous siltstone and shale layers of variable thickness also occur, together with thin coal layers and root beds. The shale and siltstone layers are locally very rich in plant fossils.

The fossil flora (the 'Scoresby Sound flora') of the Kap Stewart Formation has been described by Harris (1926, 1931, 1932 a & b, 1935, 1937) and divided into
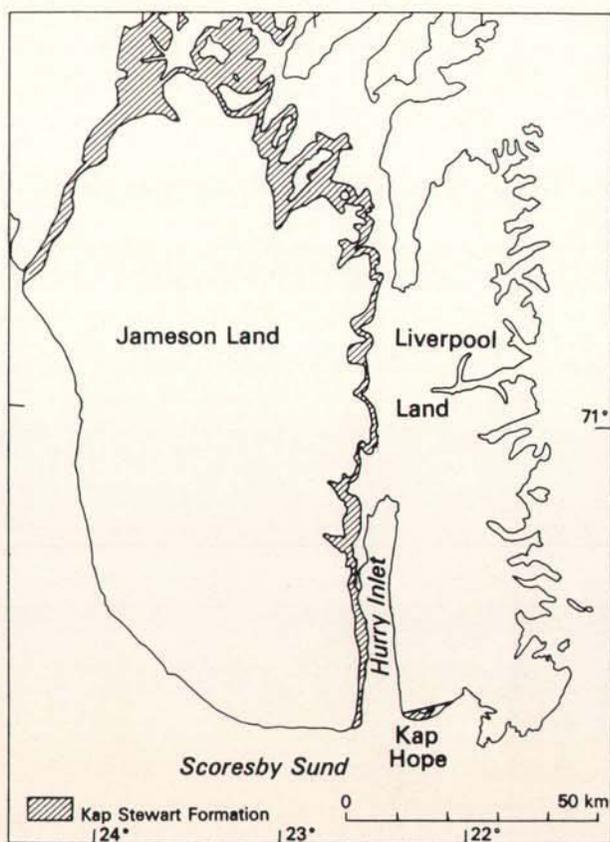
Fig. 1. Map of Jameson Land and Liverpool Land showing the outcrop of the Kap Stewart Formation. (After Surlyk & Birkelund, 1972).

two floral zones, the *Lepidopteris* flora and the *Thaumatopteris* flora, which have few species in common. The two zones are dated by Harris as Upper Rhaetic and Lower Liassic respectively. The two floras, with about 200 species in total, are dominated by gymnospermous plant groups: Cycadophytes, Ginkgophytes and conifers, with ferns and horsetails also common. No angiosperms are found in the Kap Stewart Formation. Most of the described species are leaf fossils, but reproductive structures and wood have been collected.

Among the Cycadophytes the extinct order Bennettitales is best represented, with many types of leaves and reproductive structures, although members of the extant order Cycadales occur. The Ginkgophytes are especially represented by many leaf species of *Baiera* and *Ginkgoites*. Single-veined, and several-veined broad leaves, and reproductive structures of several species of conifers are present. The most common types of fern are leaves of the families Dipteridaceae (especially *Dictyophyllum*) and Osmundaceae, while *Equisetites* and *Neocalamites* are the common horsetails.

The fossil plants represent the vegetation of an estaurine marsh area with good growth conditions under a rather warm and humid climate (Harris, 1937).

Plant beds are especially common in the upper part of the formation (fig. 3). In some of the beds many large and well-preserved fossil leaves of local origin are found (autochthonous plant layers). Other beds with coarser sediments contain fragments of transported leaves and wood. These allochtonous plant beds have a flora brought together from a greater area. Root horizons are found together with the first mentioned type of plant beds, and pass up into thin coal seams. Most of the coal seams are only a few centimetres in thickness but locally a few seams reach a maximum of about 50 cm. Some of the thin coal seams are composed of fossil leaves, others of tree trunks up to 50 cm or more in diameter. The thick coal seams are mainly composed of fossil wood (Harris, 1937).
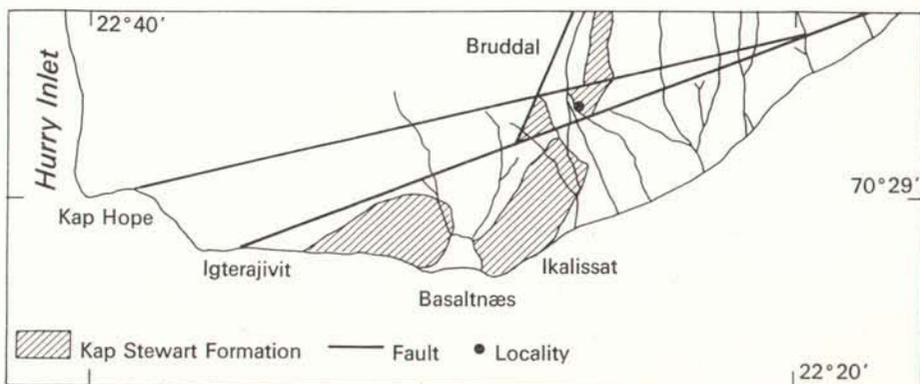
Fig. 2. Map of the Kap Hope area, southern Liverpool Land, showing the position of the location of the sample. (After Rosenkrantz, 1942).

Fig. 3. Exposure of upper part of the Kap Stewart Formation in Astartekløft, western Jameson Land.

The investigated coal sample was derived from the Kap Hope area, between the settlement of Scoresbysund and Hurry Inlet, at the south-west corner of Liverpool Land. Here an area with Mesozoic sediments is bounded by faults (figs 1, 2). The sediments have been mapped and described by Rosenkrantz (1934, 1942) who recognized Triassic, Rhaetic-Liassic and Jurassic limnic and marine sediments. The Kap Stewart Formation is exposed in a low coastal section and in small profiles in Bruddal about 1.5 km from the coast.

Fossil plants collected in the Kap Stewart Formation of the Kap Hope area demonstrate that both the *Lepidopteris* zone and the *Thaumatopteris* zone of Harris are represented.

Thin coal seams have been reported from several localities in the plant-bearing series and at two of these a seam of about 50 cm thickness occurs. The fossil plants of the sediments containing the thick coal seam are referred to the flora of the *Thaumatopteris* zone and therefore belong to the upper part of the Kap Stewart Formation of Lower Liassic age. At Ikalissat, in the coastal section, the lenticular coal seam has been followed for about 40 m and attains a thickness of 53 cm. The seam lies in an easterly dipping series overlain by a compact coarse, cross-bedded sandstone which is followed by the marine 'Pecten sandstone' of Middle

Table 1. Analyses of coal from the Kap Hope area

Constituents	Ikalissat	Igterajivit near Kap Hope settlement	Bruddal
Volatile matter	38.5	39.7	24.4
Fixed carbon	48.5	42.4	42.7
Moisture	2.4	2.0	3.6
Ash	10.6	15.9	29.3
Moisture	2.4	2.0	3.6
Ash	10.6	15.9	29.3
Sulphur	1.03	1.72	0.76
Hydrogen	5.5	5.0	3.3
Carbon (total)	71.2	66.5	53.0
Nitrogen	1.00	0.87	0.64
Oxygen	8.3	8.0	9.4
Coke	59.1	58.3	72.0
Upper calorific power cal/kg	7117	6857	5061
Lower calorific power, effective cal/kg	6810	6583	4866
Specific gravity	1.45	1.53	1.72

Analyses from Rosenkrantz, 1942, p. 21.
Analyst: Danish State Testing Laboratory.

Liassic age, and younger Liassic marine sediments. This coal seam has been mined for local use for many years and in 1970 it was not possible to reach the seam below the sandstone spoil from the old mining operations.

Rosenkrantz (1942) states that the coal seam is of rather uniform composition throughout the whole bed and consists of durite with numerous veins of vitrite. Analyses of the coals of the Kap Hope area classify them as bituminous coals but samples from three different localities do show some differences (table 1).

About 1.5 km north of Ikalissat, on the eastern slope of Bruddal near the coastal plain, Rosenkrantz (1942) describes a coal seam in a small section in the upper part of the plant-bearing series overlain by marine Middle Liassic sediments (fig. 4) which probably represents about the same level in the Kap Stewart Formation as the seam at Ikalissat.

This Bruddal coal seam has also been mined, but less extensively, and it was possible in september 1970 to obtain a sample of the coal at this locality (fig. 5). The coal was rather cracked and with many thin shale layers. The sample (GGU 134323) was dug out from below the surface. There is no vegetation at the locality so that there should be no contamination of the sample by recent hydrocarbons.

The coal consists of alternating layers of durite and vitrite. The vitrite layers re-

Fig. 4. The locality of the coal seam in the southern part of Bruddal. The plant-bearing
series to the left is overlain by marine sediments with large blocks in the scree.
(After Rosenkrantz, 1942).

present stems of probably gymnospermous trees. The sample from Bruddal in table
1 is from the same seam as the sample analysed here for hydrocarbon compounds.
The lower calorific value could be a result of less disturbance of this part of the
series.

Fig. 5. The coal layer in the plant-bearing series in the outer part of Bruddal.

EXTRACTION AND ANALYTICAL METHODS

As stated in previous publications (Pedersen & Lam, 1970; Lam & Pedersen, 1972) the possibility of contamination with various phthalic esters is very great. This fact has been taken into consideration in the choice of apparatus and material for the purpose of extraction and separation of the organic substances present in the geological material. Thus, the crude material was rinsed with a mixture of distilled benzene-methanol 3:1 v/v, which is also the solvent mixture used for extraction of the geological material.

The rinsed sample was finally milled in an automatic mortar and the pulverized material extracted in portions of about 700 g. Each portion was extracted three times with a 900 ml mixture of benzene and methanol (3:1 v/v) with an extraction time of three days. The first extract of the 1st 700 g portion was stored, the second extract was used for the first extraction of the 2nd 700 g portion of sample and then stored. The third extract of the 1st 700 g portion was first used for the second extraction of the 2nd 700 g portion and then for the first extraction of the 3rd 700 g portion of sample. This procedure allows a minimum use of solvent and hence a limitation of contamination possibilities.

After re-extraction of the material, the evaporated crude material was separated into fractions on a silicagel column with n-heptane, and subsequently with mixtures of n-heptane and ether, in which the amount of ether was gradually increased from 1 per cent to 100 per cent, as eluting solvents.

Finally the column was washed with methanol. The first fractions passing through the column contain saturated hydrocarbons followed by hydroaromatic and aromatic hydrocarbons. The polarity of compounds passing through the column increases with the increasing ether per cent.

Infra-red and ultra-violet spectral data were used for primary orientation but the main identification was accomplished by a combination of gas chromatography and mass spectrometry. The instruments used for the analytical work were a Perkin-Elmer 880 gas chromatograph, equipped with a 2 m 10 per cent S.E. 30 gaschrom Z column, and a Hitachi-Perkin-Elmer RMU-6D mass spectrometer, and a Beckmann GC-M gas chromatograph in combination with a CEC 21-104 mass spectrometer for part of the work.

Single compounds such as dibenzofuran and pyrene were isolated by the use of thin layer chromatography. The data obtained by this technique for these compounds agreed well with the corresponding data for authentic compounds. In addition the ultra-violet spectra and the gas chromatography data agreed well with the commercial compounds.

RESULTS

The sample was treated according to the method described by Pedersen & Lam (1970). An amount of 3185 g of finely milled material was extracted with benzene-methanol 3:1 v/v. The residue after evaporation gave a black tarry material. After repeated extraction of this residue with n-heptane, and after evaporation of the solvent, 5.7464 g of dark brown material resulted. About 10 per cent (582 mg) of the extract was separated on a silicagel column and 23 fractions were collected, the first fractions being the less polar and the subsequent ones showing an increase in polarity. Fractions 1 to 7 contained only about 20 per cent of the material separated on the column and these fractions mainly consisted of a complex mixture of saturated hydrocarbons, largely of branched and cyclic type but with some straight chain forms. The main part of the remaining fractions consisted of aromatic and hydroaromatic hydrocarbons. The aromatic hydrocarbons contribute a proportionally higher amount of the total organic material than in the previously examined samples from the Precambrian where the percentage of aliphatic hydrocarbons was higher. Besides the naphthalenic and phenanthrenic structures, a great number of more or less saturated ring systems related to naphthalene and phenanthrene in the various forms of alkyl derivatives were distributed over several fractions. They indicated fragments from steroid structures.

A series of mass spectra from the fractions following the aromatic fractions indicated the presence of monoterpenoid substances with molecular weights of 150, 152 or 154. The infra-red spectra of the most polar fractions indicated the presence
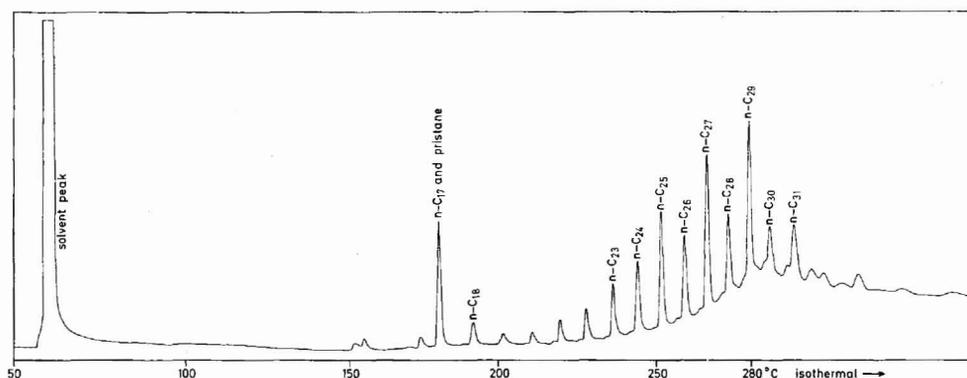
Fig. 6. GGU sample no. 134323. Gas chromatogram of fraction of saturated hydrocarbon, mainly normal paraffins. Pristane (a C_{19} isoprenoid) is, however, present in a relatively high amount. Column 10% S. E. 30, 20 m, 6°C/min. 50–250°C, then isothermal.
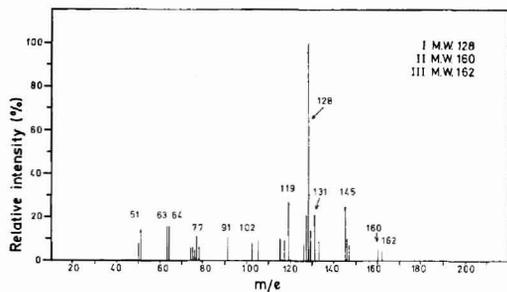
Fig. 7. Mass spectrum of naphthalene (M.W. 128), an alkyl tetrahydronaphthalene (M.W. 160) and an alkyl benzene (77, 91, 105, 119, 133, 147 and 162 m/e; M.W. 162).
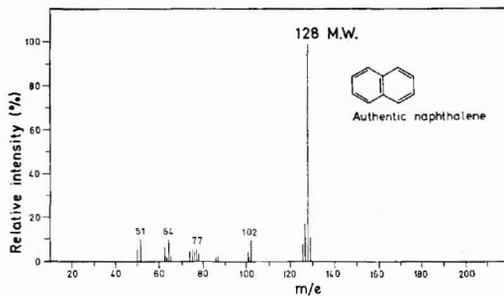
Fig. 8. Mass spectrum of authentic naphthalene.
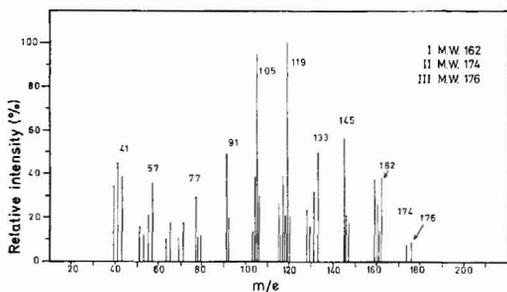
Fig. 9 Mass spectrum of alkyl benzenes M.W. 162 and M.W. 176 and an alkyl tetrahydronaphthalene (131, 145, 159 and 174 m/e; M.W. 174).
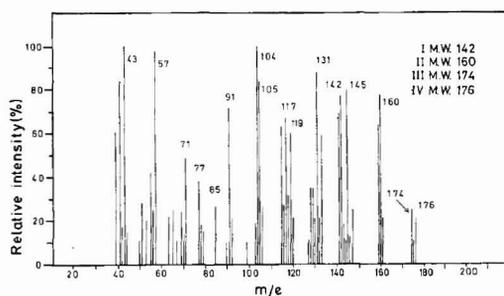
Fig. 10. A complex mass spectrum from which may be deduced the presence of a methyl naphthalene (M.W. 142), two alkyl tetrahydronaphthalenes (M.W. 160 and M.W. 174) and an alkyl benzene (M.W. 176).
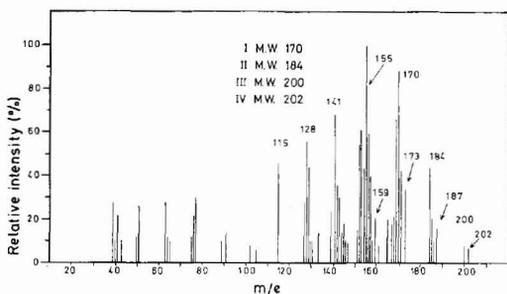
Fig. 11. Mass spectrum of two alkyl naphthalenes M.W. 170 and M.W. 184, an alkyl tetrahydronaphthalene M.W. 202 and an alkyl dihydronaphthalene (M.W. 200).
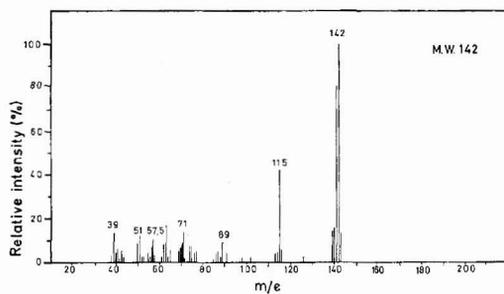
Fig. 12. Mass spectrum of a methyl naphthalene.

of acids, apparently both aliphatic and aromatic, but these were not fully characterized.

A more detailed discussion of the spectral data illustrated in fig. 6 to fig. 24 is given below. Figure 6 shows a gas chromatogram of a fraction from the sample containing a mixture of saturated straight chain and branched chain hydrocarbons with a dominating pattern of normal alkanes ranging from C_{13} to C_{32}. The peak corresponding to C_{17} is, however, dominated by pristane identified by its characteristic mass spectrum and from a gas chromatogram after addition of pristane to the mixture of alkanes. The normal alkanes C_{13} to C_{18} were detected by their mass spectra, whereas the higher alkanes were detected by gas chromatographic comparison with authentic normal C_{24} and normal C_{28} hydrocarbons. From the gas chromatogram it is seen that the odd/even carbon number ratio is higher than one in the region C_{24} to C_{32}. This distribution shows similarity to that of recent plant material (Stransky, Streibl & Herout, 1967) and deposits such as lignite, brown coal and montan wax (Mazliak, 1968; Wollrab et al., 1962) whereas in the previously investigated Precambrian material the odd/even ratio was closer to one.

Saturated isoprenoid C_{13} to C_{21} hydrocarbons were detected, with pristane as the most dominant compound of this series of hydrocarbons. The aromatic fractions consist of a large number of alkyl phenanthrenes and hydroaromatic compounds such as di- and tetrahydronaphthalenes and di- and tetrahydrophenanthrenes.

Dibenzofuran and alkyl dibenzofuran appear in the aromatic hydrocarbon fractions. The dibenzofurans are oxygen containing heterocycles with a polarity similar to that of the aromatic hydrocarbons.

Fig. 7 shows the mass spectrum of naphthalene of molecular weight (M.W.) 128 in a mixture with an alkyl benzene and an alkyl tetrahydronaphthalene, the benzene derivative with a molecular weight of 162 and the alkyl tetrahydronaphthalene with a molecular weight of 160. Fig. 8 is the mass spectrum of authentic naphthalene. A fragmentation corresponding mainly to benzene derivatives M.W. 162 and M.W. 176 (alkyl benzenes with six and seven carbon atoms respectively attached to the benzene ring) is shown in fig. 9. An alkyl tetrahydronaphthalene (M.W. 174) contributes to the pattern of fragments in the mass spectrum with prominent peaks at 131, 145, 159 and 174 m/e. Fig. 10 shows a complex fragmentation pattern of a methyl naphthalene (M.W. 142), a dimethyl and trimethyl tetrahydronaphthalene (M.W. 160 and 174 respectively) and finally of an alkyl benzene (M.W. 176). Fig. 11 predominantly shows alkyl naphthalenes (M.W. 170 and 184 respectively) with a minor contribution from an alkyl tetrahydronaphthalene (M.W. 202) and perhaps also a dihydronaphthalene derivative (M.W. 200). Mass spectra of alkyl naphthalenes with one and two carbon atoms respectively in the side chains are shown in figs 12 and 13. The mass spectrum of a mixture of alkyl naphthalenes with 3 and 4 carbon atoms attached to the naphthalene ring (M.W. 170 and M.W. 184) is shown in fig. 14. Generally alkyl naphthalenes appear in the separation on S.E. 30 columns in the gas chromatograph together with tetra-
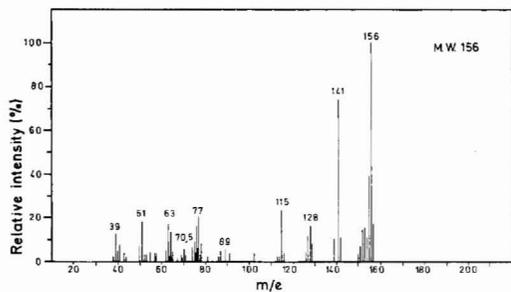
Fig. 13. Mass spectrum of a dimethyl or an ethyl
naphthalene.
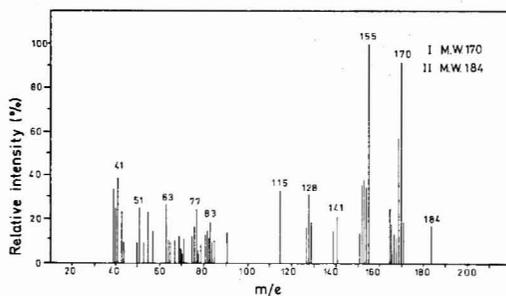
Fig. 14. Mass spectrum of alkyl naphthalene
(M.W. 170 and 184).
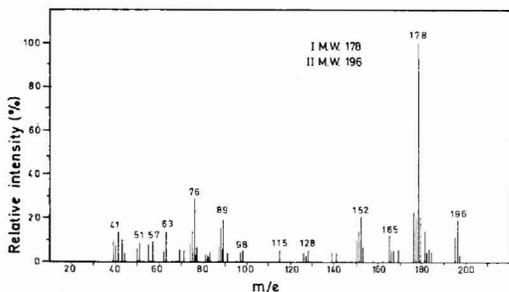
Fig. 15. Mass spectrum of phenanthrene M.W.
178 and a methyl tetrahydrophenanthrene
(M.W. 196).
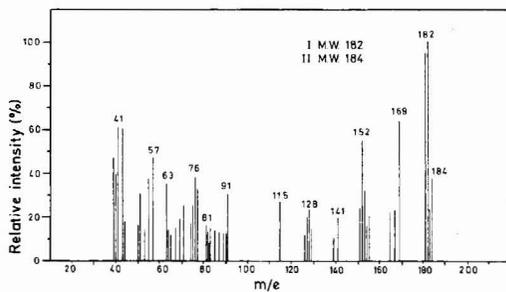
Fig. 16. Mass spectrum of an alkyl naphthalene
(M.W. 184) and tetrahydrophenanthrene?
(M.W. 182).
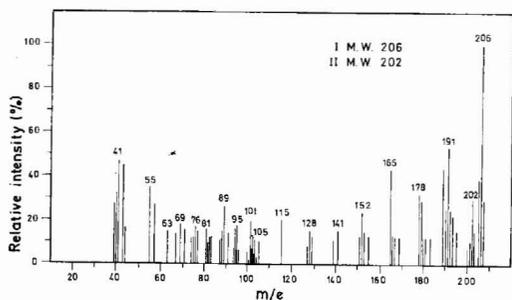
Fig. 17. Mass spectrum of a dimethyl or ethyl
phenanthrene (M.W. 206) and pyrene (M.W. 202).
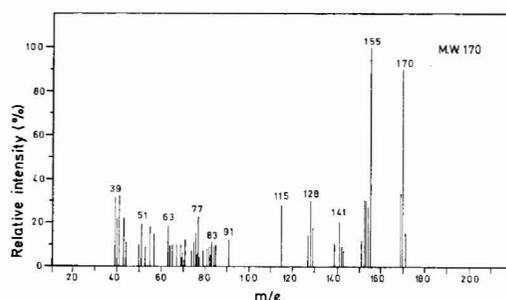
Fig. 18. Mass spectrum of a trimethyl naphthalene.

hydronaphthalenes possessing a molecular weight 18 units higher (4 hydrogen atoms plus 14 from the substitution of a hydrogen atom with a methyl group). Similarly phenanthrene is present in a mixture with a methyl tetrahydrophenanthrene M.W. 178 and M.W. 196 respectively (fig. 15). Fig. 16 shows a naphthalene derivative with 4 carbon atoms attached to the naphthalene ring system (M.W. 184) and possibly tetrahydrophenanthrene M.W. 182. (90.5 and 91 m/e fragments indicate
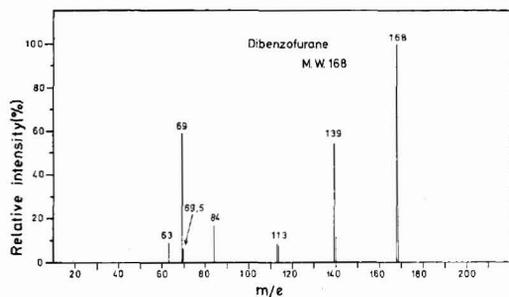
Fig. 19. Mass spectrum of authentic dibenzofuran.
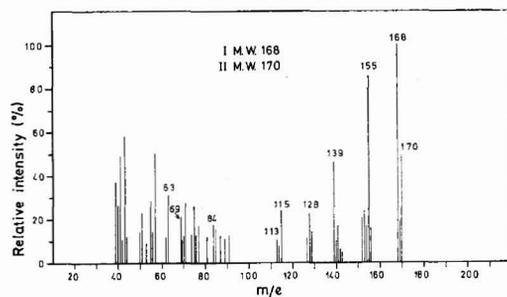
Fig. 20. Mass spectrum of dibenzofuran (M.W. 168) in a mixture with trimethyl naphthalene (M.W. 170).

double charged ions of 181 and 182 m/e). Fig. 17 is the mass spectrum of a dimethyl phenanthrene (M.W. 206).

It furthermore shows fragments indicating the presence of pyrene 202 m/e (parent peak) and 101 m/e, which are the most prominent peaks of authentic pyrene. Double charged fragments at 94.5 and 95.5 and at 100.5 and 101.5 agree well with the mass spectra of alkyl phenanthrenes and pyrene respectively. Phenanthrene and pyrene were isolated and the ultra-violet spectra agree well with the ultra-violet spectra of the authentic compounds.

In successive fractions of aromatic hydrocarbons dibenzofuran appeared together with a trimethyl naphthalene (M.W. 168 and M.W. 170 respectively). Fig. 18 shows the mass spectral pattern of a trimethyl naphthalene, fig. 19 is the mass spectrum of authentic dibenzofuran and fig. 20 shows a mass spectrum from the sample of a mixture of dibenzofuran and a trimethyl naphthalene. Fig. 21 and fig. 22 are the ultra-violet spectra of the isolated dibenzofuran and the commercial compound respectively. Calculation on the basis of the ultra-violet spectrum revealed the presence of about 1 part per thousand of dibenzofuran in the total organic extract.

Minor fractions following the 'aromatic' hydrocarbon fractions contain monoterpenoid and perhaps sesquiterpenoid compounds. They can be revealed due to the fragmentation pattern, shown by their mass spectra, of the monoterpenoids having mole peaks in the range of 150 to 154 or occasionally missing a mole peak, where a possibility of splitting off an OH-group or water is likely. Fig. 23 is the mass spectrum of a compound having the mole peak of 152 m/e. Furthermore, the mass spectrum shows some similarity with that of camphor which was first reported to be present in Precambrian material from the Ketilidian of South-West Greenland (Pedersen & Lam, 1968). Fig. 24 is the mass spectra of a monoterpenoid compound which most likely has a molecular weight of 154. The mole peak is missing but fragments of 139, 136 and 137 m/e indicate a molecular weight of 154; in that case the 139 m/e fragment will be due to a (M-15) fragmentation (methyl), the

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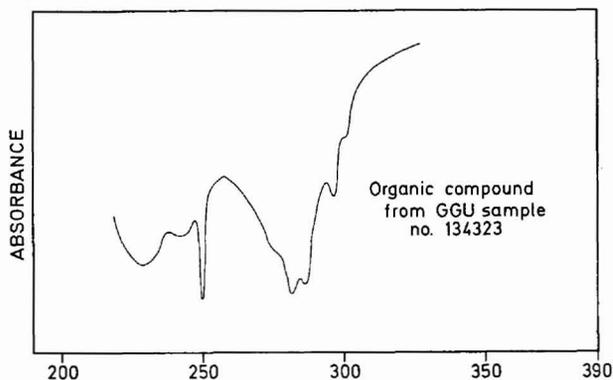
Fig. 21. Ultra-violet spectrum of dibenzofuran isolated from the GGU sample no. 134323.
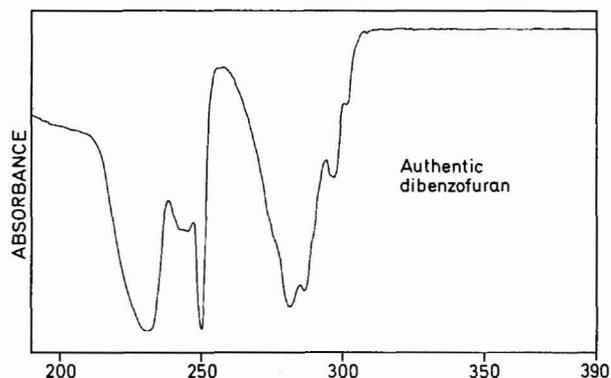
Fig. 22. Ultra-violet spectrum of the commercial dibenzofuran used for comparison with the isolated compound.

137 m/e fragment and 136 m/e fragment originating from the splitting off of OH and H$_2$O respectively.

A minor fatty acid fraction revealed a large number of fatty acids after their conversion to the corresponding methyl esters, the identification being mainly based on the mass spectral data (74, 87, 101, 115, 129, 143 m/e and higher fragments characteristic for methyl esters of fatty acids). The small quantity of complexly
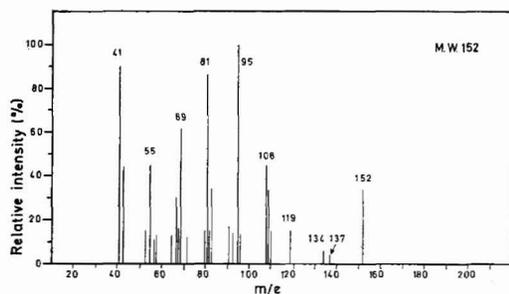
Fig. 23. Mass spectrum of a monoterpenoid compound from GGU sample no. 134323 (for explanation see text).
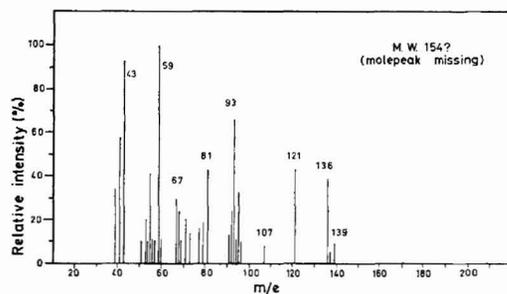
Fig. 24. Mass spectrum of a monoterpenoid compound from GGU sample no. 134323 (for explanation see text).

mixed material was insufficient to allow full characterization of the fatty acids. C_{14} and C_{16} fatty acids of saturated nature were, however, identified with some confidence.

Fragmentation patterns of the most polar fractions indicate the presence of naphthalenic acids and phenols.

CONCLUDING REMARKS

The chemical data of the present sample (table 2) of organic material from the Kap Stewart Formation (Rhaetic-Liassic) are somewhat different from those obtained from the previous analyses of Precambrian organic material from Greenland (Lam & Pedersen, 1972). Most of the compounds in the homologous series of hydrocarbons (aliphatic and aromatic) seem to be present in each of the extracts, as well as hydroaromatic alkyl substituted compounds. However, there are important differences between the samples in the relative proportions of the various constituents.

Table 2. Organic compounds derived from the Mesozoic bituminous coal

Saturated aliphatic hydrocarbons	paraffins (normal alkanes C_{13}–C_{32}) pristane (C_{19} isoprenoid)
Aromatic hydrocarbons	alkyl benzene (M.W. 162, 176) naphthalene (M.W. 128) methyl naphthalene (M.W. 142) dimethyl/ethyl naphthalene (M.W. 156) alkyl naphthalenes (M.W. 170, 184, etc.) alkyl dihydronaphthalene (e. g. M.W. 200) alkyl tetrahydronaphthalenes (M.W. 160, 174, 188, 200) phenanthrene (M.W. 178) dimethyl/ethyl phenanthrene (M.W. 206) tetrahydrophenanthrene? (M.W. 182) methyl tetrahydrophenanthrene (M.W. 196) pyrene (M.W. 202)
Heterocyclic compounds	dibenzofuran (M.W. 168) methyl dibenzofuran (M.W. 182) monoterpenoids (M.W. 150, 152 or 154)
Fatty acids	C_{14}–C_{16} myristic acid palmitic acid aromatic (naphthalenic) acids and phenols seem to be present in the most polar fractions

Sample GGU 134323 from Bruddal, near Kap Hope.

The Rhaetic-Liassic material contains a large amount of pristane but little or no phytane whereas in the Precambrian samples there was only a small amount of pristane. Furthermore, the pattern of the saturated normal hydrocarbons in the range of C_{24} to C_{32} shows an odd/even distribution greater than one, which was not seen in the previously published analyses of Precambrian samples, where the distribution was closer to one. The pattern of the relative distribution of the hydrocarbons is seen in fig. 6, and is very much like that reported from recent gymnospermous and angiospermous plants (Stransky, Streibl & Herout, 1967; Eglinton & Hamilton, 1963) although the odd/even ratio of paraffins in recent plant material is usually higher than in the paraffins isolated from the material extracted here.

Alkyl benzenes, alkyl naphthalenes, alkyl phenanthrenes and their parent compounds are present in homologous series together with hydroaromatic alkyl derivatives. Other aromatic compounds such as diphenyl, alkyl diphenyl and pyrene were also characterized. Large amounts of alkyl benzenes, alkyl naphthalenes, alkyl phenanthrenes and corresponding hydroaromatic homologous series may be explained as originating from steroids and other terpenoid compounds. Dibenzofuran and methyl dibenzofuran are heterocyclic compounds, which have previously been reported to be present in petroleum (Eglinton & Murphy, 1969) and in brown coal distillates (Popl & Mostecky, 1972). Dibenzofuran derivatives are reported only from a few recent materials. Naturally occurring dibenzofuran derivatives have been isolated by Santesson, Åkermark, Erdtmann, Wachtmeister & Huneck (Huneck, 1968) from lichens and by Anderson *et al.* (1969) from the angiosperm *Rhodomyrtus macrocarpa* Benth. (Myrtaceae).

The coal sample investigated is the remnant of a coalification of a Liassic fossil flora. The large fossil flora known from the same formation (the Kap Stewart Formation) as the coal layer is dominated by gymnospermous plants without angiosperms. The aliphatic hydrocarbons are known from recent gymnosperms while dibenzofuran could possibly be derived from lichens. No fossil representatives of lichens are known from the Kap Stewart Formation, but a possible explanation of the presence of these compounds could be that certain lichen types lived on stems and perhaps were active during the decomposition of the plant remains.

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