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54. *DATA REPORT*: THE CRETACEOUS/TERTIARY BOUNDARY AT SITES 761 AND 762 (NORTHWEST AUSTRALIAN MARGIN), AT EL KEF (TUNISIA), AND IN THE NEGEV DESERT (ISRAEL): A COMPARISON OF THE ORGANIC GEOCHEMICAL RECORDS[1]

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ABSTRACT

Regional consequences of the biotic extinctions and of the changes in biological productivity that occurred at the time of the Cretaceous/Tertiary (K/T) boundary were investigated by comparison of organic matter in sediments from three southern Tethyan margin locations. Organic matter characterization comprised Rock-Eval pyrolysis and organic carbon measurements. Low concentrations of organic matter precluded additional detailed determinations. At all three locations, the organic matter has been microbially reworked and evidently was deposited in oxygenated marine environments.

INTRODUCTION

The Cretaceous/Tertiary (K/T) boundary occurs in sediments cored at Site 761 on the Wombat Plateau and at Site 762 on the Exmouth Plateau during Ocean Drilling Program (ODP) Leg 122. Recovery of Maestrichtian and Danian sediments from these sites provided an opportunity to compare the organic matter contents with those of sediments from two other locations—El Kef, Tunisia, and the Negev Desert, Israel—that have paleosettings on the southern edge of the Tethyan Seaway.

Changes in the amount and type of organic matter deposited in sediments would be expected to accompany the massive reductions in the numbers and diversity of organisms that occurred at the end of the Cretaceous period ca. 66 Ma. The marine fossil record, for example, shows marked depletions in calcareous plankton in sediments deposited in the earliest Danian (e.g., Jiang and Gardner, 1986; Keller, 1988a, 1988b). On land, dinosaurs became extinct at the end of the Maestrichtian (e.g., Sloan et al., 1986), and forests were destroyed (e.g., Tschudy et al., 1984; Saito et al., 1986).

Published reports of the results of organic geochemical studies of the K/T boundary are few (e.g., Simoneit and Beller, 1985, 1987; Venkatesan and Dahl, 1989; Meyers and Simoneit, 1990). This paucity of information may reflect the difficulty of doing such studies on rocks lean in organic carbon, as well as the limited availability of samples suitably preserved for organic geochemical studies.

SAMPLES AND ANALYSIS

Samples

Samples were obtained from three settings that were formerly on the southern edge of Tethys. All of the samples were present on the *JOIDES Resolution* during Leg 122. Their organic geochemical contents were consequently analyzed using the same shipboard procedures and equipment.

Northwest Australian Margin

The paleolatitude of the northwest Australian margin was ~45°S at the end of the Maestrichtian (Fig. 1), which is notably farther to the south than its present position at ~21°S.

Site 761, Wombat Plateau

The Wombat Plateau is located in the Indian Ocean off the northwest coast of Australia. Site 761 is in 2168 m of water, and the interval containing the K/T boundary was cored using rotary core barrel (RCB) drilling. The K/T boundary appears to be free of hiatuses and comprises a pelagic carbonate sequence.

Site 762, Exmouth Plateau

The Exmouth Plateau is to the southwest of the Wombat Plateau of the northwest coast of Australia. Site 762 is in 1500 m of water. Coring was done with the extended core barrel (XCB). Only pelagic Danian sediments were obtained at this site because of incomplete core recovery.

El Kef, Tunisia

The El Kef location has experienced little tectonic movement from its paleolatitude in the Maestrichtian (Fig. 1). The section has an expanded and nearly complete boundary record, as summarized by Keller (1988a, 1988b; Keller and Lindinger, 1989). The paleoenvironment was on the southern Tethyan upper slope to outer shelf, resulting in virtually no interruption in sedimentation leading up to and following the K/T boundary. The boundary clay itself, however, may represent a fairly extended hiatus. A large number of small subsamples were collected from the outcrop as part of a micropaleontological study. These samples were stored dry at room temperature for several years before being used in this study.

Negev Desert, Israel

This location has a sequence similar to the well-known Gubbio sequence in Italy. Its paleoenvironment was a shallow carbonate platform on the southern margin of the Tethyan Sea (Fig. 1), separated from Africa by fairly deep water. A hiatus in sedimentation exists between the late Maestrichtian and the early Danian, possibly indicating a lowstand. Numerous Negev sites have been sampled; the ones at Ein Mor and Hor

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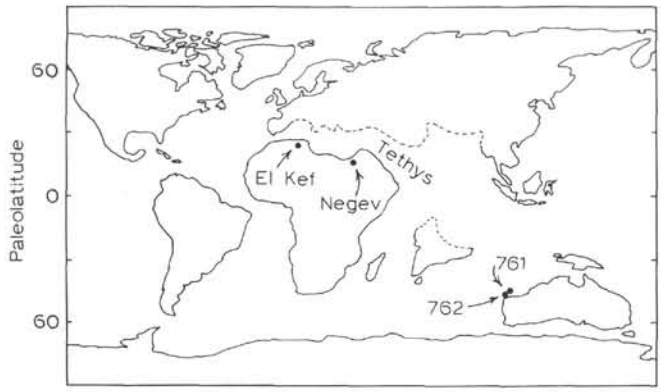

Figure 1. Paleolocations of K/T boundary sites investigated in this study. The areas corresponding to the present-day locations of El Kef, Tunisia, the Negev Desert, Israel, and Sites 761 and 762 are shown relative to the southern Tethyan margin in the Maestrichtian on the plate tectonic reconstruction of Barron (1987). The Australian sites have subsequently moved much closer to the equator, whereas the other two sites have experienced little motion.

HaHar have been described by Magaritz et al. (1985) and Magaritz (1989), who present microfossil, isotope, and paleomagnetic data for this sequence. My samples come from two outcrop locations, one very near the Egyptian/Israeli border in the Sinai and the other at Ein Mor. Both are not far from Hor HaHar. The samples were collected in 1988 and were kept frozen until this analysis began.

Carbonate Carbon Concentrations

Concentrations of inorganic carbon were determined on freeze-dried samples using a Coulometrics 5010 coulometer equipped with a 5030 carbonate carbon analyzer (cf. Engleman et al., 1985). In this instrument, carbonate carbon was converted to CO_2 by treatment with HCl, and the amount of liberated CO_2 was measured by titration in a monoethanolamine solution with a colorimetric indicator. A photodetection cell was used to monitor the end point. Inorganic carbon concentrations were converted to carbonate percentages, assuming all of the inorganic carbon was present as calcium carbonate.

Total Organic Carbon and Rock-Eval Analyses

Total organic carbon (TOC) concentrations were determined as part of the Rock-Eval analysis of samples (cf. Espitalié et al., 1977). The Rock-Eval instrument on the *JOIDES Resolution* is a Girdel Rock-Eval II equipped with a TOC module. Programmed pyrolysis of samples from 300° to 600°C gives the amount of hydrocarbons released during heating in a helium atmosphere (S_2) and the amount of CO_2 released during pyrolysis to 390°C in an oxygen atmosphere (S_3). These values are used to calculate the hydrogen index ($HI = 100 \times S_2/TOC$), the oxygen index ($OI = 100 \times S_3/TOC$), and the concentration of total organic carbon. The TOC module combusts the residue of the temperature-programmed sample in air at 600°C and sums the product of this oxidation with those of the preceding temperature-programmed pyrolysis to give the total organic carbon. Samples rich in carbonate and poor in organic matter can yield artificially elevated and unreliable OI values, partly because a small proportion of the carbonate may break down during heating to yield CO_2 and partly because of the errors introduced by dividing the S_3 value by a small TOC value (cf. Katz, 1983). Results from such samples must be interpreted cautiously.

Clay Mineralogy

Mineral compositions of Negev Desert samples were surveyed using the shipboard Philips ADP 3520 X-ray diffraction system. This instrument is equipped with a Cu X-ray source and a monochromator. Scanning was performed over the 2θ range of 2° to 70° at a rate of 0.12° 2θ/min. Peak areas were measured by computer-aided electronic integration. Mineralogical determinations were limited to 13 samples because of recurring failures of the ship's regulated power supply.

RESULTS

All of the samples contained <1% organic carbon, and many were near or below the limit of TOC measurement (0.01%) (Table 1). Carbonate carbon contents, in contrast, were quite variable. The low concentrations of organic carbon could be caused by poor preservation of organic matter during or after sediment deposition, or they could indicate originally low production of organic matter by marine and continental plants. Anticipated biomarker and isotopic analyses of these K/T boundary samples were frustrated by the low concentrations of organic matter, especially in the case of the Australian margin samples.

Organic carbon concentrations of the El Kef samples were adequate to yield reliable Rock-Eval data (Fig. 2); the combination of these results and the rock lithology suggests that most or all of the organic matter at this site is oxidized Type II marine material. Microfossil analyses (Keller, 1988a, 1988b; Keller and Lindinger, 1989) support this inference. An attempt to perform Rock-Eval analyses on the organic-carbon-lean Negev Desert samples was only partially successful because the carbonate matrix of the rocks contributed pyrolytic CO_2, yet the low HI values that were obtained similarly suggest that this organic matter is heavily oxidized marine matter. The extremely low TOC concentrations of the Site 761 and 762 samples (Table 1) precluded obtaining reliable HI and OI Rock-Eval values from these samples. Preservation of organic matter evidently was not good at the shallow water locations in Tunisia and Israel nor at the deeper sites on the Australian margin.

Clay mineral assemblages show no differences between Maastrichtian and Danian sediments from the Negev sites. Quartz is a notable noncarbonate component of the mineral

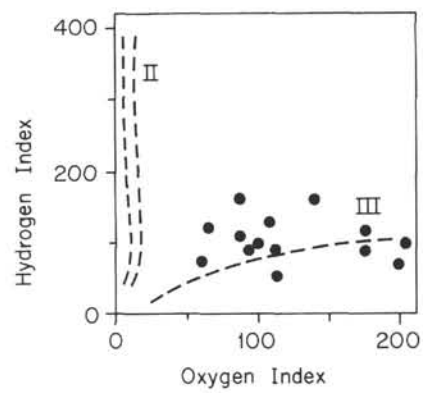

Figure 2. A van Krevelen-type plot of Rock-Eval hydrogen index and oxygen index values obtained from pyrolysis of organic matter in samples from El Kef, Tunisia. Consideration of the lithology and microfossil contents of these samples indicates that these samples contain Type II marine organic matter that has been oxidized to resemble Type III continental material.

Table 1. Organic carbon and calcium carbonate concentrations of K/T boundary samples from the three southern Tethyan margin locations.

Location and sample number	C_{org} (%)	$CaCO_3$ (%)
Northwest Australian Margin		
Wombat Plateau		
Danian samples:		
122-761C-3R-1, 14–16	0.02	68
122-761C-3R-1, 105–107	0.01	66
122-761C-3R-2, 10–12	0.01	67
122-761C-3R-3, 55–57	0.01	68
Maestrichtian samples:		
122-761C-3R-3, 85–87	0.15	88
122-761C-3R-3, 126–128	0.01	89
122-761C-3R-4, 126–128	0.02	91
122-761C-3R-5, 69–71	0.03	91
Exmouth Plateau		
Danian samples:		
122-762C-42X-5, 12–16	0.03	25
122-762C-42X-5, 80–81	0.03	30
122-762C-42X-6, 124–128	0.01	30
El Kef, Tunisia		
Danian samples:		
AFN 599	0.19	23
AFN 595	0.24	37
AFN 590	0.32	43
AFN 587	0.63	40
AFN 585	0.95	22
AFN 580	0.60	8
AFN 570	0.66	9
AFN 560	0.30	12
AFN 555	0.27	6
AFN 550	0.27	8
AFN 549	0.26	6
AFN 548	0.29	6
AFN 547	0.25	4
AFN 546	0.23	5
AFN 545	0.30	4
AFN 544	0.34	3
AFN 543	0.33	4
AFN 542	0.41	2
AFN 541	0.50	6
Maestrichtian samples:		
AFN 540D	0.31	45
AFN 540L	0.23	41
AFN 538	0.29	37
AFN 537	0.25	38
AFN 536	0.31	38
AFN 538	0.16	43
AFN 534	0.23	43
AFN 533	0.18	41
AFN 532	0.28	42
AFN 531	0.33	39

compositions and may be an eolian component of these fine-grained carbonate samples.

DISCUSSION

The widespread biotic perturbation at the K/T boundary remains unexplained, notwithstanding considerable discussion from a large segment of the earth science community. The general occurrence of iridium enrichment in the boundary clay layer has been interpreted as evidence of an extraterrestrial cause—a massive meteorite impact (Alvarez et al., 1984). This enrichment has also been considered prime evidence of extensive vulcanism at the K/T transition (Officer et al., 1987), a time that coincides with the Deccan Plateau basalt flows (Jaeger et al., 1989). Either scenario could induce a global change in climate, yet the iridium anomaly may arise from microbial activity and authigenic precipitation of this metal from sediment pore waters (Schmitz, 1988; Schmitz et al., 1988; Dyer et al., 1989). A third possible explanation of the boundary extinctions is that of climatic change brought about by the culmination of gradually accumulating, noncatastrophic factors (Crowley and North, 1988). Indeed, many of the extinctions on land and sea appear to be gradual, rather than abrupt (e.g., Sloan et al., 1986; Keller, 1989; Barrera and Keller, 1990).

Mineral Assemblages

Rampino and Reynolds (1983) report the results of a study of the clay mineral assemblages from four K/T boundary locations. The study investigated the possible existence of the exotic minerals that would be expected to be found in boundary clays as a result of an asteroid impact. All of the boundary clays—from Nye Kløv (Denmark), Gubbio (Italy), El Kef (Tunisia), and Caravaca (Spain)—contain common clay minerals indistinguishable from clays below and above the boundary. Differences in mineral assemblages from the four locations imply that the clays originated locally (Rampino and Reynolds, 1983). Although a boundary clay layer does not exist in the Negev, my survey of the clay minerals from Maestrichtian and Danian limestones from this locality similarly shows no difference between samples from below or above the boundary. Small differences consistently appear, however, between Danian samples from Ein Mor and Danian samples from the Sinai border site, indicating that local conditions influenced sediment compositions.

Biomarker Molecules

The results of analyses of compositions of extractable biomarker molecules have been reported for samples from Deep Sea Drilling Project (DSDP) Sites 577 (northwestern Pacific Ocean) and 605 (New Jersey continental margin) and from Stevns Klint (Denmark) by Simoneit and Beller (1985, 1987). Microbial biomarkers dominate the geolipid contents of the Site 577 and Stevns Klint samples. The Atlantic Margin Site 605 material contains n-alkanes from continental higher plant waxes in addition to the bacterial biomarkers. Polycyclic aromatic hydrocarbons derived from abietic acid, a component of conifer resin, are present in the DSDP samples but not in the Stevns Klint rocks, evidently as a result of better preservation of organic matter in the submarine locations than in the subaerial K/T exposures. Other aromatic hydrocarbons present in the DSDP samples, including components of the phenanthrene and the pyrene series, may have originated from either combustion or from erosion of old continental deposits. A predominance of alkyl substitution in these aromatic series suggests that land-derived aromatic hydrocarbons are abundant in the sediments of the Site 605 section, where van Hinte et al. (1987) postulate on the basis of a change to coarser sediment texture that a lowered sea level existed at the time of the K/T boundary. Continental organic matter would more easily be delivered to this location during times of lowered sea level. Distributions of polycyclic aromatic hydrocarbons from K/T exposures on land, however, contain strong evidence of pyrolytic generation of these molecules (Venkatesan and Dahl, 1989). A possible origin of these types of aromatic hydrocarbons is from a hypothesized widespread, perhaps even global, combustion of land vegetation at the time of the K/T biotic extinctions (Wolbach et al., 1985, 1990).

Isotopic and Elemental Compositions

Schimmelmann and DeNiro (1984) measured the isotopic contents of carbon, nitrogen, and hydrogen in organic matter across the freshwater K/T boundary located in the York Canyon exposure of the Raton Basin, New Mexico. They

found a shift of 1.8‰ to lighter organic carbon isotopic values in the early Tertiary, which parallels a similar shift of 1‰–2‰ found in marine carbonates from the South Atlantic (Shackleton and Hall, 1984), Israel (Magaritz, 1989), the North Pacific (Zachos et al., 1989), and Tunisia (Keller and Lindinger, 1989). The common finding of the isotope shift in organic and inorganic carbon implies a global perturbation of the carbon cycle. The nature of this perturbation is thought to be globally depressed bioproductivity (Zachos and Arthur, 1986; Magaritz, 1989). A period during which the rate of oxidative recycling of organic matter exceeded photosynthetic carbon fixation would result in the return of isotopically light organic carbon to the inorganic carbon reservoir and would produce the observed isotope shifts.

In contrast to the systematic change in organic carbon isotopes, δD ratios remain between –105‰ and –125‰, and $\delta^{15}N$ values vary randomly between 2‰ and 4‰ in samples from the York Canyon site. The lack of systematic change in hydrogen or nitrogen isotopic compositions suggests that no major fluctuations occurred in the water balance or the temperature of this swampy freshwater setting during this period of other types of global change.

An exception to the general pattern of lighter carbon isotope values in sediments deposited after the K/T boundary is found in the K/T section in eastern Hokkaido, Japan. At this location, organic carbon isotopic values change from –26‰ in upper Maestrichtian sediments to –23.7‰ in basal Danian sediments (Saito et al., 1986), which is opposite to the change expected from depressed marine productivity. This deviation from the typical pattern is evidence that local conditions at this site might have differed from the general global situation.

Like the isotopic values, elemental C/N and N/H ratios of organic matter can provide information about the paleoenvironmental and depositional conditions at the K/T boundary. The C/N ratios from the coal layers and mudstones in the Raton Basin region remain between 45 and 60 (Schimmelmann and DeNiro, 1984), typical of vascular plant material. Neither C/N nor N/H values change significantly across the Raton K/T boundary, even though angiosperm and fern pollen contents of strata here and at Hokkaido indicate extensive devastation of forests at the end of the Maestrichtian (Tschudy et al., 1984; Saito et al., 1986).

PALEOENVIRONMENTAL SIGNIFICANCE

Consequences of Biotic Change on the Organic Contents of Sediments

The molecular information retained in sediments from around the K/T boundary has not yet provided incontrovertible geochemical corroboration of the biotic changes evident in the marine and continental fossil records. Microbial reworking has erased much of the geochemical record of the original biotic sources of organic matter in the samples that were analyzed. Further studies are needed. Isotope ratios of organic and inorganic carbon give tantalizing indications that changes in biotic productivity and populations may be preserved in other parts of the geochemical record.

Evidence of Differences in Paleoproductivity

Although none of the samples listed in Table 1 has a high concentration of organic carbon, the sediments from the ocean margin paleolocation at El Kef contain more organic matter than do locations with paleoenvironments separated from continents by deep channels—the Wombat and Exmouth plateaus and the Negev. The El Kef samples contain less carbonate than do most of the more open-marine sediments, recording continental contributions of clastic sediment components. The continental runoff probably also included nutrients, which would encourage marine productivity. In the modern ocean, coastal areas commonly have enhanced rates of productivity. In addition, land-derived organic matter would augment concentrations of organic carbon in sediments deposited in ocean margin paleoenvironments.

The generally low concentrations of organic matter present in sediments deposited at the three Tethyan margin locations are consistent with the hypothesis, inferred from the global inorganic carbon isotopic shift to lighter values, that marine productivity was depressed in both open-ocean and coastal locations during the early Danian (Zachos and Arthur, 1986; Zachos et al., 1989). An apparent enhancement of organic carbon production exists in early Danian samples from El Kef and the Negev; TOC levels are higher than in Maestrichtian samples. Carbonate concentrations, however, are lower in the early Danian rocks. Consequently, TOC is not diluted as much by carbonate sediment components in these samples. The diminished carbonate concentrations have been interpreted as possible evidence of depressed marine productivity (e.g., Keller, 1988a, 1988b).

Differences in Preservation of Organic Matter

The general dominance of microbial biomarkers in extracts of K/T boundary sediments indicates that organic matter was poorly preserved and was mostly remineralized by benthic microbes (Meyers and Simoneit, 1990). Little difference in the extent of remineralization was suggested by the biomarker contents of sediments deposited before or after the boundary in open marine settings. Sediments from ocean margin paleoenvironments have higher concentrations of organic carbon than do those from open ocean locations, and these sediments may contain an important component of detrital terrigenous material, which would resist microbial degradation more successfully than would marine organic matter. Biomarker distributions suggest that organic matter is less microbially reworked in Danian than in Maestrichtian sediments deposited on ocean margins (Simoneit and Beller, 1987). Impingement of an oxygen minimum layer on the continental slopes during a time of lowered sea level may have diminished bacterial activity during this time and improved preservation of sedimented organic matter.

Concentrations of organic carbon are often elevated in K/T boundary clay layers, reaching 5% in the boundary clay at El Kef (Keller and Lindinger, 1989). In this section, concentrations of organic carbon in the clay layers are highest close to the boundary and diminish as carbonate concentrations recover into the Danian. The inverse relationship between the concentrations of organic carbon and calcium carbonate suggests that dissolution of carbonates may have contributed to the enhanced organic carbon concentrations. Oxidation of marine organic matter is a major factor in the dissolution of carbonate sediments (Berger, 1973; Diester-Haass et al., 1986). The production of CO_2 from readily oxidized organic matter makes sediment pore waters more corrosive to calcium carbonate and at the same time consumes dissolved oxygen, improving preservation of the remaining organic matter.

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