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Serravallian shales in the Monte dei Corvi pelagic sequence (Ancona, Italy): an organic geochemical perspective

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Abstract

In addition to the predominant marly lithologies, the Serravallian-Tortonian sequence at Monte dei Corvi (MDC), south of Ancona, Italy, contains at least 85 thin, dark calcareous shales. Sixteen of the Serravallian shales were sampled for a preliminary organic geochemical evaluation. All the MDC shales appear to have been deposited during periodic anoxic events, as demonstrated by the presence of significant quantities of organic matter and authigenic pyrite. The degree of anoxicity (and thus the amount of organic matter preserved) appears to have differed from one event to the next. While the quantity of organic matter preserved is highly variable, the type is remarkably consistent from sample to sample, as evidenced both by analytical pyrolysis-gas chromatography/mass spectrometry (GC/MS) of the solid organic matter and by the GC/MS analysis of the extractable material. This implies a regeneration of similar microflora/microbial assemblages and depositional conditions during each anoxic event. The dominant organic matter types are marine, including several types of fossil algae and amorphous material (largely the product of bacterial reworking of organic matter). There is evidence of minor terrestrial input. The presence of isoprenoid hydrocarbons (prist-1-ene) in the pyrolyzates and 17 β (H), 21 β (H) hopanes in the extractable organic matter attest to the low level of thermal alteration of the MDC shales. The MDC organic matter appears to be partly oxidized, probably from weathering at the outcrop. It is recommended that fresh, unweathered samples be obtained prior to further organic geochemical studies, preferably by coring.

KEY WORDS: kerogen pyrolysis, Miocene black shales, Apennines, Italy.

Introduction

Approximately 90 m of Serravallian-Tortonian pelagic carbonate rocks of the Schlier Formation are exposed in the Monte dei Corvi (MDC) sequence, in the east-central Apennines, along the coast south of Ancona, Italy. The sequence is remarkable for the rhythmic intercalations of hard calcareous marls with softer marls, with each couple averaging about 0.5 m in thickness throughout much of the section. This marl sequence is punctuated by at least 85 thin, dark calcareous shales (Fig 1). The shale thicknesses vary from 1 to 40 cm, averaging 13 cm. The amount and type of organic matter in these shales are of interest, as they may offer insights into the nature of the periodic anoxic events during the shale deposition. The Serravallian portion of the sequence was sampled at the outcrop for a preliminary organic geochemical examination.

Methods

During the summer of 1991, 16 of the Serravallian dark shales, in the interval between 8 and 54 m above the base of the section, were collected for analysis (Fig. 1, Tab. 1). In addition, the strata between 32.7 and 35.2 m were sampled in detail (4 m⁻¹), so that the intervening marly lithologies could also be evaluated. The samples are from exposures in a steep cliff at the level of the beach, at a site of active erosion. The shales at the surface of the exposure had a bleached appearance, the result of oxidation. Therefore, the samples were collected from a depth of several centimeters, where the shales were darker and apparently less oxidized.

The rock samples were crushed and divided into four aliquots. One aliquot was submitted for pyrolysis assay using the Rock Eval technique (Tissot and Welte, 1984). A

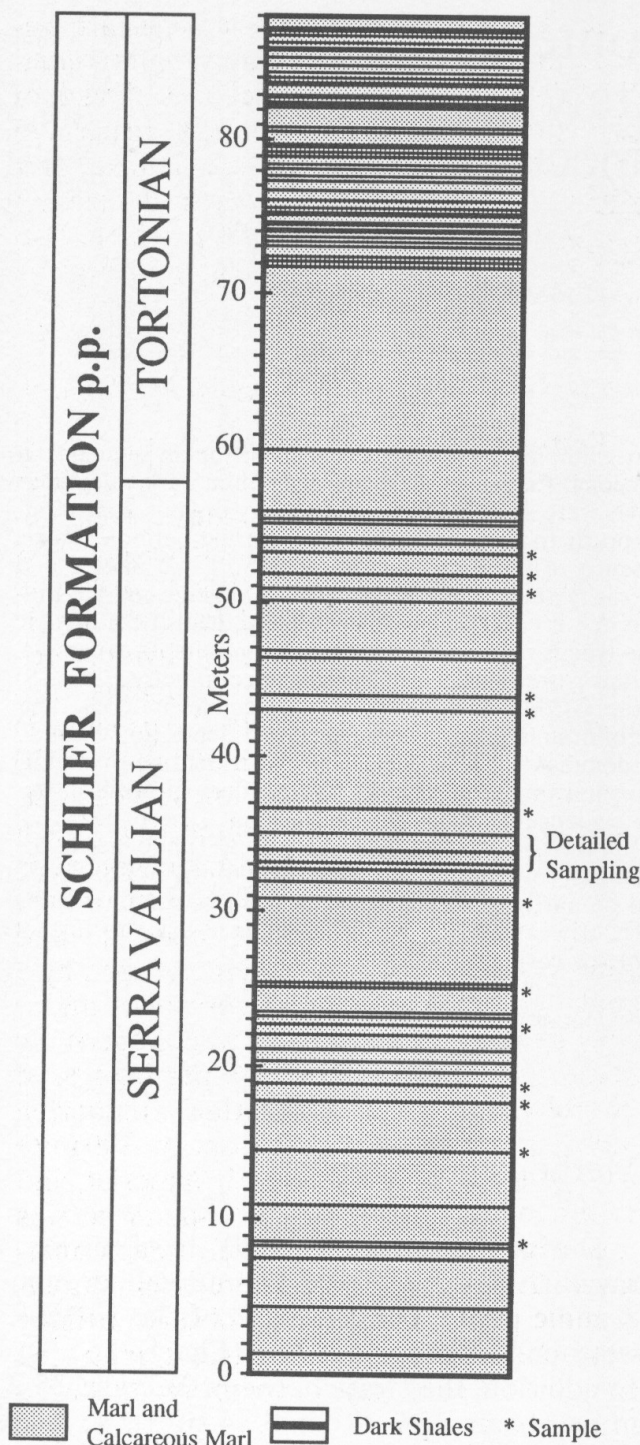


Fig. 1 - Simplified stratigraphic section of the Monte dei Corvi sequence emphasizing the occurrence of dark, calcareous shales. Measurements in meters are relative to the base of the section (from Coccioni *et al.*, 1992) The * indicates points at which geochemical samples were collected.

second aliquot (samples between 33.00 and 35.19 m only) was analyzed for carbonate content by weighing before and after treatment with HCl. A third aliquot (of the 3 samples richest in organic matter, as deter-

mined by Rock Eval, Tab. 1) was mounted in epoxy pellets and examined petrographically in reflected white and blue light.

A fourth aliquot (from the 7 richest samples, Tab. 1) was extracted with dichloromethane and the residues subjected to analytical pyrolysis-gas chromatography (py-GC-FID). Pyrolysis was at 610° C for 20 sec., using a CDS 120 Pyroprobe coupled to an HP 5890A GC, equipped with a flame ionization detector, which is sensitive to hydrocarbons.

Chromatography was performed on a 25 m OV-1 column (0.2 mm i.d., 0.33 μm film thickness), initially held at -20° C for 1 min., then raised to 300° at 5°/min., then held for 15 min. Two rich samples were also analyzed by pyrolysis-gas chromatography/mass spectrometry (py-GC/MS), which facilitates identification of the pyrolysis products, non-hydrocarbons in particular (Sinningh Damsté *et al.*, 1989, 1992; Eglinton *et al.*, 1992). Pyrolysis was again at 610° C for 20 sec., using a CDS 120 Pyroprobe coupled to an HP 5890A GC and an HP 5970B Mass Selective Detector. The GC was equipped with the same type of column as used for py-GC-FID, but initially held at 0° C for 5 min., then raised to 300° at 5°/min., then

Tab. 1 - List of MDC samples with Rock Eval pyrolysis assay results and carbonate contents. * Samples designated by stratigraphic position (meters above base of section).

a, py-GC-FID; b, py-GC/MS; c, GC/MS of whole extract; d, organic petrology.

Sample*	Lithology	S1 (mg/g)	S2 (mg/g)	CaCO ₃ (wt. %)	Other Analyses
8.72	dark black calc. shale	0.30	6.96	—	
14.60	black calc. shale	0.18	4.85	—	
17.70	brown calc. shale	0.15	3.27	—	
18.83	brown calc. shale	0.28	6.24	—	
22.64	brown calc. shale	1.43	27.37	—	a, b, c
25.04	brown calc. shale	0.34	7.87	—	
30.88	brown calc. shale	1.43	26.77	—	a, c
32.78	brown calc. shale	0.43	11.24	77	c
33.00	marl	0.02	0.12	77	
33.15	marl	0.02	0.09	81	
33.27	brown calc. shale	0.47	3.60	67	
33.50	marl	0.03	0.14	85	
33.75	marl	0.02	0.16	82	
33.98	brown calc. shale	1.38	27.60	59	a, b
34.25	marl	0.02	0.13	78	
34.50	marl	0.03	0.10	77	
34.75	marl	0.03	0.08	83	
35.19	brown calc. shale	0.62	12.94	58	a
36.70	brown calc. shale	0.36	8.96	—	a
43.10	brown calc. shale	0.05	0.44	—	
44.10	brown calc. shale	0.27	5.30	—	
50.99	brown calc. shale	0.45	11.61	—	a
51.91	brown calc. shale	0.25	6.54	—	
53.31	brown calc. shale	0.19	5.15	—	

held for 15 min. The mass spectrometer was in full scan mode with an ionizing voltage of 70 eV. The extracts of 3 of the samples (Tab. 1) were submitted for gas chromatography/mass spectrometry (GC/MS) using the same GC conditions as above, except that the oven was held at 100° C for 10 min., programmed from 100 to 300° C at 3°/min., then held at 300° C for 21 min. The mass spectrometer, using an ionization energy of 70 eV, was run in selective monitoring mode, collecting data on the diagnostic fragment ions of normal alkanes (m/z 99), steranes (m/z 217, 218, 231, 259) and hopanes (m/z 191).

For purposes of comparison with MDC samples, several samples of classic kerogen types were analyzed by py-GC/MS, after pre-extraction with dichloromethane. These included kerogens from the Eocene Green River shale («Mahogany Zone», Piceance Basin, Colorado, U.S.A.), Toarcian shale of the Paris Basin, Miocene Monterey Formation (Naples Beach, Santa Barbara County, California, U.S.A.) and demineralized coals of the Westphalian Herrin No. 6 Seam (Illinois Basin Coal Sample Project sample 101, Illinois, U.S.A.) and from the Eocene of Kalamantan, Indonesia.

Results and Discussion

Pyrolysis assay

The dark calcareous shales have moderate to high pyrolyzable kerogen contents, with Rock Eval S_2 ranging from 3.3 to 27.6 mg/g (Tab. 1). The three richest samples in the suite ($S_2 \sim 27$ mg/g) occur between 22 and 35 m and, unlike the other shales, show little or no evidence of bioturbation. The marls have only negligible amounts of kerogen (<0.2 mg/g). For the closely-sampled segment between 32.7 and 35.2 m, kerogen contents increase with decreasing $CaCO_3$, such that only rocks with less than 70% carbonate have significant quantities of kerogen (Tab. 1). Amounts of thermally-extractable bitumen (Rock Eval S_1) in the shales are surprisingly low, even for the samples richest in S_2 , ranging from 0.15 to only 1.43 mg/g. Solvent extractable bitumen contents are even lower (<0.1 mg/g). Both S_1 and S_2 values correlate inversely with $CaCO_3$ con-

tent, indicating that the black shales are significantly enriched in organic matter compared to the marls, as expected. The lack of biodegradation in the samples richest in kerogen indicates that severe anoxia prevailed during the time of their deposition (Demaison and Moore, 1980), which also significantly enhanced the degree of organic matter preservation.

Kerogen characterization by organic petrology

Petrographic examination of the three shales richest in organic matter (22.64, 30.88 and 33.98 m) reveals that marine algal remains are the dominant organic matter. This "alginite" is yellow-fluorescing under blue light and represents at least two taxonomic groups: 1) types preserved as individual bodies ("telalginite") and their fragments, ranging roughly in size from 10 to 100 μm and 2) banded agglomerations ("lamalginite") ~ 30 μm in width. The yellow fluorescence indicates a low level of thermal alteration (Stach *et al.*, 1982). Amorphous organic matter is also present, most likely the product of bacterial degradation of algal material and other organic debris. It is characterized by a weak greenish-brown fluorescence under blue light, a feature possibly enhanced by weathering. The rare vitrinite particles have oxidized rims, acquired during weathering and/or pre-depositional transport. Vitrinite is diagenetically-altered woody material, and its low abundance indicates that there was only minor input of terrestrial organic matter to the MDC shales. There is ample authigenic pyrite, occasionally observed filling foraminiferal cavities, consistent with the existence of a reducing environment during and after sedimentation.

Kerogen characterization by analytical pyrolysis-gas chromatography

The seven samples with the greatest pyrolyzable kerogen contents ($S_2 \geq 8$ mg/g) were subjected to py-GC>FID. The resulting chromatograms were all very similar, dominated by hydrocarbon gases (C_1 to C_4) and simple aromatic compounds, including benzene, toluene and xylenes. The exception is the sample at 32.78 m, which showed a high concentration of high molecular weight normal alkanes, indicating an incomplete

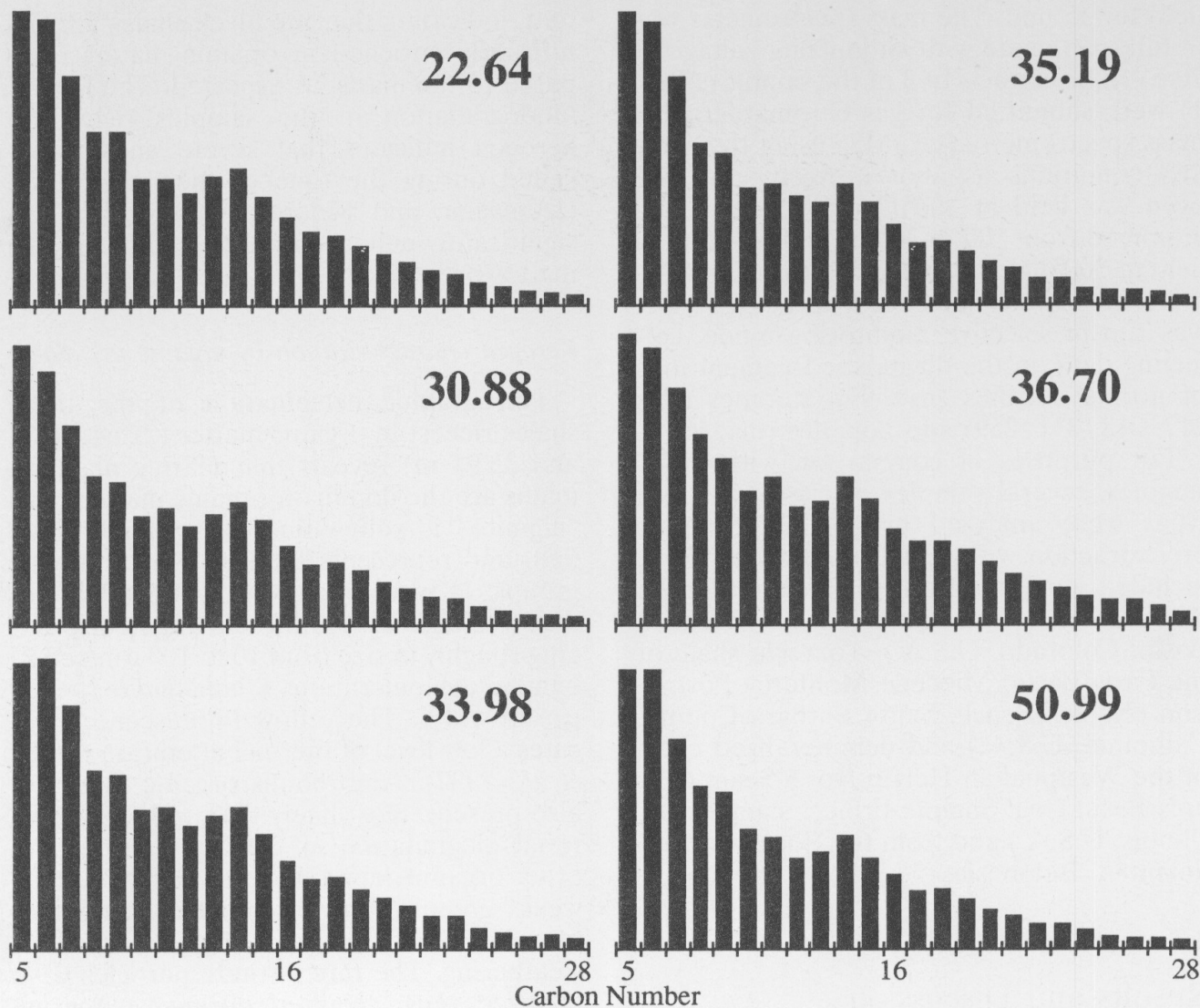


Fig. 2 - Distributions of aliphatic hydrocarbons (*n*-alkanes + *n*-alkenes) by carbon number for the six richest MDC samples from quantitation of py-GC-FID data. Samples are identified by their position in meters above the base of the section. See Fig. 1 for stratigraphic context.

extraction during sample preparation. Because of this analytical problem, the pyrolysis results for this sample are not considered further. Other important compounds include the two series of long chain aliphatic hydrocarbons (normal alkanes and alk-1-enes) ranging from C₅ to at least C₂₈, the branched-chain hydrocarbon prist-1-ene and several phenolic compounds. The distributions of normal hydrocarbons are very similar in all 6 samples (Fig. 2), regardless of the amount of pyrolyzable kerogen present. There is a gradual decrease in the relative amount of each normal alkane/alkene pair as chain length increases from 5 to 28, with an apparent minor increase at C₁₄, consis-

tent for all samples. It is evident that there is a very similar type of organic matter present in each shale sample analyzed.

Two of the samples (at 30.88 and 33.98 m) were also analyzed by py-GC/MS, which permitted a definitive identification of compounds in the pyrolyzate. The two pyrolyzates are seen again to be very similar to each other. The same characteristics noted in the py-GC-FID analyses are again apparent using py-GC/MS, except that the lighter hydrocarbons (<C₆) are not detected, due to the experimental conditions employed (Fig. 3). However, additional compounds are recognized, including hydrocarbons such as cyclopentadienes, indenes, and naphtha-

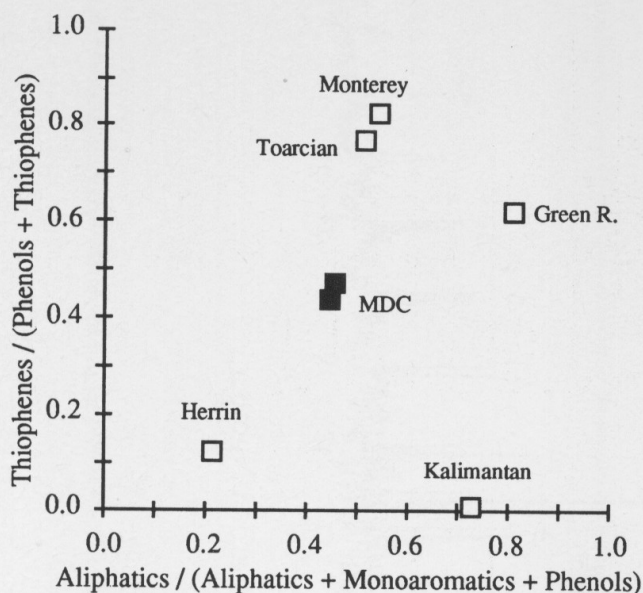


Fig. 4 - Cross-plot comparing MDC pyrolyzates (py-GC/MS data of the samples at 22.64 and 33.98 m) with those of samples representing the classic kerogen types, including the Green River Shale (Type I, lacustrine), the Toarcian of the Paris Basin and the Monterey Formation (Type II, marine), and the Herrin No. 6 and Kalimantan coals (Type III, terrestrial). The parameter on the abscissa is an index of aliphaticity and that on the ordinate indicates the relative degree of incorporation of sulfur (thiophenic) or oxygen (phenolic). See text for further discussion.

lenes. Thiophenes and pyrroles, which are sulfur- and nitrogen-containing compounds, are also detected. The prominence of prist-1-ene indicates that the samples have not experienced much thermal alteration during burial and are thermally immature with respect to petroleum generation (Larter, 1984).

Overall, the pyrolysis-GC data indicate that the samples contain Type II organic matter, i.e., they show a predominance of monoaromatic (benzene series) compounds, with secondary normal hydrocarbons and thiophenes. This is illustrated more clearly if the MDC shales are placed in context with examples representing all the classic kerogen types (Tissot and Welte, 1984). The samples may be conveniently compared using two parameters derived from py-GC/MS data: a) an index of the content of aliphatic hydrocarbons, relative to the amount of monoaromatic and phenolic compounds and b) an ratio of the thiophene and phenol content, which exploits the tendency for marine-derived organic matter to be enriched in

sulfur and terrestrial to be oxygenated (Fig. 4). A lacustrine, algal-dominated (Type I) kerogen, such as the Eocene Green River Shale (Colorado, U.S.A.), shows a highly aliphatic character and little preference for either sulfur or oxygen-containing compounds. At the other extreme, the Westphalian Herrin No. 6 coal (Illinois, U.S.A.) is strongly phenolic, reflecting the predominance of vitrinite (Senftle and Larter, 1987), and is a classic example of Type III organic matter. The Eocene Kalimantan coal, typical of Tertiary coals from southeast Asia, represents another Type III variant, having both aliphatic and phenolic character, the former due in part to a high concentration of fossil resin. The classic Type II example, from the Toarcian of the Paris Basin, shows a moderate content of aliphatic material and a preference for sulfur over oxygen compounds. The Miocene Monterey Formation example (California, U.S.A.) represents a sulfur-enriched Type II variant and plots closely to the Paris Basin sample in Fig. 4, except that it shows a greater preference for thiophenes.

In Fig. 4, the two MDC samples plot closely together, which further confirms their similarity. They resemble the Type II examples with their moderate aliphatic content. However, their thiophene to phenol ratio is significantly lower, which suggests that either a) terrestrial organic matter (vitrinite or its degradation products) is a significant secondary component in the MDC kerogen, or b) the kerogen has been partially oxidized during exposure at the outcrop. As noted above, vitrinite is in fact seen in the MDC samples upon petrographic examination, but only as a rare component. However, there may also be smaller vitrinite fragments or bacterially-altered vitrinite, which would be difficult to detect with an optical microscope. Phenols may also possibly indicate oxidation, as they are essentially monoaromatic compounds with -OH functions attached (see structure diagram in Fig. 3). To eliminate the oxidation variable, the analyses should be repeated on fresh samples, preferably obtained by coring to a depth below any suspicion of weathering. The effects of clay catalysis during pyrolysis (Dembicki *et al.*, 1983) are likely to be minimal in the case of the MDC samples, due to their high carbonate content (59-70 % on those dark shales analyzed, Tab. 1). The character of

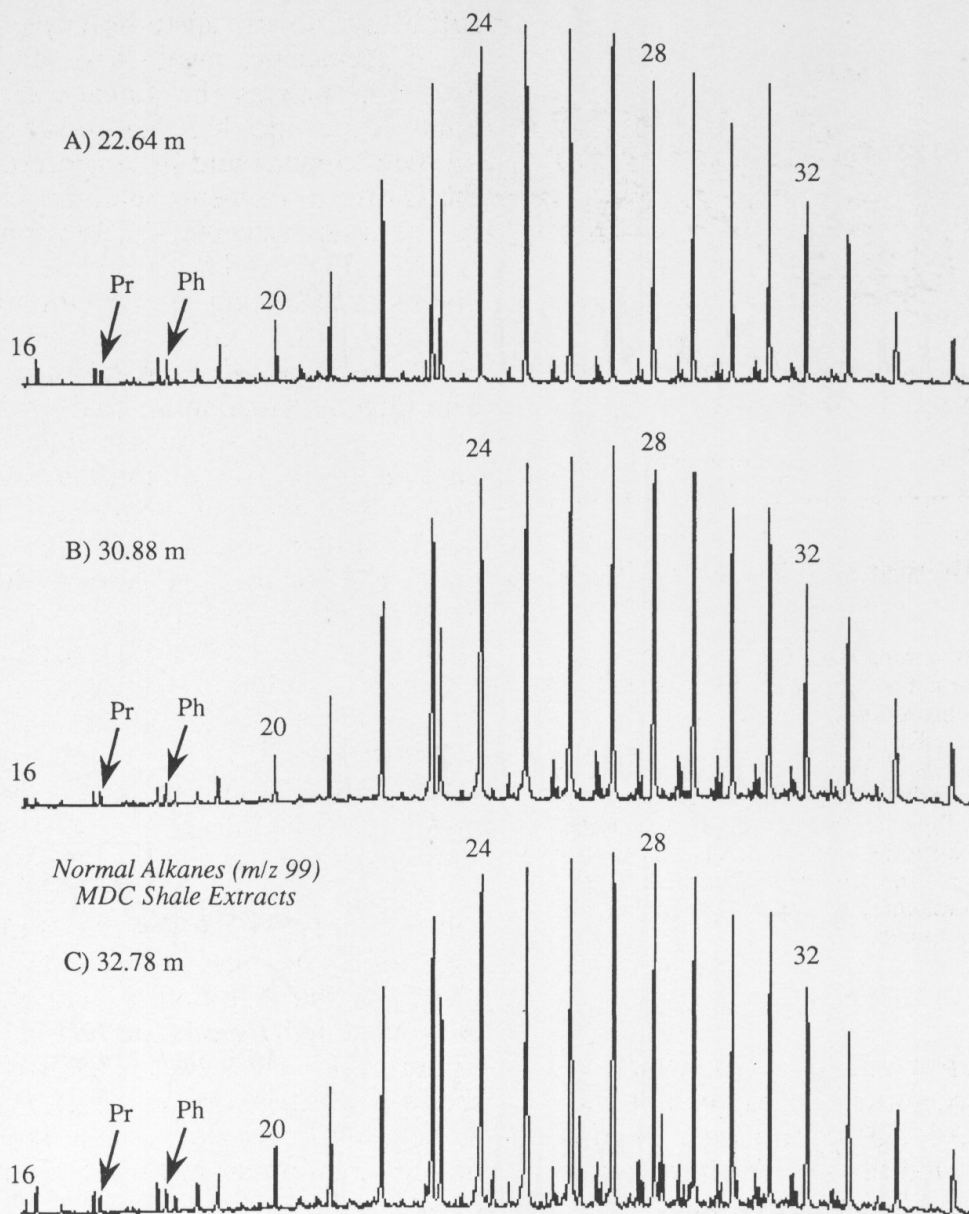


Fig. 5 - Three m/z 99 mass chromatograms showing the distribution of normal and isoprenoid alkanes in the solvent extracts of three MDC samples. n -Alkane carbon numbers are shown, as are the isoprenoids pristane (Pr) and phytane (Ph). Samples are identified by stratigraphic position (see Fig. 1) and data was collected on whole extracts.

the pyrolyzates should be confirmed by re-analysis of kerogen concentrates, an exercise outside the scope of this preliminary study.

The MDC pyrolyzates are also remarkable for the occurrence of a series of nitrogen-containing compounds, the C_1 to C_5 pyrroles (Fig. 3). Monterey kerogen pyrolyzates also contain abundant pyrroles (Sinninghe Damsté *et al.*, 1992). These compounds may possibly derive from degradation of chlorophyll or from the by-products of bacterial or fungal activity. In either case, the strong presence of pyrroles in both the MDC and

Monterey samples demonstrates an additional important chemical similarity between these two Miocene marine kerogens.

Extractable organic matter

Since the solvent-extractable bitumen yields were so low, it was not practical to isolate the saturate fraction by liquid chromatography, which would have been the preferred method for the examination of biological marker compounds or "molecular fossils" (Peters and Moldowan, 1993). In-

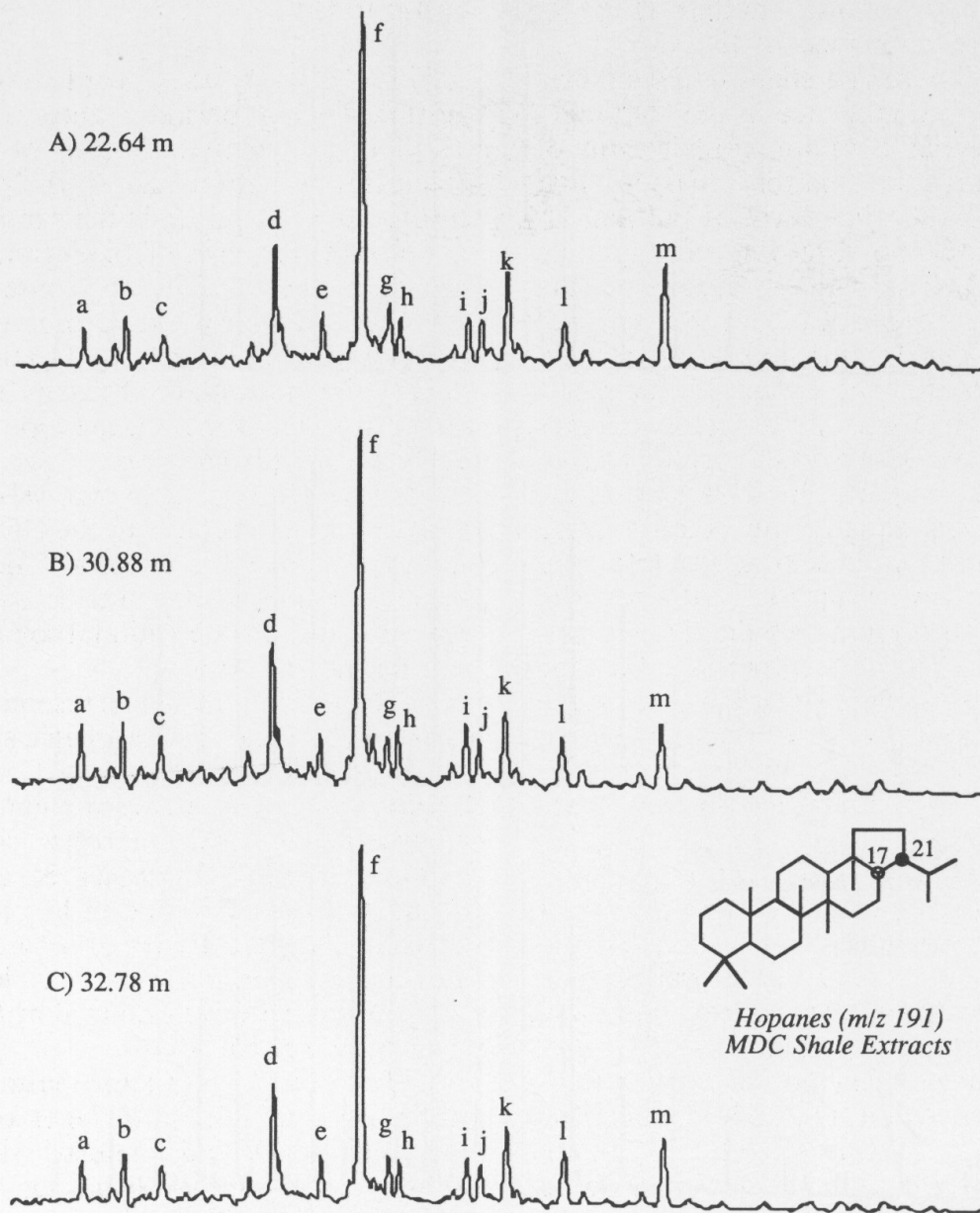


Fig. 6 - Three m/z 191 mass chromatograms showing the distribution of hopanes in solvent extracts of three MDC samples. Samples are identified by stratigraphic position (see Fig. 1) and data was collected on whole extracts. An example of a hopane structure (C_{30} $17\alpha(H)$, $21\beta(H)$) is shown with the C-17 and C-21 positions marked. The hopane peaks (a through n) are identified in the following table by carbon number (C_{27} and C_{29} to C_{32}) and by stereochemical configuration (α or β at the C-17, C-18 and C-21 positions), as described by Peters and Moldowan (1993).

	C_{27}	C_{29}	C_{30}	C_{31}	C_{32}
$18\alpha(H)$	a				
$17\alpha(H)$	b				
$17\beta(H)$	c				
$17\alpha(H)$, $21\beta(H)$		d	f	i,j	l,m
$17\beta(H)$, $21\alpha(H)$		e	h		
$17\beta(H)$, $21\beta(H)$		g	k	n	

stead, GC/MS analyses were performed on the whole extracts of three of the richest shales (22.64, 30.88 and 32.78 m), tuning the mass spectrometer to monitor the key fragment ions of common saturated hydrocar-

bons. This latter method is less sensitive, but should at least give an indication of the major compounds present. Mass chromatograms showing the distributions of normal alkanes demonstrate that all three samples

are very similar to one another (Fig. 5), showing a predominance of the C_{22} to C_{33} homologues. There is a slight odd/even carbon number predominance in the C_{27} to C_{33} range, suggesting a minor contribution of terrestrial organic matter (Tissot and Welte, 1984) and a low level of thermal alteration, consistent with the petrographic and pyrolysis data. Notably weak are the normal and isoprenoid alkanes in the C_{15} to C_{21} range, including pristane and phytane. It is apparent that these samples have suffered evaporative loss of the lighter components over and above that which is usual during sample preparation. This may be explained by weathering at the outcrop. Such natural exposure may also account for the anomalously low bitumen contents. The truncation of the shorter chain alkanes precludes a definitive interpretation, but the distribution of alkanes is not incompatible with an assemblage dominated by marine organic matter.

The hopane distributions in the three extracts are again all very similar (Fig. 6). The C_{30} $17\alpha(H)$, $21\beta(H)$ hopane predominates, while the C_{27} , C_{29} , C_{31} and C_{32} are of secondary importance. The extended hopanes ($>C_{32}$) are nearly indiscernible, unlike bitumens from many other carbonate sequences (Peters and Moldowan, 1993), but this may again be a function of the low thermal maturity. Secondary components also include the C_{29} to C_{31} $17\beta(H)$, $21\beta(H)$ hopanes, providing additional strong evidence for the low thermal maturity of the rocks. Hopanes are products of the activity of prokaryotic bacteria (Ounsson *et al.*, 1984). Their strong presence in the extract indicates that such anaerobes contributed significantly to the mix of organic matter in the MDC samples and provides further support for a paleoenvironmental interpretation evoking low-oxygen conditions. Steranes are relatively weak in the MDC samples, perhaps due to the nature of the original organic material or perhaps due to weathering at the outcrop.

Conclusions

1) All MDC shales appear to have been deposited during periodic anoxic events, as testified by the preservation of significant quantities of organic matter, the presence of authigenic pyrite, and, in the samples richest in kerogen, the lack of biodegradation. The strength of the anoxicity, and consequently the amount of organic matter preserved, appears to have varied from one event to the next.

2) While the amount of organic matter preserved is highly variable, the type is remarkably consistent from sample to sample, as evidenced both by analytical pyrolysis-GC of the solid organic matter and by the GC/MS analysis of the extractable material. This implies a regeneration of similar microfloral/microbial assemblages and depositional conditions during each anoxic event.

3) The MDC pyrolyzates resemble those of classic Type II marine kerogens, such as that from the Miocene Monterey Formation of California, in the relative amount of aliphatic, monoaromatic and pyrrolic compounds. However, the MDC samples are enriched in phenols, most likely due either to admixed terrestrial organic matter or to weathering at the outcrop. They are also not as rich in sulfur as the Monterey, as evidenced by their lower thiophene concentrations.

4) The dominant organic matter types are marine, including several types of fossil algae and amorphous material (largely the product of bacterial reworking of primary organic matter). There is petrographic and chemical evidence of minor terrestrial input.

5) The presence of isoprenoid hydrocarbons (prist-1-ene) in the pyrolyzates and $17\beta(H)$, $21\beta(H)$ hopanes in the extractable organic matter attest to the low level of thermal alteration of the MDC shales.

6) The MDC organic matter appears to be partly oxidized, probably from weathering at the outcrop. It is recommended that fresh, unweathered samples be obtained prior to further organic geochemical studies, preferably by coring.

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