22. PRELIMINARY ORGANIC ANALYSES OF THE DEEP SEA DRILLING PROJECT CORES, LEG 10¹

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ABSTRACT

The solvent soluble organic matter extracted from two core samples from DSDP Leg 10 was analyzed using GC and GC/MS techniques. The samples from Site 90 was of Early Miocene age and the extract consisted mainly of normal alkanes, $C_n H_{2n+2}$, ranging from n = 14 to n = 33, with an odd over even predominance and maximum abundance above C24. Approximately eight per cent of the extract consisted of normal carboxylic acids, $C_n H_{2n} O_2$, ranging from n = 13 to n = 31, with an even over odd predominance and palmitic and lignorceric acids most abundant. The sample from Site 92 was of Late Pleistocene age and the extract consisted mainly of normal alkanes $C_n H_{2n+2}$ with n =11 to n = 33, with an odd over even predominance and maximum abundance above C24. Again, approximately 10 per cent of the extract consisted of normal carboxylic acids, $C_n H_{2n} O_2$, ranging from n = 13to n = 31, with an even over odd predominance and lignoceric acid most abundant. In this sample, minor amounts of terpenoidal compounds were detected. Organic contamination was minor for both samples and consisted mainly of butyl esters from the core tubes. The bulk of the solvent soluble organic matter in these samples (especially Site 92) is of terrigenous origin, probably from turbidite runoff from the Mississippi Cone.

INTRODUCTION

Leg 10 of the Deep Sea Drilling Project was conducted in the Gulf of Mexico. Only two samples were received at this laboratory for analysis. The sample from Site 90 (10-90-7-2, 40-140), drilled within the deep basin in a water depth of 3,713 meters, was recovered from 353 meters below the sea bed. It is of Early Miocene age (approximately 20×10^6) and consists mainly of silts with a low fossil content. The sample from Site 92 (10-92-5-4, 50-150), drilled just above the Sigsbee Scarp in a water depth of 2,573 meters, was recovered from 178 meters below the sea bed. It is of Late Pleistocene age (< 10^6) and consists of gray silty clay.

EXPERIMENTAL PROCEDURES

Gas chromatographic (GC) analyses were carried out using a Perkin-Elmer Model 900 gas chromatograph fitted with a flame ionization detector and operating under the conditions stated in the respective figure legends. Analyses using gas chromatography-mass spectrometry were carried out on a modified Perkin-Elmer Model 270 GC/MS linked on-line to an XDS Sigma 2 computer (Smith et al., 1971). The GC conditions used in the GC/MS analyses are cited in the respective figure legends, and the mass spectrometric and computer operating parameters are as reported (Smith et al., 1971).

All solvents used, e.g., benzene, methanol, and *n*-heptane, were Mallinckrodt Nanograde quality. Benzene and methanol were redistilled prior to use.

The samples were extracted with benzene and methanol (3:1) in a Soxhlet apparatus for eight days. The heptane-diethyl ether soluble components of these extracts were divided into acidic and neutral fractions. The methods are essentially the same as discussed in earlier reports (Simoneit and Burlingame, 1971 a and b; and 1972 a), where a flow diagram of this procedure is given.

RESULTS

The salient analytical results and yields for the samples are listed in Table 1.

Sample 10-90-7-2 (40-140)

The GC trace of the heptane-ether soluble neutral fraction is given in Figure 1a and the GC/MS sum plots and individual scans are shown in Figure 2. The GC/MS results, listed in Table 2, indicate this mixture is composed predominantly of normal alkanes, C_nH_{2n+2} , ranging from n = 16 to n = 33. In the GC data, the range is from C $_{14}H_{30}$ to $C_{33}H_{68}$. The higher weight alkanes, above C_{24} H_{50} , are much more abundant than the lower weight alkanes. There is an odd over even predominance which is especially noticeable above *n*-tetracosane. In the mass spectra of normal alkanes, m/e 57 is the base peak. The sum plot of the m/e 57 ions (Figure 2b) contains most of

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TABLE 1 Salient Results, Samples 10-90-7-2 (40-140) and 10-92-5-4 (50-150)

Sample	Organic Carbon ^a (%)	Heptane/ Ether Soluble	Acid Fraction (%)	Neutral Fraction (%)
10-90-7-2 (40-140)	0.5 1.9	90 ppm (0.009%)	8.3	91.7
10-92-5-4 (50-150)	0.8 2.2	300 ppm (0.03%)	10	90

^aData provided by DSDP Staff, Scripps Institution of Oceanography, University of California at San Diego, and Chemistry Department, Woods Hole Oceanographic Institution.

the intense peaks found in the total ionization sum plot (Figure 2a). However, that plot has a much higher background level than the m/e 57 plot. This indicates that there are many compounds in the mixture which are not resolved on the gas chromatographic column. On the basis of the GC/MS data, the most abundant species in this extract is dibutyl azelate, $C_{17}H_{32}O_4$. This and four other dibutyl esters found in the neutral fraction are contamination from the core tubes. These compounds were also detected in other DSDP core samples (Simoneit and Burlingame, 1972 a; Simoneit et al., 1972). Phthalate ester contamination is minor. One phthalate ester has been indentified in scan 117 by its base peak at m/e 149 which has the composition $C_8H_5O_3$ (Structure 1).



I m/e 149, C₈H₅O₃

The most abundant ion in normal saturated acid ester mass spectra is m/e 74. The sum plot of the m/e 74 ions (Figure 2c) indicates only minor amounts of esters in the neutral fraction. The series, $C_nH_{2n}O_2$, ranges from n = 15to n = 31, with n = 25 most abundant. Methyl lignocerate ($C_{23}H_{30}O_2$) is also the most abundant species in the acid ester fraction of this core. The presence of methyl esters in the neutral fraction can probably be attributed to esterification of free fatty acids with methanol during Soxhlet extraction by catalysis of the slightly acidic clay content of the sediment.

The m/e 191 sum plot has no definite peaks above the background noise level which indicates that there are no terpenoidal components which exhibit a m/e 191 fragment ion in this mixture.

The GC trace of the acid fraction (as methyl esters) is shown in Figure 1b and the GC/MS data of this fraction are shown in Figure 3. The GC/MS results, listed in Table 3, indicate that this sample is mainly a series of saturated normal esters, $C_nH_{2n}O_2$. In the GC/MS data, the esters range from n = 13 to n = 31 and in the GC data the range



Figure 1a. Gas chromatogram of the heptane-ether soluble neutral extract from Sample 10-90-7-2(40-140). (Conditions: 10 ft. × 1/8 in. stainless steel column packed with 3% OV-1 on 100-200 mesh gaschrom Q, programmed from 100 - 275°C at 8°/min and using helium carrier gas at 60 ml/min.) The arrows indicate the relative positions at which n-pentadecane, n-tetracosane and n-octacosane elute under the same chromatographic conditions.



Figure 1b. Gas chromatogram of the heptane-ether soluble acid ester extract from Sample 10-90-7-2(40-140). (GC conditions as cited in Figure 1a.) The arrows indicate the relative positions at which methyl n-pentadecanoate, methyl n-heptadecanoate, and methyl lignocerate elute under the same chromatographic conditions.

is from n = 13 to n = 33. Methyl palmitate ($C_{17}H_{34}$ O_2) and methyl lignocerate ($C_{25}H_{50}O_2$) are the most abundant esters. The even-numbered acids predominate over the odd.

All the peaks found in the total sum plot are also found in the sum plot of the m/e 74 ions (McLafferty rearrangement). This indicates that all the resolved peaks are normal esters. Seven peaks in the m/e 74 sum plot (Figure 3d) are not normal esters. In the spectrum of each of these

Spectrum at Scan Number (see Figure 2a)	Compound Name	Molecular Weight and Composition		
35	biphenyl	154	C12H14	
72	n-hexadecane	226	C16H34	
75	dibutyl glutarate*	244	C13H2404	
80	branched alkane	198	10 21	
86	n-heptadecane	240	C17H36	
92	dibutyl adipate*	258	C14H2604	
95	unknown	346		
101	n-octadecane	254	C18H38	
105	unknown ketone	240	C16H32O	
114	n-nonadecane	268	C19H40	
117	phthalate ester			
120	dibutyl suberate*	286	C16H30O4	
128	<i>n</i> -eicosane	282	C20H42	
132	dibutyl azelate*	300	C17H32O4	
140	n-heneicosane	296	C21H44	
152	n-docosane	310	C22H46	
155	dibutyl hendecanedioate*	328	C19H36O4	
164	n-tricosane	324	C23H48	
175	n-tetracosane	338	C24H50	
186	n-pentacosane	352	C25H52	
197	n-hexacosane	366	C26H54	
211	n-heptacosane	380	C27H56	
227	n-octacosane	394	C28H58	
246	n-nonacosane	408	C29H60	
270	n-triacontane	422	C30H62	
304	n-hentriacontane	436	C31H64	
345	n-dotriacontane	450	C32H66	
396	n-tritriacontane	464	C33H68	

TABLE 2 Major Components of the Neutral Fraction from Sample 10-90-7-2 (40-140)

*Core Tube Contamination

peaks, the m/e 55 ion (Figure 3b) is more intense than the m/e 57 ion (Figure 3c) indicating the esters may be unsaturated.

The m/e 191 sum plot has a low intensity for the most abundant peak compared to the intensity at the most abundant ester peak. The m/e 191 sum plot contains no distinct peaks above the background noise; therefore, there appear to be no terpenoidal compounds which form m/e 191 ions present in this mixture. Contamination from phthalate esters, as indicated by the m/e 149 sum plot, appear to be very small.

Sample 10-92-5-4 (50-150)

The GC trace of the neutral fraction is shown in Figure 4a. The GC/MS data of the neutral fraction are shown in Figures 5 and 6. The GC/MS results, listed in Table 4, indicate that the major components are normal alkanes, C_nH_{2n+2} , ranging from n = 11 to n = 31 with *n*-heptaco-sane ($C_{27}H_{56}$) and *n*-nonacosane ($C_{29}H_{60}$) as the most abundant components. The GC data indicate a range from n = 15 to n = 33. The odd-numbered alkanes are predominant over the even. For the major peaks, the m/e 57 sum plot intensities (Figure 5c) are generally three times greater than the m/e 55 sum plot intensities (Figure 5b),

which indicates that the major peaks are all saturated hydrocarbons. There are two peaks (at scans 84 and 113) with m/e 55 peaks more intense than m/e 57 peaks. Scan 84 has a molecular ion at m/e 210 and an intense m/e 58 peak. It is possible laboratory contaminant, since it was found in another sample of this laboratory (Welch et al., unpublished results). Scan 113 fits the fragmentation pattern of dibutyl azelate, a core tube contaminant (Simoneit et al., 1972).

There does not appear to be any contamination from phthalate esters as indicated by the m/e 149 sum plot. A series of normal acid esters is indicated by the m/e 74 sum plot (figure 5d). From an intensity correlation of the data it can be concluded that the esters constitute approximately 1 per cent of the alkanes. The m/e 191 sum plot (Figure 5e) indicates a peak at scan 232. The scan 231 spectrum (Figure 6b shows scan 232, a mixture) has a molecular ion at m/e 384, loss of a methyl radical to m/e 369, and a peak at m/e 191 (Structure II or III). The likely compositions for this compound



are $C_{28}H_{48}$ (possible Structure IV) or $C_{28}H_{48}$ (a pentacyclic triterpane).

TABLE 3
Major Components of the Heptane-Ether Soluble Acid Ester
Fraction From Sample 10-90-7-2 (40-140)
as Determined by GC/MS and GC

Spectrum at Scan Number (see Figure 3a)	Compound Name	Molecular Weight and Composition	
54	methyl laurate	214	C13H26O2
70	C14 methyl branched ester	242	C15H30O2
85	methyl myristate	242	C15H30O2
100	methyl pentadecanoate	256	C ₁₆ H ₃₂ O ₂
113	methyl palmitate	270	C ₁₇ H ₃₄ O ₂
126	octadecane	254	C18H38
140	methyl stearate	298	C19H38O2
151	methyl nonadecanoate	312	C20H40O2
163	methyl arachidate	326	C21H42O2
173	methyl heneicosanoate	340	C22H44O2
186	methyl behenate	354	C23H46O2
196	methyl tricosanoate	368	C24H48O2
206	methyl lignocerate	382	C25H50O2
215	methyl pentacosanoate	396	C26H32O2
225	methyl cerotate	410	C27H54O2
257	methyl octacosanoate	438	C29H58O2
270	methyl triacontanoate	466	C ₃₁ H ₆₂ O ₂



Figure 2. GC/MS data for the neutral fraction from Sample 10-90-7-2(40-140). (a) Total ionization sum plot; (b) m/e 57 sum plot; (c) m/e 74 sum plot; (d) Mass spectrum scan 101; (e) Mass spectrum scan 105.

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10 050 050 040 050 060 070 060 090 100 110 150 130 140 150 160 170 160 190 200 210 250 230 240 250 260 270 260 280 300 310 350 350 340 350 SUMM Ν/E=55 36 MRX TL = 21:53







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Figure 3. GC/MS data for the acid ester fraction from Sample 10-90-7-2(40-140). (a) Total ionization sum plot; (b) m/e 55 sum plot; (c) m/e 57 sum plot; (d) m/e 74 sum plot; (e) Mass spectrum scan 114.



IV m/e 384, C27H440

The scan 233 spectrum exhibits a molecular ion at m/e 410, loss of a methyl radical to m/e 395, and an intense m/e 191 peak (Structure II or III). There is also a minor peak at m/e 426, which could be an oxygenated species of the compound of molecular weight 410. The likely composition for m/e 410 is $C_{30}H_{50}$ (e.g., possible Structure V). The m/e 77 sum plot exhibits



V m/e 410, C30H50

no distinct peaks above the background indicating this sample contains no aromatic compounds.

The GC trace of the acid fraction (as methyl esters) is shown in Figure 4b and the GC/MS data appear in Figures 7 and 8. The GC/MS results, listed in Table 5, indicate that the sample consists predominantly of a series of normal saturated methyl esters, C_nH_{2n}O₂. In the GC/MS data, the esters range from n = 13 to n = 31 and in the GC data the range is n = 15 to n = 31. Methyl lignocerate $(C_{25}H_{50}O_2)$ is the most abundant ester. The even-numbered acids predominate over the odd. C₁₅ and C₁₇ methyl branched acid esters are found in scans 74 and 102 respectively. The mass spectra of the branched C15 acid ester and methyl n-pentadecanoate are shown in Figures 8a and 8b. Since there are no intense fragment ions at higher mass of scan 74, the branching is likely to be near the end of the chain, perhaps in the iso or anteiso position. The m/e 74 sum plot (Figure 7d) closely resembles the sum plot of the total ionization current (Figure 7e). All of the intense peaks coincide. However, in the higher molecular weight regions of the total ion sum plot (above methyl triacontanoate), a great deal more background, not present in the m/e 74 plot, is found.

A comparison of the intensities of the m/e 55 sum plot (Figure 7b) and the m/e 57 sum plot (Figure 7c) reveals a close similarity in the lower molecular weight range (below methyl triacontanoate) and more intense m/e 55 ions at higher molecular weights, indicating the presence

TABLE 4 Major Components of the Neutral Fraction From Sample 10-92-5-4 (50-150) as Determined by GC/MS and GC

Spectrum at Scan Number (see Figure 5a)	Compound Name	Molecular Weight and Composition	
14	n-undecane	156	C11H24
31	n-tridecane	184	C13H28
48	n-pentadecane	212	C15H32
65	n-heptadecane	240	C17H36
80	n-octadecane	254	C18H38
84	unknown*	210	C14H260
94	n-nonadecane	268	C19H40
108	n-eicosane	282	C20H42
113	dibutyl azelate [†]	300	C17H32O4
120	n-heneicosane	296	C21H44
132	n-docosane	310	C22H46
144	n-tricosane	324	C23H48
156	n-tetracosane	338	C24H50
166	n-pentacosane	352	C25H52
176	n-hexacosane	366	C26H54
186	n-heptacosane	380	C27H56
196	n-octacosane	394	C28H58
205	n-nonacosane	408	C29H60
219	pentacyclic triterpane unknown	398	C ₂₉ H ₅₀
229	n-hentriacontane	436	C31H64
231	pentacyclic triterpane unknown	384	C28H48
233	pentacyclic triterpane unknown	410	C ₃₀ H ₅₀

*Possible laboratory contaminant

[†]Core tube contamination

of unsaturated compounds in the higher mass regions which were not resolved by the gas chromatographic column. There appear to be no aromatic compounds in this mixture (m/e 77 sum plot at background levels) and contamination by phthalate esters (based on the m/e 149 sum plot) appears to be negligible.

There are a few distinct peaks in the m/e 191 sum plot (Figure 7e). The peaks at lower scan numbers are unresolved from the large ester peaks, but scans 371 and 432 are good spectra of terpenoidal compounds. The scan 371 spectrum exhibits a molecular ion at m/e 468 and fragment ions at m/e 453, 369, 249, 191, 95, and 81 as well as low intensity alkane fragmentation. The chemical separation technique for this fraction strongly suggests that these two compounds are triterpenoidal acids. A likely composition fitting for m/e 468 is $C_{31}H_{48}O_{3}$ with the possible example Structures VI and VII. Structure VI is a compound related to oleanolic acid and Structure VII is an oxidized form derived from B-boswellic acid. The



scan 432 spectrum (Figure 8d) exhibits a molecular ion at m/e 484, with loss of a methyl radical to m/e 469; fragment ions at m/e 369, 263, 191, 95, and 81; and low intensity alkane fragmentation. A likely composition fitting for m/e 484 is $C_{31}H_{48}O_4$ with the possible example Structure VIII, a compound related to glycyrrhetinic acid.



Figure 4a. Gas chromatogram of the heptane-ether soluble neutral extract from Sample 10-92-5-4(50-150). (Conditions as cited in Figure 1a.)



Figure 4b. Gas chromatogram of the heptane-ether soluble acid ester extract from Sample 10-92-5-4(50-150). (Conditions as cited in Figure 1a.)

TABLE 5
Major Components of the Heptane-Ether Soluble Acid Ester
Fraction from Sample 10-92-5-4 (50-150) as
Determined by GC/MS and GC

Spectrum at Scan Number (see Figure 7a)	Compound Name	Mole and	ecular Weight Composition	Figure Reference
34	methyl laurate	214	C13H2602	-
49	methyl tridecanoate	228	C14H28O2	-
64	methyl myristate	242	C15H30O2	-
74	C ₁₅ methyl branched ester	256	C ₁₆ H ₃₂ O ₂	8a
78	methyl pentadecanoate	256	C16H32O2	8b
92	methyl palmitate	270	C17H34O2	-
102	C ₁₇ methyl branched ester	284	С18Н3602	
106	methyl margarate	284	C18H36O2	\simeq
118	methyl stearate	298	C19H38O2	
130	methyl nonadecanoate	312	C ₂₀ H ₄₀ O ₂	-
142	methyl arachidate	326	C21H42O2	-
153	methyl heneicosanoate	340	C22H44O2	
163	methyl behenate	354	C23H46O2	-
173	methyl tricosanoate	368	C24H48O2	-
182	methyl lignocerate	382	C25H50O2	-
191	methyl pentacosanoate	396	C26H52O2	
199	methyl cerotate	410	C27H54O2	-
210	methyl heptacosanoate	424	C28H56O2	-
223	methyl octacosanoate	438	C29H58O2	8c
239	methyl nonacosanoate	452	C30H60O2	~
259	methyl triacontanoate	466	C31H62O2	-
371	pentacyclic triterpane acid unknown	468	C ₃₁ H ₄₈ O ₃	-
432	pentacyclic triterpane acid unknown	484	$C_{31}H_{48}O_4$	8d



Figure 5. GC/MS data of the neutral fraction from Sample 10-92-5-4(50-150). (a) Total ionization sum plot; (b) /6e 55 sum plot; (c) m/e 57 sum plot; (d) m/e 74 sum plot; (e) m/e 191 sum plot.



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Figure 6. GC/MS data of the neutral fraction from Sample 10-92-5-4(50-150). (a) Mass spectrum scan 94; (b) Mass spectrum scan 232.





Figure 7. GC/MS data for the acid ester fraction from Sample 10-92-5-4(50-150). (a) Total ionization sum plot; (b) m/e 55 sum plot; (c) m/e 57 sum plot; (d) m/e 74 sum plot; (e) m/e 191 sum plot.

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CONCLUSIONS

The solvent soluble organic matter from both samples consists of significant amounts of nonmarine derived components. The Pleistocene sample (Site 92) was drilled just above the Sigsbee Scarp, near the Mississippi Cone, and contains mainly clay minerals. The alkane and acid distribution histogram for this sample is shown in Figure 9b. It clearly indicates the strong odd to even predominance of the normal alkanes with the maximum concentration in the C25 to C33 range and the even to odd predominance of the normal carboxylic acids with the maximum concentration in the C24 range. Such a distribution pattern in the long carbon chain range is characteristic of terrestrial plant wax residues. These patterns (cf. Figure 9b) and the presence of terpenoidal compounds strongly indicate a terrigenous source for this soluble organic matter, probably from turbidites and particulate organic matter carried down the Mississippi River system (Simoneit and Burlingame, 1972b).

The Miocene sample (Site 90) from the deep basin also had a clay mineralogy and an abundance of methane gas was encountered. It exhibits a normal alkane distribution with a strong odd to even predominance, maximizing in the range of C_{25} - C_{33} (cf. Figure 9a) and the normal carboxylic acid distribution has an even to odd predominance with maxima at C_{16} and C_{24} (cf. Figure 9a). Terpenoidal compounds were however not detected. The general compound distribution is analogous to Sample 10-92-5-4 as well as to samples analyzed from the Black Sea (Simoneit, in press) and it appears that the Gulf of Mexico may have had significant nonmarine influx during the Miocene. The total extractable organic matter from this sample is about



Figure 9. Alkane and acid distribution histograms for the Leg 10 samples (a) Sample 10-90-7-2 (concentrations in approximate ppm of dry sample); (b) Sample 10-92-5-4.

one-third that from the Pleistocene sample, although the organic carbon values are approximately the same for both. This indicates that diagenetic processes are operative, polymerizing and incorporating soluble organic matter into "kerogenic" matter.

Organic contamination in these samples was minor. The contaminants which were identified consisted mainly of butyl esters from the core tubes (Simoneit et al., 1972) and phthalate esters.

ACKNOWLEDGMENTS

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