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Biological Markers in Lower Jurassic Synrift Lacustrine Black Shales, Hartford Basin, Connecticut, U.S.A.

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Abstract-The East Berlin Formation (Lower Jurassic, Hartford basin, Connecticut, U.S.A.) is distinctive for its six cyclic units of lacustrine black shale and gray mudstone. separated by playa and fluvial redbeds. The black shales arc each about a meter thick and were deposited in subtropical, thermally stratified, oligomictic lakes, the youngest of which (lakes 3 through 6) were large enough to flood most of the basin and attained depths of several tens of meters.

The saturate fractions of solvent extracts of organic-rich black shales from each of the six lakes, collected at fresh roadcuts near East Berlin, arc dominated by extended homologous series of *n*-alkanes, alkylcyclohexanes, and branched chain alkanes. A striking feature of the black shales is the presence of a series of extended tricyclic terpanes from C_{20} to at least C_{41} . Hopanes are either not detectable or present only in subordinate quantities relative to the tricyclic terpanes. The samples arc depleted in hopanes in part because of the elevated maturity level (mid to late oil window). Tricyclic terpane concentrations may also have been enhanced by fractionation effects related to oil expulsion out of the black shales. In addition, the original organic matter may have been exceptionally rich in tricyclic terpane precursors, i.e. fossil lipids of prokaryotes present in anoxic, moderately saline, alkaline lakes.

Key words - tricyclic terpanes, lacustrine depositional environments, petroleum source rocks, Connecticut, rift basins. Jurassic.

INTRODUCTION

Numerous rift basins developed in the early Mesozoic on both sides of the future Atlantic, in response to regional extension during the breakup of Pangaea (Van Houten, 1977; Lorenz, 1988). Along the eastern margin of North America, synrift deposits filling these basins are collectively termed the Newark Supergroup, and include continental strata (redbeds), basaltic flows and dikes, and lacustrine rocks, the latter commonly organic-rich. There is now considerable interest in the petroleum potential of these basins, particularly offshore. The onshore basins of the Newark Supergroup, especially the Newark basin (northern New Jersey and adjacent Pennsylvania) and the Hartford basin (central Connecticut), have been well-studied geologically and are potential analogs for offshore frontier provinces.

Personnel from the U.S. Geological Survey and their collaborators have recently made significant advances in our knowledge of the organic geochemistry of the Newark Supergroup, particularly in the Newark and Hartford basins. Pratt et al. (1986) examined lacustrine shales from these basins for petroleum source potential, using bulk analysis

(organic carbon contents and Rock-Eval pyrolysis) and stable carbon isotope techniques. Spiker et al. (1988) used stable carbon isotope and aromaticity data (from solid state ¹³C NMR) to conclude that the kerogens in black shales from the Newark and Hartford basins contain lacustrine algae and woody plant detritus in highly variable proportions, and therefore are gas-prone. These observations were refined by Kotra et al. (1988), who note that, while kerogens are mixed, the pyrolyzable fractions are predominantly oil-prone. They also note a regional decrease in maturity from north to south in the Hartford basins to be more complex. In addition to the effects of elevated regional heat flow due to rifting, they note local thermal anomalies near large basalt intrusions, as well as anomalies suspected to result from the migration of hydrothermal fluids. Pratt and Burruss (1988) observe that, while the organic-rich shales of these basins are thin and stratigraphically discontinuous, their aggregate thickness is adequate for economic petroleum generation. Through field observations, fluid inclusion studies and chromatographic examination of extractable organic matter, they document the occurrence of oil expulsion.

This paper focuses on the molecular organic geochemistry, especially the unusual biological marker distributions, of lacustrine black shales in the Lower Jurassic East Berlin Formation of the Hartford basin, a topic which has not previously been discussed in adequate detail.

EXPERIMENTAL

Black shale samples were collected from fresh roadcuts in the East Berlin Formation near the intersection of Connecticut state highways 72 and 15 (lakes 1, 2 and 3) and near the intersection of Connecticut state highway 9 and U.S. interstate highway 91 (lakes 4, 5, and 6), approximately 2 km west and 2 km east of the village of East Berlin, respectively (Fig. 1).

Samples were pulverized and exhaustively extracted (CH₂Cl₂) in a soxhlet apparatus. The dried extracts were separated by liquid chromatography into "saturate", "aromatic", "polar 1" and "polar 2" fractions (silica gel, with C_6H_{14} , 9:1 C_6H_{14} :CH₂Cl₂, CH₂Cl₂ and 1:1 CH₂Cl₂:CH₃OH as the respective eluants). The saturate and aromatic fractions were analyzed by gas chromatographic mass spectrometry (GC-MS) with a 25 m OV-1 column (0.2 mm i.d., film thickness of 0.33µm), initially held at 100°C for 10 min, then programmed from 100 to 300° C at 3°C min⁻¹, then held at 300°C for 18 min, using a Hewlett-Packard 5890A GC coupled to an HP 59708 Mass Selective Detector run in selected ion monitoring mode.

PALEOLIMNOLOGICAL SETTING

The Upper Triassic-Lower Jurassic Hartford basin is filled with about 4.7 km of terrestrial redbeds, lacustrine gray-black strata and basalt lava flows that accumulated in a half-graben bordered on the east by a listric normal fault (Fig. 1). This synrift basin formed in the subtropics at about 25°N paleolatitude in response to tensional forces associated with the initial rifting of North America from Europe and Africa. Our research focuses on one unit of the Hartford Group, the East Berlin Formation, which, in central Connecticut, is made up of 170 m of playa, lacustrine and fluvial deposits. In the middle and upper parts of the formation, six intervals of lacustrine gray-black strata 4-8 m in thickness are interbedded with playa and fluvial red mudstones and sandstones (Fig. 2) (Hubert et al., 1978; Hubert et al., 1982; Demicco and Kordesch, 1986; Meriney, 1988). The lacustrine intervals tend to be vertically spaced at about 10 m, defining Van Houten cycles, named after the pioneering work of Van Houten (1964) who described similar cycles in the Upper Triassic Lockatong Formation of the Newark basin in New Jersey. The cyclicity is interpreted as due to

Fig. 1. Generalized stratigraphy of the Triassic-Jurassic Hartford Group in central Connecticut and simplified geologic map showing the southern portion of the Hartford basin in central Connecticut. The two measured sections (●) are described in detail by Meriney (1988). Lakes 1-3 were sampled at the western measured section and lakes 4-6 at the eastern.



fluctuations in precipitation controlled by the 23,000-year precession period of the earth's axis (Van Houten, 1964; Olsen, 1986). The expansion and contraction of each of the six perennial, oligomictic lakes is recorded by a symmetrical cycle consisting of gray mudstone and sandstone at the base, overlain by ≈ 1 m of black shale, overlain in turn by gray mudstone and sandstone. The central black shale reflects deposition during the wettest part of the climate cycle, when the lake was deepest and most extensive. During the times of increased precipitation, lakes were produced which deepened eastward, filling closed depressions on the basin floor where it sagged adjacent to the fault-bounded eastern highlands. At its maximum development, lake 3 was the largest, deepest and least saline of the six lakes. At its greatest extent, it was at least 100 by 20 km (Olsen, 1988), which implies depths of at least several tens of meters.

Black muds accumulated in the lakes at times of their maximum size and depth. The black shales in each lake cycle, except lake 6, contain varying amounts of "laminite" composed of alternating mm-scale laminae of dolomite and detrital clay/kerogen. These laminae pairs are interpreted as varves (Van Houten, 1964; Hubert et al., 1976). During the dry season, Mg-calcite precipitated from the epilimnion of each thermally stratified, subtropical lake, perhaps associated with algal blooms. With the onset of the rainy season, detritus was washed into the lake. O₂-poor bottom waters precluded colonization by burrowing infauna, thus preserving undisturbed laminae. In each lacustrine cycle, "laminite" is most common in the lower part or the black shale, implying that each lake reached maximum depth with stable thermal stratification early in its history, followed by gradual shallowing, recorded by the transition from black to gray mudstone. In each lake, organic matter was preserved in the black muds beneath the anoxic hypolimnion. The total organic carbon content or the black shales is 2-4% (Pratt et al., 1986; Olsen, 1988; Meriney, 1988).

The minerals precipitated from the lake waters and the authigenic phases that formed during diagenesis show that the lakes contained alkaline, hard water, with high concentrations or HCO₃, Mg²⁺ and Ca²⁺, together with lesser amounts of Na⁺ and SO₄²⁻. The Mg/Ca ratio was high. Lake water salinities increased as the lakes contracted, so that in the shallow water gray mudstones overlying the black shales, there are dolomite concretions, corrensite (April, 1981), and traces or analcime and gypsum. Hypersaline conditions were not reached or only briefly attained, as the lakes contracted to final desiccation.

ORGANIC GEOCHEMICAL RESULTS

The saturate fraction accounts for 69-75% or the black shale extracts. Within the saturate fraction, *n*-alkanes predominate. The distributions or normal and branched alkanes, as seen on m/z 99 mass chromatograms, are shown in Fig. 3. Two maxima are apparent amongst the six samples: one at C_{20} and the other in the C_{25} - C_{27} region. The lake 3 sample fits the former category, the lake 4 case fits the latter, while lakes I, 2, 5 and 6 exhibit dual maxima. The *n*-alkanes up to C_{37} are detected in most cases. The distribution of *n*-alkanes shows no significant odd/even or even/odd predominance.

Norpristane, pristane and phytane are prominent isoprenoids in all six samples, present in nearly equal relative concentrations (Fig. 3). 2,6,10-trimethyltridecane is also important, however varying amounts were lost to evaporation during sample preparation, making comparisons or its concentrations less reliable. In the extracts or lakes 2, 3 and 5, isoprenoid/*n*-alkane ratios (such as pristane/*n*-C₁₇) are high for such mature samples. This may indicate slight biodegradation (Connan and Cassou, 1980), preferential depletion of *n*-alkanes due to fractionation during oil migration (Leythaeuser and Schwarzkopf, 1986) or may simply indicate an originally isoprenoid- rich bitumen. There is a complex distribution of minor branched alkanes, seen on the m/z 99 traces (Fig. 3).

Fig. 2. Stratigraphic section of the East Berlin Formation at the sample localities. Methylphenanthrene Index values are shown for the extracts of each of the 6 black shales.



Fig. 3. Distribution of normal and branched alkanes in the saturate fractions of the extracts of each of the six black shales, shown on m/= 99 mass chromatograms. A= norpristane; B = pristane; C = phytane. Normal alkanes are labelled by carbon number.



Compounds with both chain and cyclic character are also important features of these samples. An homologous series of long chain alkylcyclohexanes is evident on m/z 83 mass chromatograms from all six samples, in amounts subordinate to the normal alkanes. The presence of γ -carotane and β -carotane is noted on m/z 123 and 83 mass chromatograms, eluting immediately after *n*-C₃₆ and *n*-C₃₇, respectively. Identifications are based on GC elution and mass spectral character, following Jiang and Fowler (1986). β -carotane has previously been detected in immature lacustrine black shales of the Newark and Hartford basins (Pratt and Burruss, 1988), but is here found in the mature as well. In the aromatic fraction, an homologous series of alkylbenzenes is present, viewed on m/z 91 traces.

Among the most striking features of these samples is the presence of series of extended tricyclic terpanes and the absence of the typical sequence of hopanes (Kruge et al., 1989), as shown on the m/z 191 mass chromatogram of the lake 4 saturate fraction (Fig. 4). With the chromatographic conditions employed in this study, the C_{30} hopane would elute between the peak pairs marked 30 and 31 on Fig. 4. A minor peak in this region matches the retention time of hopane. Clearly, if present at all. hopanes are minor constituents relative to the tricyclic terpanes in this sample. The C_{24} tetracyclic terpane is also absent in the lacustrine samples. An additional prominent feature of Fig. 4 is a pronounced "hump", due to a complex mixture of chromato- . graphically unresolvable compounds giving m/z 191 fragments.

The tricyclic terpane series in the C₁₉-C₂₉ range is commonly recognized in many rock extract and petroleum samples. In this case, the series extends to at least C₄₁ and possibly beyond. The high molecular weight tricyclic terpanes have previously been recognized in a terpane concentrate prepared from a Californian petroleum and by metastable ion GC-MS analysis of the same sample (Moldowan et al., 1983). The characteristic GC elution pattern of the series may be seen in Fig. 4. For C₂₅ and above, C-22 is a chiral center and the resulting stereoisomeric pairs appear as doublets (Ekweozor and Strausz, 1983). The retention time gap between each pair widens as the carbon number increases. Beginning with C₃₀, C-27 also becomes a chiral center and the peak doublets begin to divide into quartets. This phenomenon is difficult to discern with the chromatographic condition employed here, but close examination of Fig. 4 reveals that by C₃₅ the peaks are broadening and the peak tops are beginning to split. Another chromatographic characteristic of the series is lack of the C₂₂, C₂₇, C₃₂, C₃₇ and C₄₂ members. The skipping of every fifth carbon number is due to the isoprenoid nature of the side chain, i.e. methyl substitution at every fourth carbon in the chain, as shown in the molecular structure in Fig. 4 (Moldowan et al., 1983).

The organic-rich black shales from each of the six lakes display a predominance of the extended tricyclic terpane series on their m/z 191 mass chromatograms, as shown in Fig. 5. In all six samples, hopanes are extremely weak, relative to the tricyclic terpanes, if they are present at all. As in the case of the *n*-alkanes, the distribution of tricyclic terpanes varies from lake to lake, chiefly in the ratio of lower molecular weight tricyclics ($< C_{27}$) to higher. The samples from lakes 3 and 4 are characterized by more abundant low molecular weight tricyclics, whereas with lakes 5 and 6, the opposite is true. Lakes 1 and 2 show a more balanced distribution between low and high molecular weights.

The steranes are in extremely low concentrations in the saturate fraction of each sample, detectable only as minor peaks, barely above the baseline on m/z 218 mass chromatograms. Thus, the standard sterane maturity parameters (Mackenzie et al., 1982) could not be used. Similarly, for the aromatic fractions, the monoaromatic steroids are not detectable on m/z 253 mass chromatograms and only the lower molecular weight (C_{20} and C_{21}) triaromatic steroids are detectable on m/z 231 traces. Consequently, the standard maturity parameters using the ratio of monoaromatic to triaromatic steroids and the relative

Fig. 4. m/z 191 mass chromatogram of the saturate fraction of the extract of the lake 4 black shale. Tricyclic terpane peaks are labelled by carbon number.



Fig. 5. Distribution of tricyclic terpanes in the saturate fractions of the extracts of each of the six black shales, shown on m/z 191 mass chromatograms. Peaks are labelled by carbon number.



concentrations of low and high molecular weight triaromatic steroids (Mackenzie et al., 1983) are at their maximum possible value.

To further specify the extent of maturation, we have turned to the Methylphenanthrene Index (MPI), which is especially useful in the case of mature samples (Radke and Welte, 1983). The MPI results are plotted, along with the stratigraphic position of the sample, on Fig. 2. The values vary from 0.93 in lake 3 to 1.15 in lakes 1 and 2, without regard for stratigraphic position. In addition, we record a mean vitrinite reflectance of 1.13% (based on 75 readings) from a single lake 4 black shale sample.

DISCUSSION

Thermal maturity

We place the East Berlin samples in the middle-to-upper oil window, based. on the MPI, vitrinite reflectance and triaromatic steroid data. The presence of oil-stained sandstones and oil-fractures above and below the black shales confirm that generation and migration of petroleum has occurred. There are basaltic lava flows 20 m above lake 6, however, there appears to be no contact metamorphic effect (Fig. 2).

At their maximum burial, rocks at the site were covered by up to 2 km of Jurassic overburden (Hubert et al., 1978). A geothermal gradient of 45-50°C/km and an average surface temperature of 15-20° C are reasonable values for a subtropical site of active rifting. Subsurface temperatures of 105-120°C could thus be easily achieved by burial heating alone, close to adequate for peak oil generation, depending on the heating rate. Additional heat was input into the system by hydrothermal activity associated with magmatism (Pratt et al., 1988; Walters and Kotra, 1989).

Comparison of East Berlin black shales with other lacustrine rocks

As described above, the East Berlin black shales were deposited in anoxic, moderately saline lakes. One expects that their biomarker assemblages should differ from those of hypersaline lakes. The distributions of normal and branched alkanes from the East Berlin black shales indeed do not resemble those of samples from hypersaline lakes, which are characterized by a predominance of even carbon number *n*-alkanes over odd and by low pristane/phytane ratios (ten Haven et al., 1988; Fu et al., 1988). Non-hypersaline lacustrine deposits, such as Carboniferous-Permian units from western China (Fu et al., 1988) and the Middle Devonian of Scotland (Hall and Douglas, 1983), do resemble the Connecticut samples in their normal and isoprenoid alkane distributions. Carotanes, detected in East Berlin black shales, are also important biological markers recognized in certain organic-rich lacustrine rocks, such as the Green River Shale (Murphy et al., 1967), as well as in the western Chinese and Scottish examples just cited. In contrast, carotanes are not important constituents of hypersaline deposits (ten Haven et al., 1988; Fu et al., 1988).

As we have shown, the predominant terpanes in the East Berlin black shales are tricyclic. These compounds are also present in significant quantities \cdot in certain other lacustrine units. Middle Devonian strata of Scotland include lake deposits, similar in lithology and depositional environment to those in the Hartford basin. Duncan and Hamilton (1988) show that tricyclic terpanes are always important biomarkers for these rocks. However, unlike the Connecticut black shales, hopanes are always present as well, except for one sample. In that particular case, C₃₀ and larger extended tricyclic terpanes appear to be present, in very small amounts relative to their lower molecular weight homologues, although they are not identified by the authors. Philp and Fan (1987) discuss oils from the Jianghan basin of eastern China which have high tricyclic to pentacyclic terpane ratios and pristane/phytane ratios of \approx 1. The source of these oils is unknown, but they infer a high salinity, anoxic lacustrine depositional environmental. for it. Tricyclic terpanes are significant in Neocomian lacustrine deposits from offshore Brazil (Aquino Neto et al., 1986), but the authors do not mention any with carbon numbers above 27. The terpane distributions of hypersaline rocks are very different, with abundant pentacyclic terpanes, including gammacerane, and steranes (ten Haven et al., I 988; Fu et al., 1988).

In general, there apparently is a class of non-hypersaline, organic-rich lacustrine rocks characterized by (1) a balanced distribution of odd and even carbon number *n*-alkanes, (2) nearly equal amounts of the isoprenoids norpristane, pristane and phytane, and (3) high tricyclic to pentacyclic terpane ratios. The East Berlin black shales are representative of this class. In contrast, hypersaline rocks have a predominance of even *n*-alkanes over odd, low pristane/phytane ratios, and abundant pentacyclic terpanes, including gammacerane. These observations should allow for greater accuracy in oil to source rock correlation, especially when the source is lacustrine.

Origin of the tricyclic terpanes

The usual occurrence of the extended tricyclic terpane series and the absence of the hopanes in the Connecticut samples may be explained by (I) the high thermal maturity of the samples, (2) preferential depletion of hopanes by migration, and/or (3) production of tricyclic terpane precursors by unusual biota favored by the original depositional environment.

The high thermal maturity, as described above, may have resulted in preferential destruction of hopanes, leaving the more thermally stable tricyclic terpanes in the residuum. Thermal alteration of crude oil under laboratory conditions does increase the tricyclic to pentacyclic terpane ratio (Aquino Neto et al., 1983). However, for terpanes in source rocks under natural conditions, the situation may be different. In a concurrent project examining a complete lacustrine gray mudstone to black shale to gray mudstone sequence in the East Berlin Formation, we have found a normal series of hopanes in the gray mudstone. Therefore, thermal effects alone do not fully account for the lack of hopanes only one meter away in the black shale. Fractionation during oil migration may have preferentially depleted the black shales in hopanes. Migration has certainly occurred, documented by the presence of oil-stained sandstones and fractures.

Lakes, due to their isolated nature, may develop unusual or even unique water chemistries (Kelts, 1988), which control the assemblage of organisms capable of survival in. lake. The type of organic matter, the degree of its preservation and its early diagenetic modification are all functions of lake water chemistry. With this in mind, we speculate that unusual prokaryotes favored by the particular limnological conditions of the East Berlin lakes may have synthesized tricyclic terpane precursors, using algal debris for raw material, as part of their normal metabolism. The algal remains would include large polyprenols, which anaerobes might cyclize (Heisler et al., 1984). Alternatively, perhaps more common microorganisms, attempting to form hopanes, may have incompletely cyclized polyprenols, forming the extended tricyclic terpanoids instead, perhaps in response to environmental (chemical) stress. In any case, long chains are consistently a prominent feature of the GCamenable compounds in these samples, whether they be fully acyclic, alkylcyclohexanes, carotanes, alkylbenzenes or extended tricyclic terpanes.

CONCLUSIONS

(1) The Lower Jurassic East Berlin Formation in the synrift Hartford basin consists of playa and fluvial red beds, interrupted by six cyclical intervals of lacustrine black shale and gray mudstone. The black shales are organic-rich and were deposited during the most humid portions of the climate cycle, in subtropical, thermally-stratified, oligomictic lakes.

(2) The solvent extracts of the black shales from the six lakes are all very similar. Long chain structures predominate, especially *n*-alkanes, branched chain alkanes, alkylcyclohexanes, carotanes and alkylbenzenes. Steroids arc in low concentration. Maturity is at mid to late oil window levels, confirmed by Methylphenanthrene Indices in the 0.93-1.15 range and a vitrinite reflectance-value of 1.13%.

(3) The black shales in all six lacustrine cycles are characterized by a series of extended tricyclic terpanes (C_{20} to at least C_{41}) and by the absence of hopanes. This unusual assemblage of biomarkers most likely reflects the anoxic, moderately saline, alkaline lacustrine environment of deposition, the high level of thermal maturity, and fractionation during oil migration.

(4) The East Berlin black shales apparently represent a class of non-hypersaline, organic-rich lacustrine rocks characterized by a balanced distribution of odd and even carbon number *n*-alkanes, nearly equal amounts of the isoprenoids norpristane, pristane and phytane, and high tricyclic to pentacyclic terpane ratios. In contrast, hypersaline lacustrine rocks have a predominance of even *n*-alkanes over odd, low pristane/phytane ratios, and abundant pentacyclic terpanes, including gammacerane. These observations should allow for greater accuracy in oil to source rock correlation, especially when the source is lacustrine.

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