49. ORGANIC GEOCHEMICAL CHARACTER AND HYDROCARBON SOURCE POTENTIAL OF SELECTED BLACK SHALES, DEEP SEA DRILLING PROJECT HOLE 603B¹

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ABSTRACT

LECO analysis, pyrolysis assay, and bitumen and elemental analysis were used to characterize the organic matter of 23 black shale samples from Deep Sea Drilling Project Leg 93, Hole 603B, located in the western North Atlantic. The organic matter is dominantly gas-prone and/or refractory. Two cores within the Turonian and Cenomanian, however, contained significant quantities of well-preserved, hydrogen-enriched, organic matter. This material is thermally immature and represents a potential oil-prone source rock. These sediments do not appear to have been deposited within a stagnant, euxinic ocean as would be consistent with an "oceanic anoxic event." Their organic geochemical and sedimentary character is more consistent with deposition by turbidity currents originating on the continental shelf and slope.

INTRODUCTION

Atlantic Ocean Cretaceous black shales appear to have had several different modes of formation as reflected by their sedimentary characteristics (bedding and mineralogy) and organic geochemical nature (Tissot et al., 1979, 1980; Katz and Pheifer, 1982, 1986; Graciansky et al., 1982; Waples, 1983). This chapter deals specifically with the variation in organic matter concentration and character in a series of 23 Hauterivian to lower Eocene(?) "black shales" recovered from DSDP Hole 603B. Black shale is actually a misnomer, since many of these rocks may be better classified as marls, limestones, or mudstones and may be dark gray or dark green rather than black. The organic geochemical information derived as part of this study will be used to provide further insight into the modes of origin of the black shales and to provide some information on the hydrocarbon source-rock potential of the western North Atlantic continental rise.

Site 603 (35°29.71'N, 70°01.71'W) is located at the western extreme of the Hatteras Abyssal Plain at the foot of the lower continental rise hills, near previously drilled DSDP Site 388 (Fig. 1). The site rests within the Jurassic magnetic quiet zone on presumably Oxfordian crust (Ogg, this volume). This location was selected because it offered an opportunity to bridge the gap between the continental margin and the ocean basin proper (i.e., the depositional history has been affected by both oceanic and coastal events).

Hole 603B, the deepest of three drilling attempts at this site, penetrated 1576 m of sediment and bottomed approximately 200 m short of basement. A major discovery at the site was a Lower Cretaceous deep-sea fan complex including coarse terrigenous sands and black carbonaceous muds. Such a complex suggests that hydrocarbon reservoirs and source rocks may be closely associated in the deep-water environment of the eastern United States continental margin.

Within the marine environment three types of organic matter are commonly found: type II, well-preserved organic matter primarily of marine origin; type III, derived from higher plants or poorly preserved (oxidized) marine organic matter; and type IV (or residual organic matter—ROM), severely stressed (oxidized) and/or resedimented material. Each principal type of organic matter may be identified through the use of several techniques, including pyrolytic assay, gravimetric and chromatographic analysis of the bitumen (soluble) fraction, and elemental analysis of isolated kerogen (insoluble) components.

In general, immature type II material exhibits a moderately high atomic hydrogen-to-carbon (H/C) ratio and a low atomic oxygen-to-carbon (O/C) ratio. It may be characterized by high yields of total extractables and pyrolytic hydrocarbons relative to organic carbon content. Such material upon attainment of a sufficient level of maturation generates and expels oil (Tissot et al., 1974). Type III organic matter exhibits low to moderate H/C ratios and moderate O/C ratios. The extractable and pyrolytic yields relative to organic carbon are commonly low. Consequently, type III materials produce gas on attainment of sufficient levels of maturation (Tissot et al., 1974). Type IV material exhibits very low H/C ratios and characteristically displays highly variable O/C values. This material yields very few extractable products of pyrolytic hydrocarbons (Tissot et al., 1979) and is incapable of acting as a hydrocarbon source.

ANALYTICAL METHODOLOGY

Twenty-three samples, ~30 g each, were analyzed. Stratigraphic positions of the samples are summarized in Figure 2. A general lack of floral and/or faunal remains renders many of the biostratigraphic assignments highly speculative. The present assignments are based on the dinoflagellate and pollen stratigraphy of Habib and Drugg (this volume). Each sample was oven-dried at 60°C and ground to a diameter of approximately 44 μ m. Organic carbon and total sulfur content

van Hinte, J. E., Wise, S. W., Jr., et al., *Init. Repts. DSDP*, 93: Washington (U.S. Govt. Printing Office).
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Figure 1. Location map of DSDP sites in the western North Atlantic.

were determined using a LECO combustion system after decarbonating. Those samples with greater than 0.5 wt.% organic carbon were analyzed by pyrolysis in a Rock-Eval II system as originally described by Espitalié et al. (1977). A portion of each sample was extracted using an azeotropic mixture of chloroform, acetone, and methanol. This was followed by the precipitation of asphaltenes with pentane and separation of the soluble component fractions by high performance liquid chromatography (HPLC). Kerogen was then isolated from the rock residue for subsequent elemental analysis (C, H, O, and N).

ANALYTICAL RESULTS

LECO Analysis (organic carbon and total sulfur)

The total organic carbon (TOC) content exhibited a wide range with significant stratigraphic variability, ranging from 0.10 wt.% in Sample 603B-16-2, 23-26 cm to 14.30 wt.% in Sample 603B-34-1, 118-120 cm (Table 1;



Figure 2. Stratigraphic and geochemical summary for DSDP Hole 603B.

Fig. 2). Seven samples contained in excess of 4.0 wt.% TOC, whereas only two samples contained less than 0.3 wt.% TOC, the mean for DSDP material (McIver, 1975). The richest samples were obtained from Turonian, Cenomanian, Aptian-Albian, and Barremian strata.

Total sulfur (S: mineral plus organic sulfur) content also displayed variability, although the range was much less compared to that of organic carbon (Table 1). Sample 603B-34-1, 118-120 cm, which exhibit the highest levels of organic enrichment, also displayed the highest level of sulfur enrichment, containing 4.19 wt.% S.

Pyrolytic Assay (Rock-Eval)

Another indicator of organic richness, more directly bearing on hydrocarbon source quality, is the total hydrocarbon generation potential $(S_1 + S_2)$: free plus generat-

Table 1. Summary of organic carbon, sulfur, and pyrolysis^a data, Hole 603B.

Core-Section (interval in cm)	Depth (m)	TOC (wt.%)	S (wt.%)	s ₁	S ₂	нı ^b	oıc		<i>T</i>
				(mg HC/g rock)		(mg HC/g rock)		TR	(°C)
16-2, 23-26	968.14	0.10	0.11						
26-3, 43-47	1060.05	0.26	0.14						
29-1, 79-84	1081.61	2.68	0.34	0.14	0.19	7	28	ins.d	ins.
33-2, 30-33	1120.41	4.22	1.31	0.40	21.66	513	27	0.02	414
33-3, 130-133	1123.11	4.76	0.69	0.73	34.61	727	27	0.02	411
34-1, 30-33	1128.69	8.39	1.79	1.33	65.17	777	26	0.02	409
34-1, 118-120	1129.57	14.30	4.19	3.32	93.14	651	22	0.03	405
34-2, 134-136	1130.35	6.28	2.18	1.18	32.68	520	18	0.03	408
35-2, 7-10	1138.08	0.91	0.55	0.08	0.00	0	73	ins.	ins.
36-3, 52-54	1149.03	2.15	1.74	0.20	2.26	105	34	0.08	423
37-5, 33-35	1160.84	3.11	2.29	0.28	4.84	156	23	0.05	424
42-2, 47-50	1199.18	1.67	0.22	0.09	0.57	34	47	ins.	ins.
42-3, 46-49	1200.67	3.06	0.09	0.12	2.86	94	31	0.04	419
42-4, 16-18	1201.87	2.96	0.60	0.19	2.50	85	25	0.07	428
44-1, 46-48	1214.87	4.34	1.39	0.31	9.53	220	35	0.03	427
49-2, 106-109	1264.98	4.15	0.76	0.31	6.94	167	25	0.04	425
49-5, 62-65	1269.03	1.91	1.11	0.13	0.84	44	114	ins.	ins.
54-5, 45-47	1316.86	1.70	0.34	0.09	0.73	43	76	ins.	ins.
55-1, 50-53	1320.50	1.57	0.45	0.13	0.79	50	115	ins.	ins.
64-3, 119-121	1409.89	1.64	0.27	0.13	0.42	26	93	ins.	ins.
66-2, 73-75	1425.94	2.31	0.69	0.14	0.98	42	46	ins.	ins.
67-3, 49-51	1436.30	1.85	0.46	0.23	0.69	37	50	ins.	ins.
67-5, 58-61	1439.28	1.66	1.03	0.09	0.65	39	68	ins.	ins.

^a Pyrolysis limited to samples with greater than 0.5 wt.% TOC; blanks represent no analysis.

^b Hydrogen index.

C Oxygen index.

d ins. = insufficient yields for reliable calculation.

able hydrocarbons) as determined by pyrolysis. This is a direct measure of the rock's capacity to generate hydrocarbons, accounting for the quantity and quality of the organic matter and the effects, if any, of mineral matrix. The measured generation potentials range from 0.08 to 96.46 mg HC/g rock (Table 1; Fig. 2). Good potential or effective hydrocarbon source rocks, depending on the level of organic maturity, display yields greater than 6 mg HC/g rock (Tissot and Welte, 1978). Seven samples have yields in excess of this value.

In addition to the measurement of generative capacity, pyrolysis provides indications of both organic character and thermal maturity. Espitalié et al. (1977) proposed that the ratio of kerogen-derived hydrocarbons to organic carbon [hydrogen index (HI): S₂/TOC] and the ratio of kerogen-derived CO2 (S3) relative to organic carbon [oxygen index (OI): S₃/TOC] are comparable to the atomic H/C and O/C ratios, respectively. The relationships between these two indexes (Fig. 3) and between the hydrogen index and T_{max} (temperature of peak pyrolytic generation of hydrocarbons; Fig. 4) suggest the presence of at least three populations. The first population is composed of samples from Cores 33 and 34 (Turonian and Cenomanian strata). The data obtained from these samples indicate that the organic matter is largely type II, exhibiting elevated hydrogen indexes (>300; see Figs. 3, 4). The second population rests in close proximity to the type III reference curve. The third population plots below the type III reference curve and is composed of material that is classified as type IV or residual organic matter. Although the Rock-Eval has limitations in organic matter characterization (Katz, 1983), most of these complications are associated with leaner rocks. In a data set where the TOC is typically greater than 1.5 wt.%, general characterizations are normally valid.



Figure 3. Modified van Krevelen diagram, displaying the relationship between hydrogen and oxygen indexes obtained on samples from Hole 603B.





Figure 4. Characterization of organic matter utilizing the hydrogen index and T_{max} values obtained on samples from Hole 603B. (R₀ is vitrinite reflectance equivalence, in percent.)

With regard to organic thermal maturation, pyrolysis may provide two indicators— T_{max} and the transformation ratio (ratio of free hydrocarbons to total hydrocarbons: $S_1/S_1 + S_2$). The T_{max} values, which are all less than 435°C (Table 1; Fig. 2), and the transformation ratios (TR), which are all less than 0.1 (Table 1), indicate that the organic matter is thermally immature (Espitalié et al., 1977) and has not entered into the principal stage of thermogenic hydrocarbon generation and expulsion. The significance of these two indicators is determined in the case of T_{max} by the presence of a well-defined S_2 peak (Fig. 5) and in the case of TR by a $S_1 + S_2$ value greater than 1.0, a level well above background noise.

BITUMEN CHARACTERIZATION

Bitumen yields ranged from 49 ppm, a background level, to 9018 ppm total extract (TOE, Table 2). These



Figure 5. A. Pyrogram from Sample 603B-34-2, 134-135 cm, displaying a well-defined S₂ peak. B. Pyrogram from Sample 603B-42-2, 47-50 cm, displaying a poorly defined S₂ peak.

Table 2. Summary of gravimetric analyses, Hole 603B.

Core-Section (interval in cm)	TOE ^a (ppm)	P-N ^b (%)	Arom. ^c (%)	Resin (%)	Asph. ^d (%)	TOE/TOC ^e (%)
16-2, 23-26	49	ins.f	ins.	ins.	ins.	4.9
26-3, 43-47	173	3.4	25.8	18.0	52.8	6.7
29-1, 79-84	184	4.0	18.4	26.4	51.2	0.7
33-2, 30-33	1823	1.9	1.3	23.9	72.9	4.3
33-3, 130-133	2867	1.2	2.4	16.7	79.7	6.0
34-1, 30-33	4736	1.7	3.4	21.4	73.5	5.6
34-1, 118-120	9018	1.6	5.3	27.6	65.5	6.3
34-2, 134-136	4479	1.9	6.9	19.5	71.7	7.1
35-2, 7-10	169	5.9	5.9	17.7	70.5	1.9
36-3, 52-54	612	1.2	13.6	23.1	62.1	2.8
37-5, 33-35	1005	2.1	9.8	25.8	62.3	3.2
42-2, 47-50	245	4.6	4.6	18.2	72.6	1.4
42-3, 46-49	382	5.6	5.6	19.4	69.4	1.2
42-4, 16-18	386	2.6	8.7	27.0	61.7	1.3
44-1, 46-48	1402	2.5	4.2	26.7	66.6	3.2
49-2, 106-109	1272	2.6	4.3	27.3	65.9	3.1
49-5, 62-65	552	4.4	2.2	22.2	71.2	2.9
54-5, 45-47	371	5.5	5.5	23.3	65.7	2.2
55-1, 40-53	319	3.3	3.3	23.3	70.1	2.0
64-3, 119-121	297	3.6	3.6	17.9	74.9	1.8
66-2, 73-75	437	4.1	5.5	23.3	67.1	1.9
67-3, 49-51	390	5.3	6.3	28.4	60.0	2.1
67-5, 58-61	313	10.3	6.9	17.2	65.6	1.9

^aTotal organic extract.

^b Paraffin-naphthenes.

d Aromatics.

d Asphaltenes

e (Total organic extract/Total organic carbon) × 100.

ins. = insufficient extract yield for reliable determination.

yields are consistent with the pyrolytic assay in both quantity and general character (i.e., low levels of free hydrocarbons). Bitumen yields in excess of 1000 ppm, the lower cutoff for good potential source rocks (GeoChem Laboratories, Inc., 1980), were restricted to eight samples: this group of samples includes all seven samples identified by pyrolysis as potential source rocks.

The extracted material was largely nonhydrocarbon (resins and asphaltenes) in nature. Total hydrocarbons (paraffins, naphthenes, and aromatics) account for less than 30% and commonly represent less than 10% of the total extract (Fig. 6). TOE/TOC ratios are all less than 0.1 and suggest that the bitumen is indigenous (i.e., there is no evidence for staining, micro-reservoiring, and/ or contamination). The lack of hydrocarbons as a significant component of the extract is, in part, a result of the low levels of thermal maturity. It is also a result of an initial hydrogen depletion of the precursor material (i.e., terrestrial and/or recycled material). Thermal immaturity is also indicated by the low nonaromatic to aromatic hydrocarbon ratios that are typically less than 1.0 (Le Tran et al., 1974).

Elemental Analysis of Kerogen

The elemental data, summarized in Table 3 and Figure 7, confirm the initial interpretations suggested by pyrolysis. The bulk of the organic matter appears to be either type III or IV. In fact, a significant number of samples have H/C ratios less than 0.8. Such values reveal the importance of recycled and/or strongly oxidized material as contributors of organic carbon during the Cretaceous. The elevated levels of oxidation may have been obtained through low-temperature subaerial weathering, biological oxidation (Tissot and Welte, 1979), or forest fires (Pocock, 1982).

These data also aid in explaining the poorly defined S_2 peak and consequently the inability to establish a T_{max} value on many samples. In this study, only organic matter isolated from Cores 33 and 34 appears closely aligned with the type II reference curve, indicating the preservation of material with a marine origin.



Figure 6. Triangular diagram displaying the gross composition of extracted bitumens, Hole 603B.

Table 3. Elemental composition, Hole 603B.

	Ash ^a	H ^a	C ^a	O ^a	N ^a	Atomic	
Core-Section (interval in cm)						H/C (ash-fr	O/C ee basis)
26-3, 43-47	0.98	2.05	35.63	8.57	1.79	0.69	0.181
29-1, 79-84	11.87	2.97	61.83	15.50	1.84	0.57	0.188
33-2, 30-33	1.83	6.25	66.72	12.22	2.36	1.12	0.137
33-3, 130-133	0.30	6.85	67.97	12.34	2.08	1.20	0.136
34-1, 30-33	2.81	6.41	64.80	11.11	2.00	1.18	0.129
34-1, 118-120	7.89	6.43	60.61	11.37	2.53	1.26	0.141
34-2, 134-136	1.41	6.62	64.88	11.53	2.99	1.22	0.133
35-2, 7-10	20.88	3.10	53.69	9.43	1.32	0.69	0.132
36-3, 52-54	15.16	3.92	47.67	10.52	2.62	0.98	0.166
37-5, 33-35	10.76	4.09	57.52	14.11	1.80	0.85	0.184
42-2, 47-50	9.26	4.11	65.56	13.35	1.77	0.75	0.153
42-3, 46-49	3.50	4.62	68.39	13.05	2.23	0.80	0.143
42-4, 16-18	3.35	4.68	68.00	14.99	2.77	0.82	0.165
44-1, 46-48	3.18	4.98	66.17	13.28	2.44	0.90	0.151
49-2, 106-109	4.92	4.94	63.35	11.75	2.26	0.93	0.139
49-5, 62-65	29.40	2.71	38.02	10.99	1.08	0.85	0.217
54-5, 45-47	6.57	4.06	64.50	12.48	1.89	0.75	0.145
55-1, 50-53	6.16	4.07	62.44	14.18	1.50	0.78	0.170
64-3, 119-121	3.90	4.25	66.34	14.17	1.65	0.76	0.160
66-2, 73-75	13.14	3.92	50.87	11.91	1.83	0.92	0.176
67-3, 49-51	3.46	3.60	50.65	14.23	1.43	0.85	0.211
67-5, 58-61	23.35	2.86	44.60	11.62	1.07	0.76	0.196

a Values in wt.%.



Figure 7. Van Krevelen diagram, displaying the relationship between atomic H/C and O/C ratios, Hole 603B

DISCUSSION

The data described here and in Meyers (this volume) clearly reveal the presence of immature oil-prone material of source quality within the Turonian and Cenomanian section. As a consequence of measured generative capacity and cumulative thickness of black shales within these two cores (>10 net m), these sediments, if thermally mature, could yield significant volumes of petroleum. In this interval, black shales occur only as thin interbeds in Core 33, but they represent the dominant lithology in Core 34. However, the lack of well control in a region where these sediments may be more thermally mature (i.e., further landward) suggests that their economic value on the U.S. Atlantic margin should be viewed cautiously.

The other samples in this study that exhibited sourcerock qualities (i.e., elevated generation potentials and extract yields) were within the middle Aptian–Albian and Barremian sections. This material was immature and gasprone. The remaining strata examined, although typically organically enriched, contained principally refractory carbon and are incapable of generating and expelling any significant volumes of hydrocarbons.

In addition to source-rock quality, these data provide information on the uniformity and character of depositional environment associated with the black shales. The variability of these shales as manifested by both the organic carbon and hydrogen content (Fig. 2) implies nonuniformity within depositional systems. At Site 603 it appears that during limited periods of the Turonian and the Cenomanian, conditions were favorable for the production and preservation of marine organic matter. Samples from other stratigraphic units suggest that conditions were less favorable. During these other periods, however, conditions were such that significant quantities of terrestrial material (up to 4.34 wt.% type III material, and up to 2.68 wt.% residual organic matter) were available. (Minor influxes of well-preserved marine organic matter have been noted elsewhere in the stratigraphic column by Meyers, this volume.)

Preservation of terrestrial organic matter is less sensitive to environmental conditions than is marine material, as a consequence of continental material having typically been partially oxidized and degraded during transport. These processes result in the loss of the more labile components prior to final deposition. Terrestrial organic matter is also less reactive than marine organic material because of its lower surface area-to-volume ratio (Waples, 1983).

There is no evidence that final deposition of the organic matter in Cores 33 and 34 occurred in a euxinic setting. The ratios of organic carbon to sulfur (Fig. 8), although not conclusive, indicate the presence of at least minor amounts of oxygen and an absence of hydrogen sulfide in the bottom waters during time of deposition. This inference is based on the apparent lack of a background sulfur signal of between 1 and 2 wt.% as suggested by Leventhal (1983) for sediments deposited under euxinic conditions. This sulfur signal is caused by the addition of reduced sulfur from the overlying water column (Sweeney and Kaplan, 1980). Such a background



Figure 8. Relationship between total organic carbon and sulfur, Hole 603B. The solid line represents the lower limits for a field representing sediments deposited under euxinic conditions in the Black Sea (Leventhal, 1983).

signal has been observed in the modern Black Sea, the Devonian black shales of the eastern United States (Leventhal, 1979), and the Cretaceous Pierre Shale of the United States mid-continent (Gautier et al., 1984).

Furthermore, organic geochemical data from the Aptian-Albian of Site 105 to the east (Tissot et al., 1979; Summerhayes, 1981; Katz and Pheifer, 1982) and the Cenomanian and Aptian-Albian of Sites 391 and 534 to the south (Herbin et al., 1984; Katz, 1984; Fig. 1) indicate a terrestrial origin for much of the organic matter. This suggests that the conditions associated with the dominance of marine material at Site 603 were areally limited and not oceanic in extent. However, there is some evidence from Site 105 (Katz and Pheifer, 1982) that during the Cenomanian, deposition of black shales dominated by marine organic matter extended farther seaward than during the Aptian-Albian.

Consequently, the black shales at Site 603, including those rich in type II kerogen, do not represent oceanic anoxic events as discussed by Schlanger and Jenkyns (1976) and Tissot et al. (1980), but more likely represent local conditions superimposed on a generally more favorable global pattern. At Site 630, sedimentologic data (Site 603 chapter, this volume) suggest that the black shales were emplaced by turbidity currents. The black shale sequences are associated with quartz silt maxima and are included in fining-upward sequences, both of which are consistent with such a mechanism of emplacement. Transport by turbidity currents has been proposed for other Atlantic Ocean Cretaceous black shales at several locations (e.g., Cornford, 1979; Summerhayes, 1981; Graciansky et al., 1982; Dean et al., 1984; Meyers et al., 1984). Rapid sedimentation, as associated with turbidity currents, would also be consistent with the general lack of evidence for massive bioturbation.

There appear to be two primary up-slope sources for the carbonaceous material. Coals and/or coaly sediments originally deposited on the continental shelf during regressions, as observed in the Baltimore Canyon (Scholle et al., 1978), would be a source for those shales rich in type III and residual organic matter. Large volumes of marine material would be available during transgressive periods when marine productivity increases, in part, as a result of an areal expansion of the continental shelves and consequently the photic zone (Tissot, 1979). Increased productivity causes, as a consequence of feedback mechanisms, an intensification and expansion of the oxygenminimum zone (Schlanger and Jenkyns, 1976). Large quantities of type II organic matter are originally deposited and preserved where this zone impinges on the shelf and slope. Instability in these organic-matter-rich sediments upslope results, in part, from biogenic gas production. This instability ultimately contributes to the initiation of slumping and hence the final deposition of the type II rich black shales in the deep basin.

SUMMARY

1. The sediments sampled within Cores 33 and 34 of Hole 603B represent a thermally immature potential oil source unit capable of generating significant volumes of liquid hydrocarbons. 2. Other black shales, although commonly rich in organic carbon, are either gas-prone or incapable of generating hydrocarbons because the organic matter is refractory.

3. The data do not support a stagnant model, but are consistent with a suboxic to oxic depositional setting.

4. The black shales, including those rich in type II kerogen, appear to be the result of turbidity current transport from the outer shelf and upper slope.

ACKNOWLEDGMENTS

The author wishes to thank the Organic Geochemistry Laboratory of Texaco Inc. for analytical support, D. J. Schunk for valuable discussions concerning the original draft manuscript, and Drs. M. A. Authur, D. A. Dunn, and R. N. Pheifer for the critical reviews and suggestions. The author acknowledges the permission of Texaco Inc. to publish this chapter. Texaco Contribution No. 2911.

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Date of Initial Receipt: 18 January 1985 Date of acceptance: 11 July 1985