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20. ISOTOPIC CHEMISTRY OF ORGANIC CARBON IN SEDIMENTS FROM LEG 184[1]

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ABSTRACT

Carbon isotopes were determined on the organic matter (OM) in sediments recovered from four sites (1143, 1144, 1146, and 1148) in the South China Sea during Ocean Drilling Program Leg 184. Concentrations of finely disseminated OM in the sediments were low (generally 0.1 to 0.5 wt%) and show organic $\delta^{13}C$ isotope values (generally –22‰ to –24‰) indicative of marine to mixed marine and land-derived OM. The shipboard sedimentologists noted and the author collected seven samples of discrete centimeter-size chunks of organic carbon–rich material, such as fragments of wood, from cores from the five sites. The $\delta^{13}C$ values in these samples ranged from –24‰ to –30‰. Humic material extracted from two wood/lignite samples and one plant fragment sample has nearly the same $\delta^{13}C$ value as the bulk material. The $\delta^{13}C$ values of humic extracts from the woody materials are different from those of marine materials, indicating that the extractable material is original, land-derived OM rather than absorbed/adsorbed OM from the marine environment. The $\delta^{13}C$ values (–28‰ to –30‰) for the two plant fragment samples appear unusually light but are probably reasonable for nonwoody land-derived OM, which is rarely observed, preserved, or analyzed in deep-sea cores. Assuming carbon isotope values of –20‰ for "pure" marine and –27‰ for "pure" terrigenous OM, samples from Leg 184 generally preserve 40%–60% marine and 40%–60% terrigenous OM. However, because marine OM is more metabolizable than terrigenous OM and because these samples contain pyrite sulfur, the actual concentration of marine OM originally deposited with the sediments was probably in the range of 60%–90%.

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INTRODUCTION

During Ocean Drilling Program (ODP) Leg 184, cores were recovered from five sites (1143, 1144, 1146, and 1148) in the South China Sea (SCS) to study climate history over the last 30 m.y. Although the total organic carbon (TOC) content of sediments from the Leg 184 sites was not particularly high (0.2–0.5 wt%; maximum = ~1 wt%), it records the source, productivity, and preservation of organic matter (OM) and provides sufficient material to be well within the analytical capability of modern, sensitive analytical laboratory instruments that measure δ^{13}C isotopic compositions. Organic matter in marine sediments can be a mixture of marine and land derived. The land-sourced OM signal is related to climate, erosion, and transport of bulk land materials (organic and mineral) to the sea. The relative amount of land-sourced OM can record this change with time.

The concentrations of TOC and δ^{13}C isotopes can often help define the source and depositional environment of organic matter. Carbon isotopes are used to define the relative amounts/sources of OM preserved in the sediments (Sackett, 1964; Nissenbaum and Kaplan, 1972; see also a recent summary of marine organic matter in Stein, 1991). The land-plant stable δ^{13}C isotope value is determined from direct measurement of "woody" material in the sediment; the marine isotope value is the heaviest δ^{13}C value measured for the bulk sediment. Thus, δ^{13}C determination for hand-picked pieces of "woody" material can be used to establish a land-plant isotopic end-member for a sediment carbon source budget.

Measurements of the δ^{13}C isotope variation in the bulk OM can be used to assess the contribution of land-derived OM in the sediment, which may give information about the relative importance of productivity, preservation, water depth, and distance from land. For example, in a perfect world, if woody OM yields a δ^{13}C value of –26‰ and most marine bulk OM yields –20‰, then other bulk samples with δ^{13}C values of –23‰ and –21.5‰ contain 50% and 25%, respectively, land-sourced OM. In general, marine OM δ^{13}C values range from –18‰ to –22‰ (Druffel et al., 1992) and land (terrigenous) OM δ^{13}C values range from –24‰ to –30‰ (Opsahl and Zepp, 2001).

MATERIALS AND METHODS

Samples from Leg 184 Sites

Total organic carbon content in Leg 184 sediments was generally low (<0.1–1.1 wt%) but typically ranged 0.2–0.5 wt% OM. The organic matter concentration in sediments is important for many reasons; one is that it controls the sediment oxidation-reduction state, which can be determined by observation of the bulk color of the sediment:

1. Brown = primary oxidized Fe (<0.1 wt% TOC);
2. Green = reduced Fe (0.1–0.2 wt% TOC);
3. Greenish gray = finely disseminated sulfidized Fe + pyrite (>0.2–0.5 wt% TOC); and
4. Dark gray = finely disseminated sulfidized Fe + visible pyrite (>0.4 and up to 1 wt% TOC).

Organic matter in sediments can have mixed marine and land sources. The land-sourced OM signal is related to climate and erosion and transport of bulk land minerals to the sea. The relative amount of land-sourced OM can record this change with time. The relative amounts of land- and marine-sourced OM are often determined using C/N ratios; unfortunately, the low concentrations of OM in the Leg 184 sediment samples made this determination difficult. Because TOC content is most often determined by difference (total carbon [CHN analysis/ Carlo Erba combustion] minus carbonate carbon [wet chemical/titration]) (Emeis and Kvenvolden, 1986) and because the sample was >30 wt% carbonate carbon, the analytical uncertainty was at least 0.1% (or 0.2%), which means the TOC value often had an uncertainty >20%. These "by difference" values were often as much as 30% lower than the Rock-Eval TOC values.

Organic-Rich Material

With the effort and sharp attention of the shipboard sedimentologists, seven centimeter-size samples of organic carbon–rich material, such as fragments of woody material, were noted and collected by the author from cores from the five sites. Five samples were later identified as lignite and two as "plant fragments." In order to determine the land-plant carbon isotopic end-member value, these seven samples were analyzed.

Laboratory Methods

On board ship, some samples were air-dried at room temperature and others were freeze-dried and stored in glass screw-top vials. Headspace samples were stored in crimp-top glass containers. The shore-based laboratory procedure was as follows. Samples were dried in air or under vacuum at <70°C. Sediment samples were pulverized/ground to <100 µm. A 1- to 5-g portion of the sample was treated successively with cold 1-N HCl and cold 3-N HCl and then warmed to 60°C in a test tube (in a hot water bath) and allowed to react for 24 hr to dissolve calcite. The sample was then centrifuged, and the acid solution was decanted. Samples were washed with deionized water (usually three or four times) and centrifuged until neutrality (pH = 7), and the solution was decanted. Finally, samples were vacuum dried at <60°C prior to isotope analyses.

Woody and OM-rich concretion samples were treated somewhat differently. Portions of three of these samples were treated with 0.1-N NaOH to extract humic material (indigenous terrestrial and/or adsorbed or absorbed marine). Soluble humic materials give a brown or tan color to the solution. The solution was decanted (and saved), and new NaOH solution was added to further extract humic material. The sample was centrifuged, and the humic material was decanted and combined with the first extract. This humic extract was acidified dropwise to pH = 1 with 1-N HCl, which caused the humic acid fraction to precipitate. The solution was decanted, and the humic acid was dried under vacuum at <60°C. Both the humic acid and the extracted woody material were analyzed for stable carbon isotopes. The humic acid extracts usually represented 4%–9% of the original OM and were sometimes only 5-mg samples. Fortunately, the new mass spectrometer systems are quite sensitive and are able to measure carbon isotopes on such small samples. The other four samples were much smaller and therefore not large enough to undergo extraction; these were analyzed without pretreatment.

Splits of the woody samples collected shipboard were given to Co-Chief Scientist Prof. Pinxian Wang for genus/species identification at the Chinese National Laboratory of Dendrology. Unfortunately, the samples were not suitable or too small(?) for identification. My colleague, Dr. Neely Bostick (U.S. Geological Survey [USGS] Emeritus Scientist), produced the identifications used in this report. He positively identified some samples as peat or lignite, but two samples do not have the characteristic "lignite" woody texture and may represent the leaf or other soft parts of plants (apparently in a clay matrix).

Combustion and Analysis for $\delta^{13}C$

The carbonate-free residue was combusted to CO_2 and purified using an elemental analyzer coupled to a Micromass Optima isotope ratio mass spectrometer in continuous flow mode (USGS laboratory in Denver, Colorado). The $\delta^{13}C$ isotope value of this CO_2 was measured relative to Vienna Peedee belemnite (VPDB) and working standards, where a δ of 1‰ is 0.1% different from the $^{13}C/^{12}C$ ratio of the VPDB standard. Several replicate preparations were run (due to unknown organic carbon values), and, therefore, multiple analyses of many samples were conducted to adjust the amount of CO_2 into the range for optimum mass spectrometer determination. The results were generally ±0.2‰ for replicate analyses of these low-TOC samples.

Problems with Headspace Gas Samples

A total of 15 headspace samples (from shipboard determinations and in their original crimp-top glass containers) from the high-methane zones at Site 1146 were analyzed after extraction with a 10-mL gas syringe and injection into a sample loop connected to a combustion furnace coupled to a Micromass Optima mass spectrometer. Unfortunately, the headspace gas contained essentially no methane (<100 ppm), probably due to air leakage (despite a silicone-sealed septum) or microbial oxidation on the sediment surface (after storage at room temperature for >2 months). The lesson to be learned from this is that headspace gas containing methane must be separated from its sediment host if not analyzed within 24 hr of sampling. The headspace gas should be transferred to a vacutainer-type container (as was done for samples collected for Dr. Matsumoto of Tokyo University, Japan).

Summary of Analyses

A total of 24 organic matter samples were measured to determine $\delta^{13}C$ stable isotope values. Seven centimeter-sized organic matter samples (wood fragments) were measured for bulk $\delta^{13}C$ values. Humic acid was extracted from several samples and the $\delta^{13}C$ value determined as well as, in one case, the $\delta^{13}C$ value of the residue after extraction (Table T1).

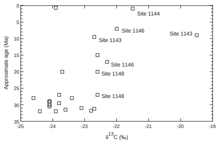
T1. $\delta^{13}C$ isotope values for organic carbon, p. 12.

RESULTS AND DISCUSSION

Sediment Organic Carbon

Disseminated organic matter from Leg 184 samples had TOC contents of 0.2–1.1 wt% and $\delta^{13}C$ stable isotope values ranging –19‰ to –24‰. One sample from Hole 1143C yielded a $\delta^{13}C$ value of –19.5‰, indicating that the OM source for this sample is mainly marine. There were 17 samples from Site 1148 that showed $\delta^{13}C$ values between –22.6‰ and –24.6‰, in the range between normal marine-sourced and land-sourced OM. Three samples from Hole 1146A, two from Hole 1144A, and one from Hole 1143C yielded similar values (–21.5‰ to –23.9‰). Therefore, most of the samples contain some (25%–75%) land-derived OM. Table **T1** lists these data. Percent carbon is organic carbon content determined as part of the isotope analysis (i.e., after removal of carbonate). Therefore, the original TOC content of the sample was one-third to one-half as much percent carbon, depending on the carbonate content. Figure **F1** shows these data plotted vs. age.

F1. Disseminated sedimentary $\delta^{13}C$, p. 10.



These $\delta^{13}C$ isotope values (–22.6‰ to –24.6‰) are in the range reported for marine-derived sediment OM. However, because “pure” marine-sourced OM is usually thought to be in the range of –18‰ to –21‰ for tropical oceans, it appears that ~25%–75% of the OM may be land derived ($\delta^{13}C$ = –26‰). Opsahl and Zepp (2001) report riverine dissolved organic carbon (DOC) and Raymond and Bauer (2001) report riverine DOC and particulate organic carbon (POC) that show $\delta^{13}C$ isotope values of –26‰ to –31‰, in the same range as most of our “woody” material.

The presence of mixed marine-terrestrial OM in these sediments is supported by the shipboard Rock-Eval data (Shipboard Scientific Party, 2000), which shows borderline type II (marine-sourced OM)/type III (land-plant–sourced OM) values on hydrogen index (HI) vs. oxygen index (OI) plots, as well as by C/N values that often exceed 10. (HI = S_2/TOC, where S_2 = mg of pyrolysis hydrocarbons/g sample; OI = S_3/TOC, where S_3 = mg pyrolysis CO_2/g sample.) Some caution must be used with all these results because of the very low TOC values (and high carbonate carbon values), which make Rock-Eval results less definitive. The very low total N values make the organic C/N values less certain, and it is possible that some N is not organic (i.e., ammonia nitrogen, probably fixed to clay minerals).

There appears to be a trend in the data with age; $\delta^{13}C$ values of older samples are slightly more negative (isotopically lighter). This can be explained by the greater amounts of land-derived organic matter (with lighter isotopic value) present in the older sediment samples. This explanation is reasonable because the amount of land material transported and preserved in the sediment would probably have been greater in the past when the SCS was not as deep or the hole locations were closer to shore or during times of greater weathering and erosion. In addition, microbial sulfate reduction preferentially uses marine OM (Westrich and Berner, 1984); this could in part explain the high amounts of land-sourced OM indicated by the $\delta^{13}C$ isotope (light values) estimate.

Woody and Concentrated Organic Matter and Extracts

Woody Samples

The presence of wood samples in the Leg 184 sediments is rare, as it is in most open-marine deep-sea sediments. It requires unusual events to transport the wood fragments from land via rivers to the ocean, further transport (floating, wind, and ocean currents) 100 km or more offshore, to sink to the seafloor, and be buried in the sediment. Indeed, most Leg 184 shipboard scientists were skeptical that any wood fragments could be found. Several collections of black sediment masses turned out to be sulfides (pyrite and troilite) rather than the sought-after land-sourced OM. After the first find at Site 1143, the search continued and organic-rich concentrations were recovered from sediments from Sites 1144, 1145, 1146, and 1148. Interestingly, the (now) more offshore southern Site 1143 yielded a 5-Ma sample from deep (230 meters below seafloor [mbsf]) sediments, northern SCS Site 1148 yielded a deep (515 mbsf) sample (27 Ma), and Site 1144 yielded a deep (235 mbsf) sample that was very young (0.2 Ma). Basin position, age, and depth do not appear to exclude the presence of woody fragments. However, four of the samples came from <130 mbsf and were young (<1.2 Ma), although only three of the sites had older sediments. The shallowest and youngest samples appeared more peaty, whereas the deeper, older samples appeared more like lignite (brown coal) (N. Bostick, pers. comm., 2000), as expected from organic maturation studies (Bostick, 1979).

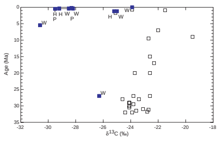
Seven samples containing centimeter-size concentrations (wood fragments) of organic matter were measured for $\delta^{13}C$ stable isotope values on the bulk sample, the extracted humic acid, and the residue after extraction. Carbon isotope values of the concentrated OM and woody OM were generally isotopically lighter (depleted in $\delta^{13}C$) than the bulk sediment OM. Carbon isotope values of the bulk and woody OM ranged from –23.9‰ to –30.6‰. Most woody OM showed $\delta^{13}C$ isotope values from –23.5‰ to –26.5‰, so some of these samples are isotopically lighter (more negative) than expected for normal land wood. This can be explained by the results of Leavitt and Long (1982, 1986), who report that leaf and twig material are 2‰–4‰ lighter (more negative) than wood of junipers and pines. Benner et al. (1987) reported that lignin from leaves and wood is 3‰–6‰ lighter than cellulose.

Humic Extracts

Extraction of humic materials was performed on three samples to remove any possible marine-sourced soluble OM that may have been absorbed into or adsorbed onto the wood during its transport from land to the deep sea or during burial in the sediment. For the three samples from which humic acid was extracted/recovered, the $\delta^{13}C$ carbon isotope values are within 0.2‰ of the bulk sample value (and within the range of replicate analyses). In one case (Section 184-1146C-7H-2), the humic extract was 0.7‰ lighter (more negative) after extraction than the lignite from which it was extracted (or 1.0‰ lighter than the unleached sample). The results are listed on Table **T2.** Figure **F2** shows these data plotted vs. age.

T2. $\delta^{13}C$ for wood, plant fragment, and humic extracts, p. 13.

F2. Woody/concentrated $\delta^{13}C$, p. 11.



Because the age of all but two of the woody samples is <1.5 Ma, it is not possible to give great significance to any isotopic trends with age.

Probably no trends would be expected, and, if found, they would be questionable because of the presumably episodic occurrence of the woody materials, which represents unusual occurrences of transport from land, probably during severe ocean storm and/or land rainfall or some other events. It is interesting that woody materials are mainly concentrated/observed during the last 1.5 m.y. (but only three sites contained older sediments).

Provisional Isotopic Modeling of Organic Matter Types

For samples that have intermediate values between marine and terrestrial OM, the relative amounts of each type can be estimated. Taking –20‰ for the marine organic matter δ^{13}C source value (the heaviest SCS value is actually –19.5‰) and using –26‰ as the terrigenous end-member:

δ^{13}C of –21.5‰ = ~75% marine and 25% terrigenous OM.
δ^{13}C of –23.0‰ = ~50% marine and 50% terrigenous OM.
δ^{13}C of –24.5 ‰ = ~25% marine and 75% terrigenous OM.

Of course, it is not this simple because some of the woody samples are lighter than –26‰ yet can be no more than 100% terrigenous OM. However, as shown by our terrestrial/woody samples, the end-member is probably between –25‰ and –29‰. Any assumptions about these end-member values will result in errors in the proportions of the source of the OM. Likewise, samples visually identified as lignite that have values of –24‰ and –25‰ are not partly marine OM, as shown by the laboratory chemical extraction of sorbed soluble OM (humic material), which has essentially the same isotope value as the bulk woody sample.

Furthermore, when estimating actual input of sedimentary OM rather than preserved (measured data) sedimentary OM, syngenetic and diagenetic microbial sulfate reduction, which results in FeS and pyrite, must be considered. This mechanism mainly uses the more metabolizable marine OM (Berner and Westrich, 1985). Therefore, the originally deposited OM probably had contained at least 30% more marine OM than now measured, based on the amount of pyrite sulfur present (0.1–0.3 wt%) in most samples (organic C/S = 3; Wang, Prell, Blum, et al., 2000). Thus, the original OM deposited in the sediments was 50%–90% marine OM.

CONCLUSIONS

Organic matter is not abundant in Leg 184 samples from the SCS, ranging from <0.1 to 0.5 wt%, with a few samples between 0.6 and 1.1 wt% TOC. Organic matter from five cores/sites from Leg 184 was analyzed for stable carbon isotope content. Disseminated OM generally shows δ^{13}C values between –22‰ and –24‰, indicating that it is a mixture of marine- and land-sourced OM. Samples of centimeter-size woody material and plant fragments give δ^{13}C isotope values generally between –24‰ and –29‰. The δ^{13}C values lighter than –26‰ are unusual for wood and lignite but are typical of twigs and lignin. Using –20‰ as the marine OM and –26‰ as the land OM end-members, the proportion of OM measured in the Leg 184 sediments ranges from 40%–60% marine sourced. This seems rather low for deep-sea sediments but actually is not unreasonable for two reasons. First, these sediments, while deep, are ~100 km from land but were probably closer to land at the time of deposition (and during ice ages the sea level was lower). Second, marine OM is preferentially metabolized (over more refractory land-sourced OM) by microbiological sulfate reduction to form iron monosulfide, pyrite, and marcasite. If the S content of the sediments is factored into the OM budget, the original amount of OM deposited with the sediments was 60%–90% marine sourced.

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Figure F1. Plot of disseminated sedimentary organic carbon isotope values vs. age.

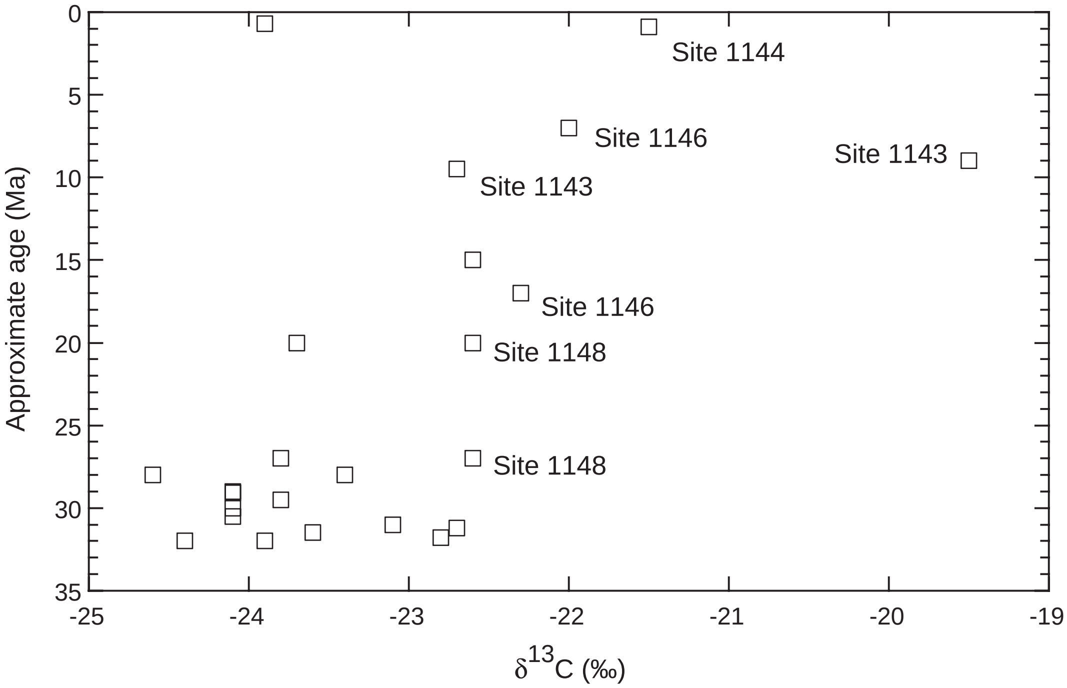

Figure F2. Plot of woody/concentrated OM carbon isotope values vs. age. Solid symbols = woody samples, open symbols = disseminated OM from Figure F1, p. 10. Organic concentrations are identified as W= lignite, P = plant fragments, H = humic extract. Humic extracts and parent samples are linked by lines.

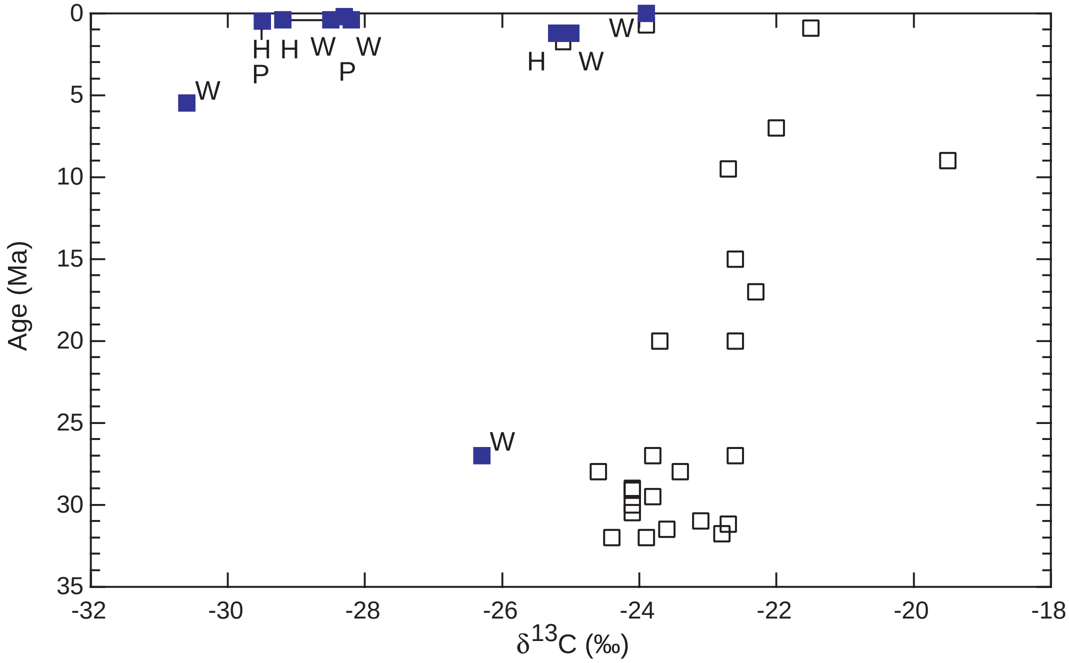

Table T1. δ^{13}C isotope values for organic carbon.

Core, section, interval (cm)	Depth (mbsf)	Carbon* wt%	δ^{13}C (‰)	Description	Approximate age (Ma)
184-1143C-					
46X-5, 99–101	440	1.0	−19.5	Sediment	9
48X-4, 0–5	460	0.2	−22.7	Sediment	9.5
184-1144A-					
32X-4, 0–5	330	0.49	−23.9	Sediment	0.7
40X-3, 0–5	420	0.57	−21.5	Sediment	0.9
184-1146A-					
36X-4, 0–5	350	0.2	−22.0	Sediment	7
56X-4, 0–5	560	0.16	−22.6	Sediment	15
64X-4, 0–5	640	0.21	−22.3	Sediment	17
184-1148A-					
47X-4, 0–5	450	0.26	−22.6	Sediment	20
47X-CC, 21–24	455	0.64	−23.7	Sediment	20
54X-1, 1–4	496	0.5	−22.6	Sediment	27
56X-1, 1–4	506	0.67	−23.8	Sediment	27
59X-2, 1–4	550	0.45	−24.6	Sediment	28
61X-CC, 24–29	561	0.72	−23.4	Sediment	28
63X-4, 1–4	580	0.50	−24.1	Sediment	29
64X-2, 107–08	585	0.5	−24.1	Sediment	29.1
66X-4, 0–5	606	0.39	−23.8	Sediment	29.5
68X-2, 105–06	624	0.50	−24.1	Sediment	30
72X-4, 0–5	666	0.48	−24.1	Sediment	30.5
74X-4, 107–08	685	0.8	−23.1	Sediment	31
77X-4, 0–5	715	—	−22.7		31.2
184-1148B-					
47X-CC, 33–34	790	0.8	−23.6	Sediment	31.5
50X-CC, 23–24	815	—	−22.8	Sediment	31.8
56X-CC, 30–31	850	1.1	−24.4	Sediment	32
56X-CC, 31–37	850	0.63	−23.9	Sediment	32

Notes: * = analyzed after leach. — = not available.

Table T2. $\delta^{13}C$ isotope values for wood, plant fragment, and humic extracts.

Core, section, interval (cm)	Depth (mbsf)	Carbon (wt%)	$\delta^{13}C$ (‰)	Description	Approximate age (Ma)
184-1143A-					
24H-CC, 27–28	230	51	–30.6	Lignite	5.5
184-1144C-					
1H-5, 36–36	6	54	–23.9	Lignite	0.01
184-1144B-					
24H-2, 35–36	235	48	–28.3	Plant fragments	0.2
184-1145A-					
13H-4, 5–7	120	46	–25.0	Lignite	1.2
13H-4, 5–7	120	32	–25.2	Humic extracts	1.2
184-1146C-					
7H-2, 96–97	70	6	–28.2	Lignite	0.4
7H-2, 96–97	70	14	–28.5	Lignite, leached	0.4
7H-2, 96–97	70	7	–29.2	Humic extracts	0.4
10H-3, 51–52	95	19	–29.5	Plant fragments	0.5
10H-3, 51–52	95	26	–29.5	Humic extracts	0.5
184-1148A-					
57X-1, 29–30	515	43	–26.3	Lignite	27