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Preliminary Results of Bitumen and Whole-Rock Elemental Analyses of CRP-2/2A, Victoria Land Basin, Antarctica

R.M. Kettler* & E. Papastavros

Department of Geosciences, University of Nebraska-Lincoln, Lincoln, NE 68588-0340 - USA

**Corresponding author* (rkettler1@unl.edu)

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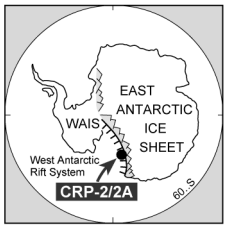
Abstract - Sediments and rocks recovered in CRP-2/2A coring operations contain relatively little organic matter (average TOC=0.28%) and very small amounts of solvent-soluble organic matter. Because Early Oligocene sediment included significant amounts of coal detritus, TOC values are higher in Early Oligocene rocks than in younger rocks. TOC values are highest in the fine-grained rocks deposited as part of the highstand systems tract (HST) and TOC values decrease from the bottom to the top of the HST. The TOC:TN ratios observed in the CRP-2/2A core typically exceed 10, and are strong evidence that the preserved organic matter comprises a mixture of detrital coal and aquatic organic matter. Solvent-soluble organic matter comprises autochthonous bitumen derived from aquatic organic matter, allochthonous bitumen derived from deeper sedimentary rocks, and recycled bitumen derived from coal detritus.



INTRODUCTION

Analysis of whole-rock elemental chemistry and the solvent-soluble organic matter present in the CRP-2/2A core was undertaken as part of the initial core characterisation effort. This effort included whole-rock measurements of the abundances of C and N, and extraction of solvent-soluble organic matter from these sediments and rocks. This preliminary information can be used to guide more detailed studies and to make some preliminary conclusions regarding the source and composition of organic matter preserved in these sediments and rocks. Although this work is intended only to provide preliminary information on the organic matter preserved in these sediments and rocks, there are two questions that are particularly relevant to the successful completion and scientific objectives of the Cape Roberts Project. 1)What is the source of the "oily overprint" noted in the CRP-2/2A core logs? 2)Can variations in the composition or amount of organic matter preserved in these rocks be related to the sequence stratigraphic interpretation developed by the Cape Roberts Science Team (1999; p. 67-71)?

The sediments and rocks penetrated by the CRP-2/2A core were deposited in environments which were influenced by glacial activity. Environments ranged from proximal glacimarine to shallow marine settings in which small amounts of detritus were ice-rafted to the CRP-2/2A drill site (Fig. 1) (Cape Roberts Science Team, 1999 p. 156-168). These different depositional environments may have led to differences in the amount, type, and provenance of organic matter deposited with the sediments. The waters in which glacimarine sediments and rocks are deposited can be quite productive. In particular, sea ice and ice-edge biological communities are very productive; thus, the carbon accumulation rate in portions of McMurdo Sound averages 45 mg $C \cdot m^{-2} \cdot d^{-1}$, a value similar to that observed in some highly productive anoxic basins (Dunbar et al., 1989). Although terrigenous plant debris is absent in the modern Antarctic environment, the terrigenous sediment input to the Ross Sea does contain an organic component. Coal is exposed throughout the Transantarctic Mountains and coal detritus has been described in previous samples of Cenozoic sediments and rocks from the Ross Sea (*e.g.* Lowery, 1989). Organic matter preservation may have been affected by depositional environment and sedimentation rate. Rapid sedimentation rates maximize preservation of aquatic organic matter whereas slow sedimentation rates permit extensive remineralization. Rapid deposition of terrigenous sediments may cause dilution of organic matter. Domack et al. (1989) demonstrated that the TOC content of sediment increases with distance from glacial ice fronts on the Antarctic Peninsula. Variations in the distance from a glacial ice front produced by glacial advance and retreat and the concomitant variations in eustatic sea level were considered to exert a first order control on the stratigraphic relationships observed in the CRP-2/2A core (Cape Roberts Scientific Team, 1999; p 67).

METHODS

All glassware and equipment used in sample preparation was washed in Micro solution and rinsed in purified water. This cleaning was followed by sequential rinses with 1% hydrochloric acid, methanol, and dichloromethane. Samples were collected at 5-10 m intervals and were intended to be representative of the major lithologies

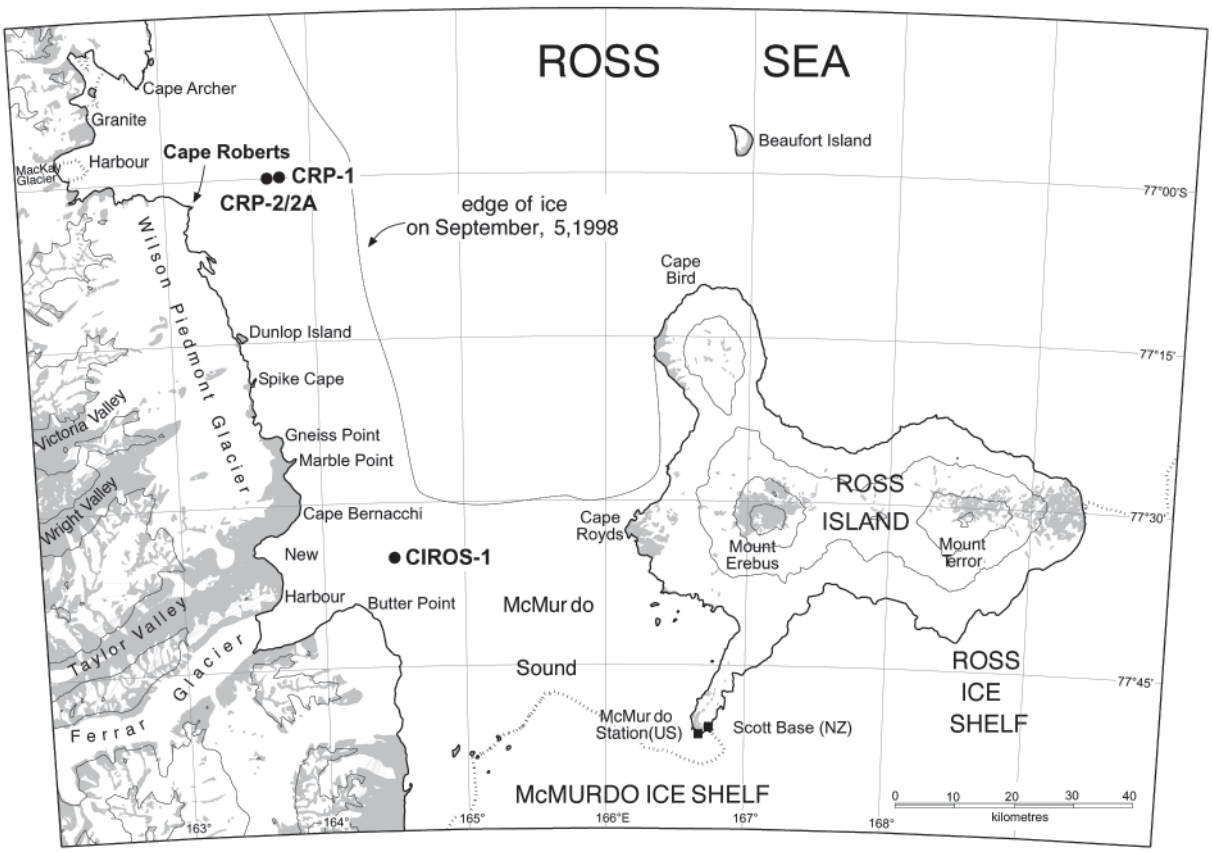


Fig. 1 - Map of the south west corner of the Ross Sea, showing the locations of the CRP-1 and CRP-2/2a drill sites (Taken from Cape Roberts Science Team, 1999).

penetrated by CRP-2/2A. The sampling was biased toward sediments and rocks with finer grain-sizes and well-preserved laminations. Although efforts were made to sample only the matrix of the diamictite and conglomerate units, some granule and pebble-sized clasts were included. An oily overprint was noted in the core logs at a number of intervals below 500 mbsf (Cape Roberts Science Team 1999; p. 58-60) and some of these intervals were sampled.

With the exception of those obtained from the rocks containing an oily overprint, samples were freeze-dried to remove excess water, ground and homogenised in a Spex 8000 Mixer/Mill, and stored in 15 ml glass vials. All glassware used in sample preparation was cleaned with a commercial detergent followed by sequential rinses with 1% hydrochloric acid, methanol, and dichloromethane. Three sets of elemental analyses were performed at the Crary Science and Engineering Center (CSEC) at McMurdo Station. These elemental analyses included measurements of total carbon (Tab. 1) as well as measurements of total organic carbon (TOC) using low-temperature vapour acidification and high-temperature vapour acidification. Although low-temperature vapour acidification effectively removes calcium carbonate (Hedges & Stern, 1984), siderite ($FeCO_3$) is present in these rocks (Baker &Fielding, 1999) and is considerably more refractory than calcite (Rosenbaum & Sheppard, 1986). The high-temperature vapour acidification (Cape Roberts Science Team, 1999; p. 103-105) was designed to attack siderite and yield more accurate values for TOC.

Bitumen was extracted from the samples using two techniques. Those samples that had been freeze-dried at CSEC were extracted for 24 hours with dicholoromethane. The dichloromethane solution was dried in a rotary evaporator and the solution was transferred to a 1 ml glass vial and dried to constant weight. Samples obtained from the portions of core described as having an oily overprint were crushed in the SPEX 8000 mixer/mill or ground and homogenised in a mortar and pestle, weighed and placed in a cellulose extraction thimble. The samples were extracted for 24 hours using an azeotropic mixture of toluene and methanol in a Soxhlet apparatus. The methanol/toluene solution was placed in a separatory funnel and a hydrophobic phase was isolated using water that had been purified in a Barnstead 4-stage purification system. The aqueous phase was transferred to another separatory funnel where a second hydrophobic phase was isolated using dicholoromethane. The hydrophobic phases were evaporated to dryness under partial vacuum using a rotary evaporator. The resulting liquid was transferred to a 1 ml glass vial using dichloromethane and dried to a constant mass.

Selected samples of bitumen were loaded on a 1H30 cm column packed with silica over alumina (3.8 g of each). Aliphatic, aromatic, and polar fractions were eluted with 25 ml aliquots of hexane, toluene, and methanol, respectively. Gas chromatography of selected aliphatic fractions was performed using a Varian 3300 gas chromatograph with a septum-equipped temperature-programmable on-column injector and flame ionisation

Tab. 1 - Values of total carbon (TC), total organic carbon determined by low temperature vapour acidification (TOC_{LT}), total organic carbon determined by high temperature vapour acidification (TOC_{HT}), and total nitrogen (TN) measured from samples of the CRP-2/2A core.

Depth	%TC	$\%TOC_{LT}$	$\%TOC_{HT}$	%TN	TOC_{HT}/TN
6.00-6.02	0.19±0.02	0.18±0.01	0.092±0.003	0.007±0.001	12
9.85-9.87	0.28±0.02	0.21±0.01	0.13±0.01	0.010±0.001	14
15.09-15.11	0.18±0.01	0.16±0.01	0.077±0.002	0.008±0.001	10
20.02-20.04	0.44±0.04	0.28±0.01	0.19±0.01	0.015±0.002	12
24.28-24.30	0.089±0.007	0.12±0.01	0.051±0.002	0.0068±0.0009	7
26.55-26.57	0.077+0.006	0.14+0.01	0.0087+ 0.0002	0.0068+0.0009	1
31.41-31.43	0.20±0.02	0.26±0.01	0.17±0.01	0.016±0.002	10
37.42-37.44	0.25±0.02	0.22±0.01	0.14±0.01	0.016±0.002	9
40.42-40.44	0.21±0.02	0.20±0.01	0.13±0.01	0.012±0.002	11
45.04-45.06	0.26±0.02	0.25±0.01	0.30±0.01	0.018±0.002	17
50.15-50.17	0.15±0.01	0.16±0.01	0.12±0.01	0.012±0.002	10
55.10-55.12	0.29±0.02	0.19±0.01	0.15±0.01	0.014±0.002	10
55.32-55.34	0.20±0.02	0.25±0.01	0.17±0.01	0.019±0.003	9
67.85-67.87	0.22±0.02	0.24±0.01	0.16±0.01	0.018±0.002	9
77.06-77.08	0.11±0.01	0.15±0.01	0.1±0.01	0.009±0.001	11
86.43-86.45	0.11±0.01	0.13±0.01	0.097±0.01	0.010±0.001	10
96.29-96.31	0.22±0.02	0.26±0.01	0.20±0.01	0.021±0.003	10
107.48-107.50	0.18±0.01	0.22±0.01	0.14±0.01	0.018±0.002	8
113.63-113.65	0.26±0.02	0.32±0.02	0.22±0.01	0.035±0.005	6
118.74-118.76	0.19±0.02	0.25±0.01	0.17±0.01	0.017±0.002	10
128.10-128.12	2.90±0.23	0.19±0.01	0.15±0.01	0.010±0.001	15
135.88-135.90	0.16±0.01	0.12±0.01	0.14±0.01	0.014±0.002	10
149.58-149.60	0.37±0.03	0.24±0.01	0.17±0.01	0.015±0.002	12
150.97-150.99	0.77±0.06	0.26±0.01	0.18±0.01	0.016±0.002	12
155.45-155.47	0.093±0.007	0.14±0.01	0.08±0.01	0.0056±0.0008	14
164.95-164.97	0.34±0.03	0.31±0.02	0.24±0.01	0.021±0.003	11
173.32-173.34	0.29±0.02	0.34±0.02	0.27±0.01	0.024±0.003	11
176.75-176.77	0.31±0.02	0.34±0.02	0.28±0.01	0.027±0.004	10
184.45-184.47	0.25±0.02	0.18±0.01	0.12±0.01	0.011±0.002	11
192.44-192.46	0.11+0.01	0.12+0.01	0.075+0.003	0.0069+0.0009	11
198.50-198.52	0.27±0.02	0.18±0.01	0.14±0.01	0.012±0.002	11
206.82-206.84	0.37±0.03	0.22±0.01	0.15±0.01	0.014±0.002	11
221.31-221.33	2.23±0.18	0.32±0.01	0.18±0.01	0.017±0.002	10
226.67-226.69	0.29±0.02	0.30±0.01	0.18±0.01	0.019±0.003	9
238.53-238.55	0.33±0.03	0.39±0.01	0.25±0.01	0.023±0.003	11
245.34-245-36	0.35±0.03	0.38±0.01	0.22±0.01	0.027±0.004	8
255.92-255.94	0.68±0.05	0.70±0.02	0.53±0.02	0.061±0.008	9
270.03-270.05	0.20±0.02	0.31±0.01	0.17±0.01	0.022±0.003	8
277.43-277.45	0.12±0.01	0.21±0.01	0.097±0.003	0.008±0.001	12
287.11-287.13	0.15±0.01	0.23±0.01	0.12±0.01	0.008±0.001	16
295.09-295.11	0.11±0.01	0.23±0.01	0.093±0.003	0.0066±0.0009	14
296.83-296.85	0.93±0.07	0.26±0.01	0.14±0.01	0.015±0.002	9
304.98-305.00	0.37±0.03	0.32±0.01	0.21±0.01	0.013±0.002	16
314.24-314.26	0.65±0.05	0.35±0.01	0.25±0.01	0.017±0.002	15
317.59-317.61	0.65±0.05	0.67±0.02	0.53±0.02	0.041±0.006	13
321.59-321.61	0.58±0.05	0.29±0.01	0.19±0.01	0.010±0.001	19
325.84-325.86	0.53±0.04	0.27±0.01	0.17±0.01	0.008±0.001	21
337.40-337.42	1.02±0.08	0.33±0.01	0.21±0.01	0.016±0.002	13
347.70-347.72	0.98±0.08	0.48±0.02	0.34±0.01	0.027±0.004	13
358.09-358.11	0.58±0.05	0.45±0.01	0.34±0.01	0.024±0.003	14
359.54-359.56	0.64±0.05	0.56±0.02	0.49±0.02	0.018±0.002	27
370.37-370.39	0.72±0.06	0.33±0.01	0.23±0.01	0.012±0.002	19
382.00-382.02	0.34±0.03	0.39±0.01	0.28±0.01	0.017±0.002	16
390.38-390.40	0.47±0.04	0.56±0.02	0.39±0.01	0.026±0.004	15
404.01-404.03	0.34±0.03	0.28±0.01	0.21±0.01	0.008±0.001	26
412.56-412.58	0.42±0.03	0.42±0.01	0.31±0.01	0.018±0.002	17
426.36-426.38	0.28±0.02	0.35±0.01	0.23±0.01	0.009±0.001	26
437.73-437.75	0.49±0.04	0.32±0.01	0.21±0.01	0.015±0.002	14
450.26-450.28	1.10±0.09	0.49±0.02	0.38±0.01	0.029±0.004	13
459.04-459.06	0.61±0.05	0.66±0.02	0.54±0.02	0.045±0.006	12
465.49-465.51	0.59±0.05	0.56±0.02	0.42±0.02	0.041±0.006	10
467.51-467.53	2.02±0.16	0.47±0.02	0.36±0.01	0.027±0.004	13
477.34-477.36	0.46±0.04	0.32±0.01	0.21±0.01	0.017±0.002	13
481.29-481.31	0.58±0.05	0.41±0.01	0.26±0.01	0.020±0.003	13
486.90-486.92	0.47±0.04	0.31±0.01	0.17±0.01	0.014±0.002	12
498.72-498.74	0.072±0.006	0.16±0.01	0.04±0.01	0.008±0.001	5
500.42-500.44	0.18±0.01	0.26±0.01	0.14±0.01	0.009±0.001	16
523.45-523.47	0.75±0.06	0.59±0.04	0.37±0.02	0.021±0.003	18
583.46-583.48	0.56±0.04	0.60±0.04	0.39±0.02	0.026±0.004	15
623.21-623.23	0.62±0.05	0.65±0.04	0.44±0.03	0.023±0.003	19

detector (FID). The initial injection temperature was 65°C with an immediate increase to 350 °C at 100°C/min. The column used was a J&W fused silica capillary column (30mx0.25mm) coated with DB-5 (0.25µm), and programmed from 100 to 350°C at a rate of 6°C/min followed by a 3 min 20 s isothermal hold. The FID temperature was held constant at 350°C. Individual n-alkanes were identified using an external standard.

RESULTS AND DISCUSSION

The sediments and rocks penetrated by the CRP-2/2A drill hole contain little organic carbon. All samples prepared using the high-temperature vapour acidification technique contain less than 0.54% TOC (Tab. 1). The average TOC value for these sediments and rocks is 0.21%; a value that is well below the average proposed for marine shales (0.99%) by Hunt (1972) and is even less than average TOC values proposed by Hunt (1972) for marine sandstones (0.28%). The TOC:TN (total organic carbon:total nitrogen) ratios are high throughout the section. Organic matter with TOC:TN ratios significantly greater than 10 is derived largely from land plants and coals, whereas aquatic organisms have much lower TOC:TN ratios (Bordovskiy, 1965). The high TOC:TN ratios observed in the CRP2/2A core (Tab. 1; Fig. 2) are strong evidence that the preserved organic matter comprises a mixture of detrital coal and aquatic organic matter. The organic geochemical data are in agreement with the report that the organic residue isolated for palynological analysis consisted largely of coal fragments (Cape Roberts Science Team, 1999; p. 122).

The TOC content of these sediments and rocks changes at three scales. 1)Samples of Early Oligocene rocks contain more organic carbon than do samples obtained from younger rocks or sediments. The average TOC values for the Early Oligocene and younger rocks are 0.27% and 0.17%, respectively. The elevated TOC values in the Early Oligocene rocks correspond to increased TOC:TN ratios (Fig. 2). The Early Oligocene rocks contain more detrital coal and fewer phytoclasts than do the younger rocks (Cape Roberts Science Team, 1999; p. 122-126). 2)Sediments and rocks assigned to highstand systems tracts contain more organic carbon than other rocks. The Cape Roberts Science Team described 25 sequences in the CRP2/2A core (Fig. 2) and fine-grained rocks were assigned to the highstand systems tract (HST) (Cape Roberts Science Team, 1999; p. 69). Biogenic detritus comprises a greater proportion of these fine grained rocks: diatom abundances are typically higher in the fine grained rocks than in the coarser sediments or rocks (Fig. 2). 3)The TOC values decrease from the base to the top of the HST. This behaviour is best observed in sequences with thicker sections of fine-grained rocks, such as Sequences 9, 10, 11, and 19. The base of many of the HSTs consists of fossiliferous shell-beds that are considered to be condensed sections (Cape Roberts Science Team, 1999; p. 69). The higher TOC values apparently correspond to periods of clastic sediment starvation, whereas the upward decrease in TOC values above the condensed sections reflects increasing proportions of clastic input and dilution of the biogenic organic debris. Although not previously described in glacimarine sections these relations have been described in a variety of other settings (*e.g.* Sageman et al., 1998; Creaney & Passey, 1993).

All samples contain little solvent-soluble organic matter (bitumen). The dichloromethane-bitumen solutions obtained by the Soxhlet extraction of those samples collected from Late Oligocene and younger rocks (above 325 mbsf) are colourless and the mass of bitumen extracted is typically 0.001 g or less (Fig. 3). Samples from Early Oligocene rocks typically yield greater amounts of bitumen and samples obtained from rocks exhibiting an oily overprint yield as much as 0.01 g (Fig. 3). Some of the solvent-bitumen solutions derived from these latter rocks exhibit a pale yellow tint. The amounts of bitumen recovered from all samples are small, however, compared to the amounts that are typically recovered from petroleum source rocks (Tissot & Welte, 1978). On the other hand, the bitumen ratios (mg bitumen/g organic carbon) calculated for some of the samples are very high. In some cases this reflects the error inherent in measuring precisely the amount of bitumen recovered and dividing that value by the small amount of organic carbon in the sample. For some of the samples collected below 450 mbsf, it reflects the fact that much of the organic matter occurs as bitumen. It is unlikely that Rock-Eval pyrolysis of these or other CRP-2/2A samples would yield useful data: Rock-Eval pyrolysis data are very difficult to interpret when the samples analyzed have very low TOC and bitumen contents (Peters, 1986).

The bitumen present in these rocks is derived from three sources. 1) Autochthonous bitumen is derived from organic matter that was biosynthesised in the water column during deposition of the sedimentary rocks penetrated by the CRP-2/2A core. 2) Allochthonous bitumen is that solvent-soluble organic matter that was generated during catagenesis of organic matter elsewhere in the sedimentary section. 3) Recycled bitumen is solvent soluble organic matter that was derived from detrital coal.

Examples of material interpreted to be autochthonous and allochthonous bitumen were obtained from two samples collected at 514.25-514.27 mbsf. This section of core comprises sandstone with interbedded mudstone. The sandstone contains material described as bituminous residue. If the Abituminous residue represents allochthonous bitumen, then the mudstone should contain little of this material. A chromatogram of the aliphatic hydrocarbon fraction obtained from the mudstone lithology (Fig. 4) is dominated by n-alkanes with 21 or fewer carbon atoms. The C_{14} to C_{21} n-alkanes are typically produced by aquatic organisms (Brassell et al., 1978). The presence of the C_{14} to C_{21} n-alkanes combined with the relative absence of n-alkanes longer than C_{21} is evidence for the interpretation that this mudstone contains autochthonous bitumen. Although the aliphatic fraction of the bitumen obtained from the sandstone also contains some of the C_{14}-C_{17} n-alkanes, a homologous series of longer chain n-alkanes (C_{25}-C_{32}) is also present. These longer chain n-alkanes are typically derived from higher terrestrial plants (Caldicott & Eglinton, 1973). Land plant waxes typically have a strong odd-even carbon number predominance; the

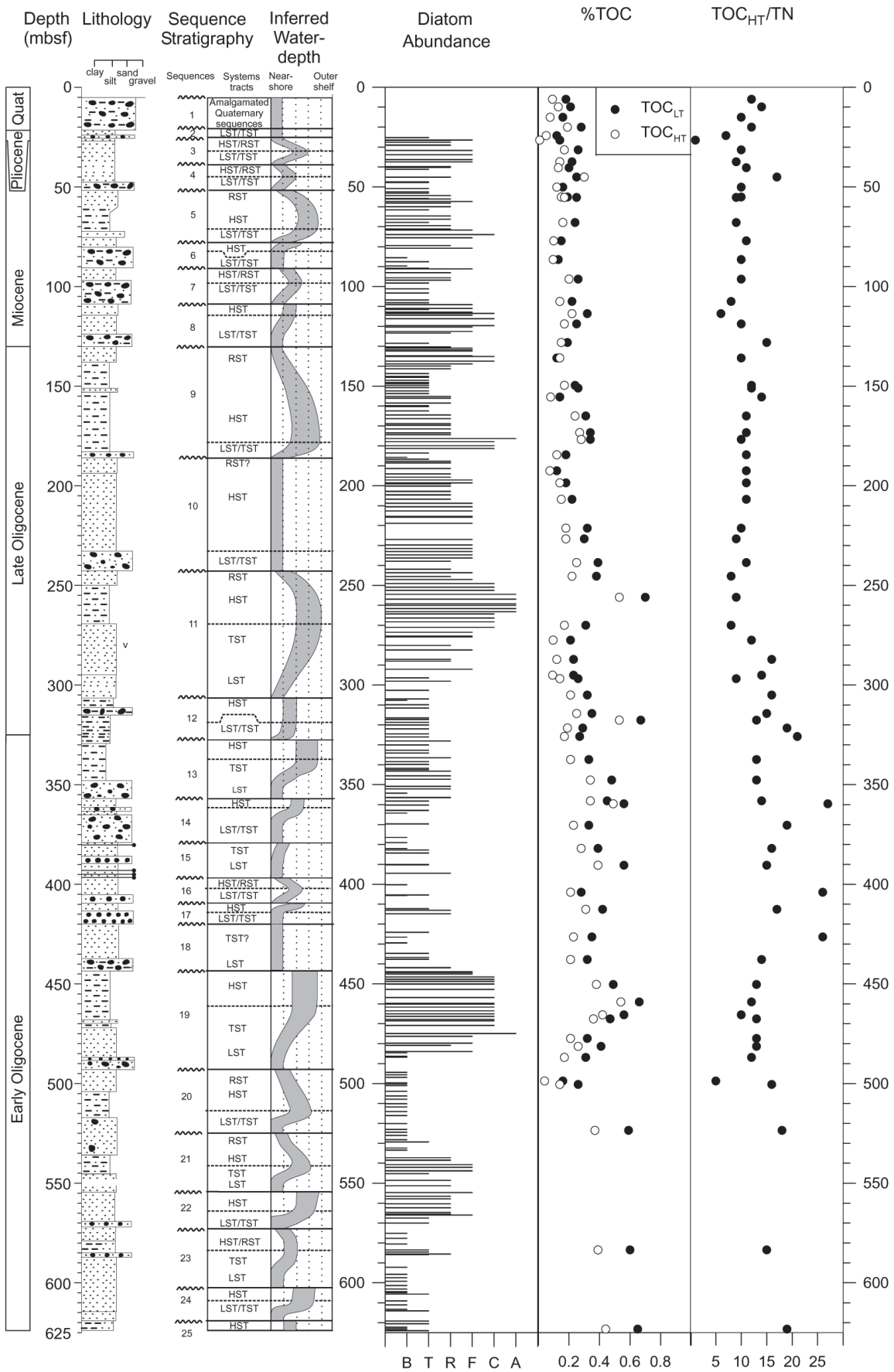


Fig. 2 - Comparison of values of total organic carbon determined using low temperature (TOC_{LT}) and high temperature (TOC_{HT}) vapour acidification, and TOC_{HT}/TN compared with the lithology of, the abundance of diatoms in, and sequence stratigraphic and bathymetric interpretations for the CRP-2/2A core (Cape Roberts Science Team, 1999; p. 68). Systems tract interpretations are lowstand systems tract (LST), transgressive systems tract (TST), regressive systems tract (RST), and highstand systems tract (HST). Diatom abundances are described as barren (B), trace (T), rare (R), few (F), common (C), and abundant (A) (Cape Roberts Science Team, 1999; p, 110).

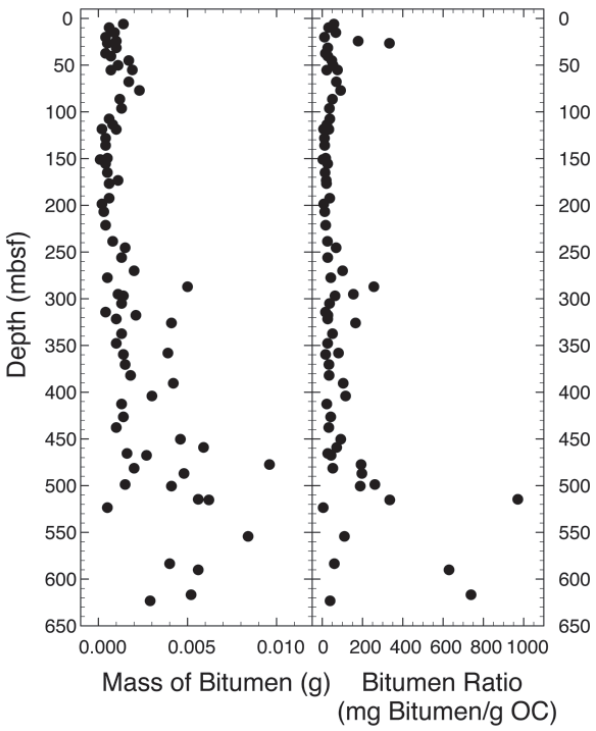


Fig. 3 - Relationships between the mass of bitumen extracted from samples of CRP-2/2A core and depth, and the bitumen ratio calculated for samples CRP-2/2A core and depth.

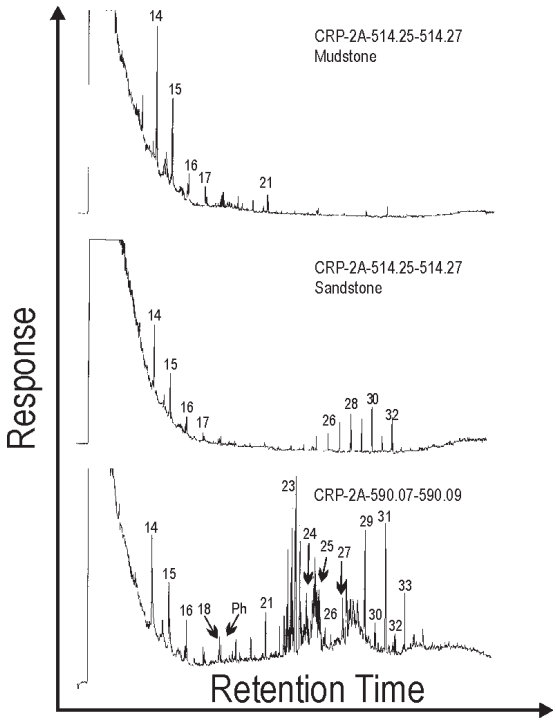


Fig. 4 - Gas chromatograms of the aliphatic fractions of bitumen extracted from three samples of CRP-2A core. Selected n-alkanes are identified by the number of carbon atoms in the molecule (*i.e.* 14 designates n-C_{14}) whereas phytane (an acyclic isoprenoid) is designated by Ph.

absence of a strong odd-even predominance in this suite of long chain n-alkanes (Fig. 4) is strong evidence that these compounds have experienced a thermal history more severe than would be expected from burial to 514 mbsf. There is little evidence for significant numbers of terrestrial plants in the source area for these sedimentary rocks: no plant detritus is observed in the core and palynological work indicates very sparse vegetation (Cape Roberts Science Team, 1999; p. 136). The long chain n-alkanes detected in the sandstone and, by extension, the material that comprises the bituminous residue is therefore considered to be allochthonous.

Much of the bitumen extracted from sample CRP-2A-590.07-590.09 is interpreted to be recycled bitumen. This sample was obtained from a muddy, fine sandstone that contained abundant coal detritus. Although the chromatogram of the aliphatic fraction of bitumen derived from this sample (Fig. 4) has the same suite of C_{14}-C_{21} n-alkanes observed the other samples, it also contains a homologous series of C_{23}-C_{33} n-alkanes. These longer chain n-alkanes differ from those identified in CRP-2A-514.25-514.27 in that the long chain n-alkanes in CRP-2A-590.07-590.09 have a strong odd-even predominance: among the most prominent n-alkane peaks in this latter sample are n-C_{29}, n-C_{31} and n-C_{33}. The presence of the strong odd-even predominance in the long chain n-alkanes is strong evidence that these compounds were derived ultimately from terrestrial land plants. It is likely that the immediate source of these long-chain n-alkanes is coal detritus within the sedimentary rocks: there is little evidence of terrestrial vegetation in the CRP-2/2A core and coal detritus is abundant. Coals will yield small amounts of bitumen in which the C_{23}-C_{31} n-alkanes have a strong odd-even predominance even at moderate levels of thermal maturity (Radke et al., 1980). The recycled bitumen apparently has a lower thermal maturity than does the allochthonous bitumen: the strong odd-even predominance observed in the recycled bitumen is not observed in the allochthonous bitumen.

The presence of allochthonous bitumen with a thermal maturity exceeding that of autochthonous or recycled bitumen requires generation and secondary migration of bitumen within the sedimentary section near Cape Roberts. Previous work on sedimentary rocks obtained from the CIROS-1 core (Fig. 1) also concluded that bitumen had been generated and undergone secondary migration in the rocks below the Ross Sea (Cook & Woolhouse, 1989).

CONCLUSIONS

The rocks and sediments penetrated by the CRP-2/2A contain little organic carbon. The data collected in this initial core characterisation effort support strongly the conclusion made in the initial report (Cape Roberts Science Team, 1999; p. 106) that much of the organic matter in these rocks and sediments is a mixture of detrital coal and aquatic organic matter. Solvent-soluble organic matter in these rocks comprises a mixture from three sources. 1)□Autochthonous bitumen is ubiquitous and can be recognized by the presence of C_{14}-C_{21} n-alkanes. 2)□Allochthonous bitumen is characterised by the presence C_{26}-C_{32} n-alkanes that lack an odd-even predominance. Its presence is direct evidence for bitumen generation and

migration in the Ross Sea environs. 3)Recycled bitumen contains a suite of C_{23}-C_{33} n-alkanes with a strong odd-even predominance and is derived from coal detritus.

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