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16. Data Report: Stable Isotopic Measurements of Sedimentary Organic Matter and *N. pachyderma* (s.) from Site 1166, Prydz Bay Continental Shelf[1]

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ABSTRACT

We report the results of downhole stable isotopic ($\delta^{13}C_{org}$ [organic carbon] and $\delta^{15}N$) and elemental measurements (total organic carbon [TOC], total nitrogen [TN], and carbon/nitrogen [C/N]) of sedimentary organic matter (SOM) along with stable isotopic measurements ($\delta^{18}O$ and $\delta^{13}C$) of left-coiling *Neogloboquadrina pachyderma* planktonic foraminifers from Ocean Drilling Program Site 1166. TOC and TN measurements indicate a large change from organic-rich preglacial sediments with primary organic matter to organic-poor early glacial and glacial sediments, with mainly recycled organic matter. Results of the stable isotopic measurements of SOM show a range of values that are typical of both marine and terrestrial organic matter, probably reflecting a mixture of the two. However, C/N values are mostly high (>15), suggesting greater input and/or preservation of terrestrial organic matter. Foraminifers are only present in glacial/glaciomarine sediments of latest Pliocene to Pleistocene age at Site 1166 (lithostratigraphic Unit I). The majority of this unit has $\delta^{13}C_{org}$ and TOC values that are similar to those of glacial sediments recovered at Site 1167 (lithostratigraphic Unit II) on the slope and may have the same source(s). Although the low resolution of the *N. pachyderma* (s.) $\delta^{18}O$ and $\delta^{13}C$ data set precludes any specific paleoclimatic interpretation, downcore variations in foraminifer

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$\delta^{18}O$ and $\delta^{13}C$ values of 0.5‰ to 1‰ amplitude may indicate glacial–interglacial changes in ice volume/temperature in the Prydz Bay region.

INTRODUCTION

An important objective of Ocean Drilling Program (ODP) Leg 188 was to penetrate glacial sediments and recover evidence of the timing for the onset of glacial conditions and ice sheet formation in Prydz Bay (O'Brien, Cooper, Richter, et al., 2001). With this in mind, Site 1166 was selected to obtain sediments below those reached at Site 742 during ODP Leg 119 and to provide a stratigraphic record that spans and includes the transition from preglacial to glacial conditions (O'Brien, Cooper, Richter, et al., 2001). Although recovery was low (16%), this objective was accomplished. The Site 1166 sedimentary section includes glacial, early glacial, and preglacial sediments that record the evolution of Prydz Bay since the late Cretaceous and the development of the adjacent Lambert Glacier-Amery Ice Shelf system (O'Brien, Cooper, Richter, et al., 2001).

Stable isotopic and elemental measurements of sedimentary organic matter (SOM) can provide valuable source and paleoenvironmental information (Meyers, 1994; Popp et al., 1997). Shipboard analysis of a small number of samples from Site 1166 for total organic carbon (TOC) and total nitrogen (TN) elemental abundances revealed significant downcore changes from glacial sediments with little organic carbon and nitrogen (<0.5 and <0.05 wt%, respectively) to preglacial sediments with more significant TOC and TN content (up to 9.27 and 0.27 wt%, respectively) (Shipboard Scientific Party, 2001). Rock-Eval analysis of the organic matter (OM) indicated that preglacial sediments at the base of lithostratigraphic Unit III and in Unit IV are mainly primary OM, whereas the younger glacial/glaciomarine sediments are mainly higher-maturity recycled OM with smaller, variable amounts of primary OM (Shipboard Scientific Party, 2001).

Oxygen stable isotopic measurements of foraminiferal calcite are an established proxy for paleoclimatic and paleoceanographic information (Emiliani, 1955). Foraminifers are rarely found in Site 1166 sediments and are confined to the glacial and glaciomarine sediments of lithostratigraphic Unit I. The majority are the left-coiling variety of the planktonic species *Neogloboquadrina pachyderma* (s.) that are commonly used for stable isotopic studies in the polar regions (e.g., Mackensen et al., 1989; Hodell, 1993). The purpose of this study is to derive further source, paleoenvironmental, and paleoclimatic information for the Cenozoic evolution of Prydz Bay, using stable isotopic and elemental measurements of SOM and *N. pachyderma* (s.) foraminifers from Site 1166 sediments.

MATERIALS AND METHODS

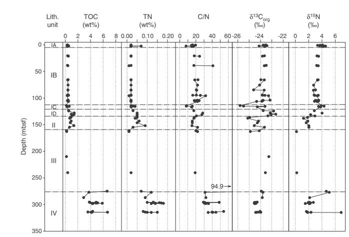
For this study, 65 samples of 10–15 cm^3 were collected shipboard and later analyzed at the Stanford University Stable Isotope Laboratory. A small portion of each sample was set aside to determine elemental and stable isotopic compositions of bulk SOM. Sediment samples were soaked in a diluted Calgon solution until they disaggregated. Samples were then wet-sieved through a 150-µm screen, and the >150-µm fraction was dried in an oven at 50°C. Inspection of the >150-µm fraction revealed that, with the exception of samples from lithostratigraphic Subunit IB, all samples were barren of foraminifers. For samples with a sufficient number of foraminifers, six to ten *N. pachyderma* (s.) specimens were picked from the >150-µm fraction. Foraminifer samples were loaded into glass reaction vials and analyzed using a Finnigan automated Kiel IV acidification unit and a MAT 252 isotope ratio mass spectrometer combination. Approximately 10% of unknowns were replicated, giving an average standard deviation of ~0.04‰ for $\delta^{18}O$ and 0.02‰ for $\delta^{13}C$. Results are reported relative to the Peedee belemnite (PDB) standard for both oxygen and carbon.

SOM sample portions were dried, ground, weighed, and loaded into silver boats. Samples were acidified in situ with 6% sulfurous acid to remove carbonate phases (Verardo et al., 1990) prior to carbon elemental and stable isotopic analysis (TOC and $\delta^{13}C_{org}$). All samples were later weighed and loaded into tin boats for measurements of TN and $\delta^{15}N$. Separate analyses for nitrogen were necessary because of the large amount of sample material needed (>120 mg in glacial sediments) to obtain reliable nitrogen isotopic values. All measurements were completed with a Carlo Erba NA1500 elemental analyzer/Conflo II device attached to a Finnigan Delta Plus mass spectrometer. Elemental compositions were measured using the mass 44 beam intensity and calibrated with at least eight standards that were analyzed throughout the course of each run. The NA1500 is equipped with a low-background, He-purged sample carousel, which is essential for properly analyzing samples with low nitrogen, such as the ones discussed in this contribution. $\delta^{13}C$ compositions were standardized against USGS-24 (U.S. Geological Society graphite), and $\delta^{15}N$ compositions were standardized against IAEA-N1 (International Atomic Energy Agency ammonium sulfate). Relative reproducibility of the acetanilide standard was 0.35 wt% for carbon and 0.11 wt% for nitrogen. $\delta^{13}C$ reproducibility of USGS-24 was 0.06‰, and $\delta^{15}N$ reproducibility of IAEA-N1 was 0.13‰. Approximately 10% of unknowns were replicated, yielding average standard deviations of 0.03 wt% for TOC, 0.16‰ for $\delta^{13}C_{org}$, 0.001 wt% for TN, and 0.16‰ for $\delta^{15}N$.

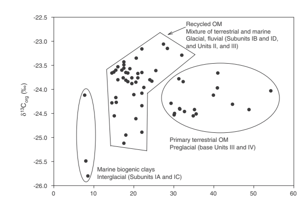
T1. Elemental and stable isotopic results, p. 10.

T2. *N. pachyderma* (s.) $\delta^{13}C$ and $\delta^{18}O$ stable isotopic results, p. 11.

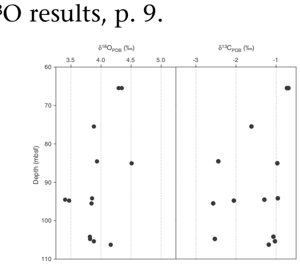
F1. Elemental and stable isotopic results, p. 7.



F2. Crossplot of $\delta^{13}C_{org}$ and C/N, p. 8.



F3. *N. pachyderma* (s.) $\delta^{13}C$ and $\delta^{18}O$ results, p. 9.



RESULTS AND INTERPRETATION

Results are listed in Tables T1 and T2 and are shown in Figures F1, F2, and F3.

Sedimentary Organic Matter

The elemental abundance and stable isotopic composition of SOM reflects the bulk signal derived from the mixture of all sources of OM that accumulated on the Prydz Bay shelf. In the antarctic marine environment this may include open-water marine phytoplankton, sea-ice algae, and terrestrial sources of OM with a wide range of $\delta^{13}C_{org}$ and $\delta^{15}N$ values (Rau et al., 1991; Villinski et al., 2000). TOC ranges from 0.17 to 6.7 wt% and varies according to the lithostratigraphic unit sampled (Fig. F1). SOM from lithostratigraphic Unit I and the majority of Unit III are characterized by very low TOC (<0.5 wt%) values. Biogenic clays in Unit II have slightly higher values (0.7 to 0.9 wt%). Values in Subunit ID reach 1.5 wt% TOC, and the preglacial carbonaceous clay sediments of Unit IV vary between 3 and 6.7 wt% TOC. TN values range from 0.01 to 0.18 wt% and follow the trends reported for weight percent TOC (Fig. F1).

SOM $\delta^{13}C_{org}$ values range from –22.7‰ to –25.8‰ and vary by up to 2‰ over short intervals (Fig. F1). All of the $\delta^{13}C_{org}$ values measured in this study fall within the known ranges for modern particulate organic matter (POM) measured in Prydz Bay (Gibson et al., 1999) and the Weddell (Rau et al., 1991) and Ross (Villinski et al., 2000) Seas of Antarctica. These values also fall on the isotopically heavier end of the known range for C_3 terrestrial plants, which average approximately –27‰ (Meyers, 1994). Values are most variable in lithostratigraphic Subunit IC and at the contact between Subunit ID and Unit II where glaciomarine sediments (–22.7‰ to –24.6‰) alternate with biogenic clays and clayey silts with slightly lower $\delta^{13}C_{org}$ values (–25‰ to –25.8‰) and the lowest carbon/nitrogen (C/N) values (8 to 8.5) (Fig. F1). The TOC and $\delta^{13}C_{org}$ values measured in sediments of lithostratigraphic Unit I at Site 1166 (with the exception of Subunit IC) are similar to those in the glacial sediments that dominate the stratigraphy (lithostratigraphic Unit II) at Site 1167 drilled on the upper slope (Theissen et al., in press). Additionally, shipboard Rock-Eval analysis of these two units indicates that they are both composed of highly mature recycled OM (Shipboard Scientific Party, 2001). The similarity between the two units suggests that they may have the same source(s) and establishes a potential link between shelf and slope sedimentation during the late Pliocene–Pleistocene.

Variability in $\delta^{13}C$ values may reflect changes in the proportions of different types and amounts of marine algae in Prydz Bay. Gibson et al. (1999) showed that Prydz Bay sea-ice algae are highly enriched in $\delta^{13}C$ (–8.2‰ to –18‰), whereas POM from the underlying water column has values ranging from –21‰ to –26‰. The variability in $\delta^{13}C_{org}$ might also indicate changes in the proportions of marine algal and terrestrial sources of OM. Several lines of evidence point to the input of terrestrial organic material at Site 1166:

1. Results of the shipboard sedimentary investigation indicate glacial, proglacial, fluvial/deltaic, and lagoonal depositional settings that have continental provenance (Shipboard Scientific Party, 2001).
2. C/N values measured in this study are mainly high (majority of section >15 and preglacial >30), characteristic of terrestrial OM (Fig. F1) (e.g., Meyers, 1994).
3. Analyses of kerogen, extractable OM, and biomarkers from glacial and preglacial sediments recovered from three Leg 119 shelf sites drilled in Prydz Bay (Sites 739, 741, and 742) indicate a strong terrestrial signal (McDonald et al., 1991; Kvenvolden et al., 1991).

Terrestrial OM, however, is more durable than marine sources of OM and may be overrepresented in the sedimentary record (de Lange et al., 1994). A crossplot of $\delta^{13}C_{org}$ and C/N illustrates three groupings of Site 1166 SOM that reflect changes in the depositional environment (Fig. F2).

SOM $\delta^{15}N$ values range from 0.18‰ to 6.7‰ (Fig. F1). To our knowledge, these are the first reported sedimentary $\delta^{15}N$ values from Prydz Bay. On average, the glacial sediments in Unit I have slightly higher values than the preglacial samples, although the highest values occur in the organic-rich sediments at the base of both Units III and IV (Fig. F1).

A shift to lower values occurs at the boundary between lithostratigraphic Units I and II, and the lowest values are recorded in Unit III. The range of Site 1166 $\delta^{15}N$ values is characteristic of both antarctic terrestrial and marine OM. POM samples from the Weddell Sea have a wide range of $\delta^{15}N$ values (–5.4‰ to +41.3‰), with the highest values occurring in sea-ice algae (Rau et al., 1991). Antarctic terrestrial values (measured in soils and lake sediments) show an even wider range (–49‰ to +31‰) (Wada et al., 1981).

N. pachyderma (s.) Foraminifers

$\delta^{18}O$ (3.4‰ to 4.5‰) and $\delta^{13}C$ values (–0.7‰ to –2.6‰) of *N. pachyderma* (s.) foraminifers from lithostratigraphic Unit I (65.5 to 106.3 meters below seafloor [mbsf]) are similar to late Pliocene–Pleistocene values for *N. pachyderma* (s.) from the slope (Site 1167) (Theissen et al., in press) and rise (Site 1165) (**Warnke et al.,** this volume) in the Prydz Bay region. The low resolution of the Site 1166 *N. pachyderma* (s.) $\delta^{18}O$ and $\delta^{13}C$ records precludes any interpretation of particular paleoenvironmental conditions, but the temporal range in the results ($\delta^{18}O$ = 1.1‰ and $\delta^{13}C$ = 1.9‰) suggests that glacial–interglacial changes in ice volume/temperature and surface water productivity are potentially preserved and warrant a more detailed investigation.

SUMMARY

Downcore stable isotopic and elemental measurements from ODP Site 1166 indicate changes in OM that correspond to changes in the depositional environment on the Prydz Bay continental shelf since the Late Cretaceous. Large changes in TOC (6.5 to <0.5 wt%) and TN reflect the transition from preglacial sediments with primary OM to highly mature recycled OM deposited during glacial conditions. $\delta^{13}C_{org}$ and $\delta^{15}N$ values indicate a mixture of marine algal and terrestrial sources of OM. Most samples have high C/N values, indicative of terrestrial OM. Although the limited foraminifer $\delta^{13}C$ and $\delta^{18}O$ data set precludes any paleoclimatic interpretation, shifts of 0.5‰ to 1‰ amplitude in $\delta^{13}C$ and $\delta^{18}O$ values of *N. pachyderma* (s.) may reflect Pliocene to Pleistocene glacial–interglacial cycling in Prydz Bay.

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Figure F1. Downcore elemental (total organic carbon [TOC], total nitrogen [TN], C/N), and stable isotopic ($\delta^{13}C_{org}$ [organic carbon] and $\delta^{15}N$) results, Site 1166.

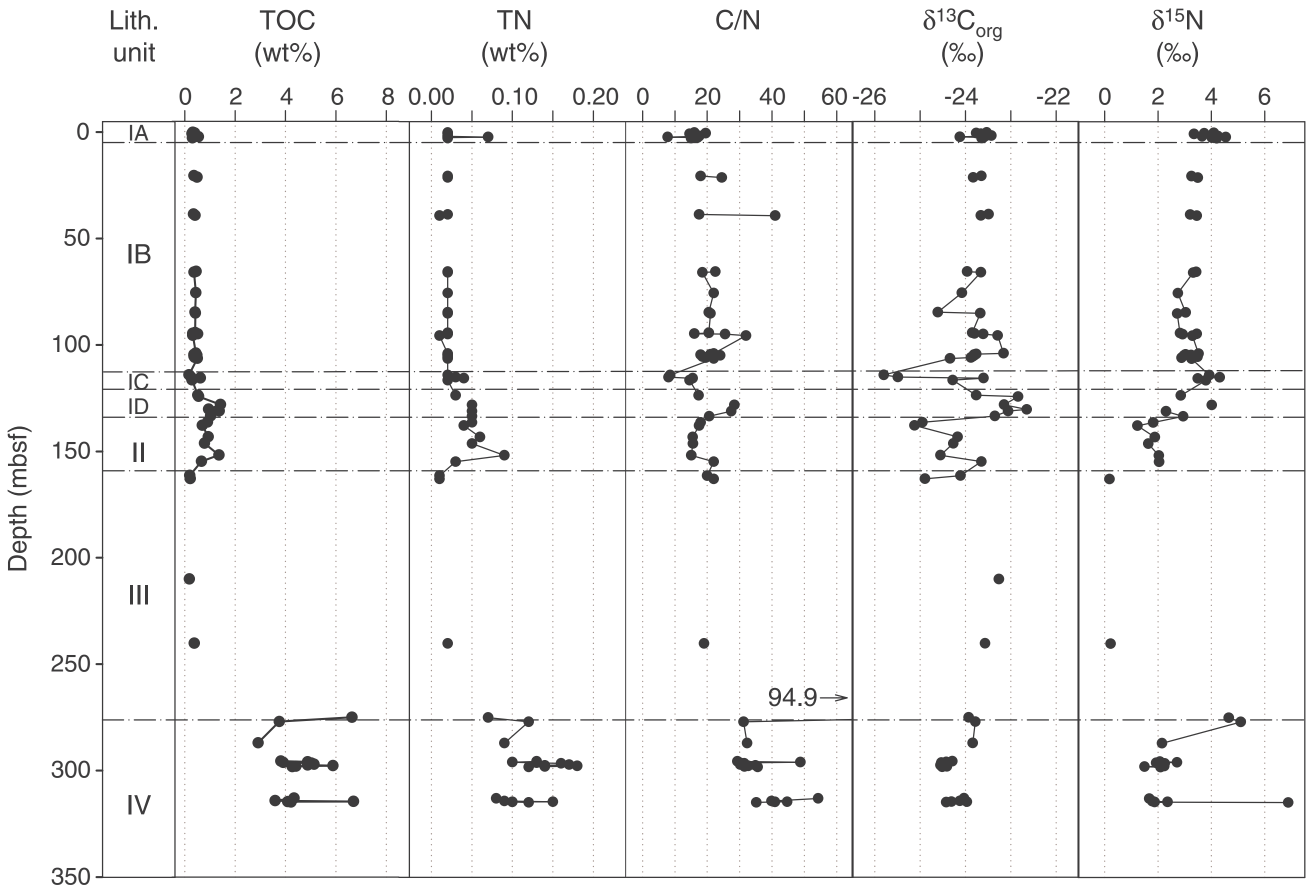

Figure F2. Crossplot of $\delta^{13}C_{org}$ (organic carbon) and C/N, Site 1166. Three groupings of organic matter (OM) reflect changes in the depositional environment and climate through the Cenozoic.

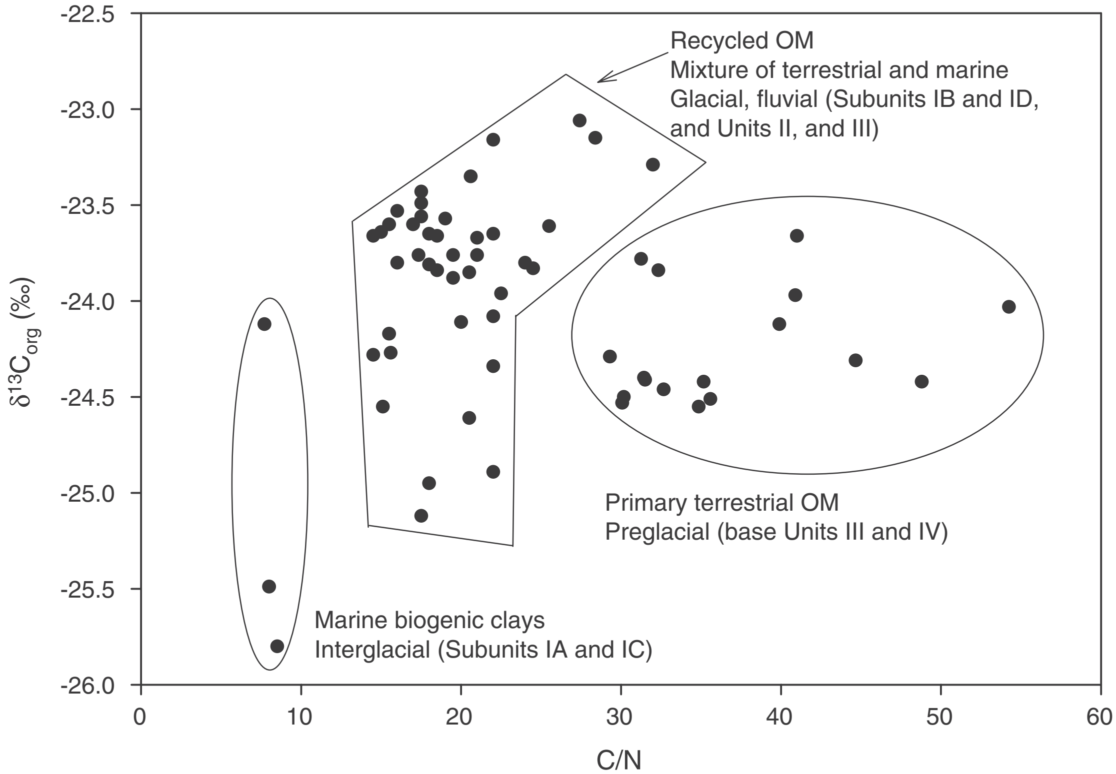

Figure F3. *N. pachyderma* (s.) $\delta^{13}C$ and $\delta^{18}O$ results between 65 and 106 mbsf. PDB = Peedee belemnite standard.

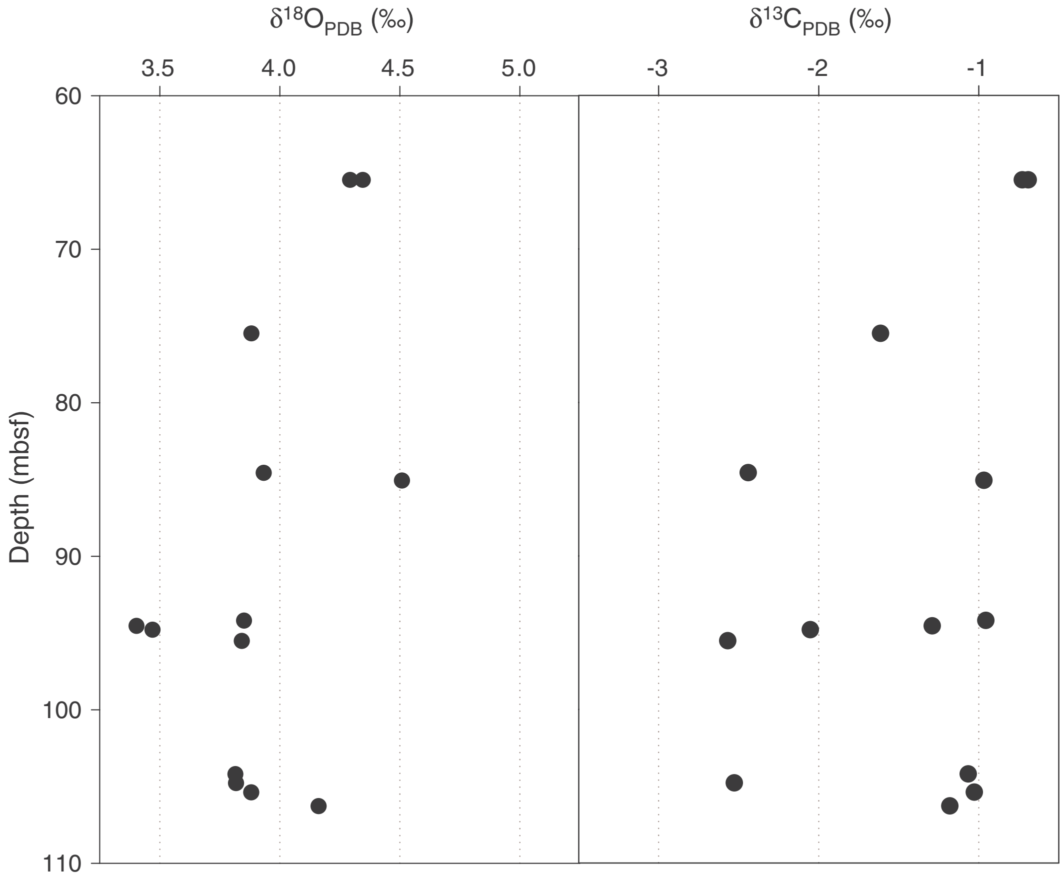

Table T1. Elemental and stable isotopic results, Hole 1166A.

Core, section, interval (cm)	Lithostratigraphic unit	Depth (mbsf)	TOC (wt%)	TN (wt%)	C/N	$\delta^{15}N$ (air) (‰)	$\delta^{13}C_{org}$ (PDB) (‰)
188-1166A-							
1R-1, 14–16	IA	0.14	0.32	0.02	16.0	4.09	–23.53
1R-1, 43–45	IA	0.43	0.39	0.02	19.5	3.73	–23.76
1R-1, 75–77	IA	0.75	0.29	0.02	14.5	3.35	–23.66
1R-2, 15–18	IA	1.61	0.35	0.02	17.5	4.24	–23.43
1R-2, 40–42	IA	1.86	0.35	0.02	17.5	3.65	–23.56
1R-2, 72–73	IA	2.18	0.54	0.07	7.7	4.54	–24.12
1R-2, 101–103	IA	2.47	0.34	0.02	17.0	4.02	–23.60
1R-CC, 6–8	IA	2.75	0.30	0.02	15.0	4.20	–23.64
3R-1, 73–76	IB	20.53	0.36	0.02	18.0	3.25	–23.65
3R-1, 147–150	IB	21.27	0.49	0.02	24.5	3.49	–23.83
5R-1, 39–41	IB	38.59	0.35	0.02	17.5	3.21	–23.49
5R-1, 90–92	IB	39.10	0.41	0.01	41.0	3.45	–23.66
8R-1, 9–11	IB	65.49	0.45	0.02	22.5	3.43	–23.96
8R-1, 40–42	IB	65.80	0.37	0.02	18.5	3.32	–23.66
9R-1, 50–53	IB	75.50	0.44	0.02	22.0	3.10	–24.08
10R-1, 17–20	IB	84.57	0.41	0.02	20.5	3.04	–24.61
10R-CC, 9–14	IB	85.07	0.42	0.02	21.0	2.72	–23.67
11R-1, 20–25	IB	94.20	0.41	0.02	20.5	2.82	–23.85
11R-1, 55–60	IB	94.55	0.32	0.02	16.0	3.45	–23.80
11R-1, 80–82	IB	94.80	0.51	0.02	25.5	2.92	–23.61
11R-CC, 9–14	IB	95.53	0.32	0.01	32.0	3.29	–23.29
12R-1, 20–22	IB	103.90	0.44	0.02	22.0	3.52	–23.16
12R-1, 50–55	IB	104.20	0.42	0.02	21.0	3.03	–23.76
12R-1, 80–85	IB	104.50	0.36	0.02	18.0	3.24	–23.81
12R-1, 110–115	IB	104.80	0.48	0.02	24.0	2.97	–23.80
12R-2, 19–21	IB	105.39	0.37	0.02	18.5	3.48	–23.84
12R-2, 70–72	IB	105.90	0.39	0.02	19.5	2.88	–23.88
12R-CC, 5–10	IB	106.29	0.50	0.02	22.0	3.26	–24.34
13R-1, 73–75	IC	114.03	0.17	0.02	8.5	3.92	–25.80
13R-2, 24–28	IC	115.04	0.24	0.03	8.0	4.31	–25.49
13R-2, 72–74	IC	115.52	0.62	0.04	15.5	3.49	–23.60
13R-3, 22–24	IC	116.42	0.29	0.02	14.5	3.79	–24.28
14R-1, 52–55	ID	123.52	0.52	0.03	17.3	2.85	–23.76
14R-1, 120–121	ID	124.20	0.55	—	—	—	–22.84
14R-4, 52–55	ID	128.02	1.42	0.05	28.4	4.01	–23.15
14R-5, 120–121	ID	130.20	0.95	—	—	—	–22.65
14R-6, 50–55	ID	131.03	1.37	0.05	27.4	2.33	–23.06
15R-1, 74–77	ID	133.34	1.03	0.05	20.6	2.94	–23.35
15R-3, 74–77	II	136.29	0.90	0.05	18.0	1.82	–24.95
15R-4, 70–73	II	137.75	0.70	0.04	17.5	1.23	–25.12
16R-1, 96–99	II	143.16	0.93	0.06	15.5	1.88	–24.17
16R-3, 102–107	II	146.22	0.78	0.05	15.6	1.63	–24.27
17R-1, 30–33	II	151.70	1.36	0.09	15.1	2.03	–24.55
17R-3, 30–33	II	154.70	0.66	0.03	22.0	2.04	–23.65
18R-1, 30–33	III	161.30	0.20	0.01	20.0	—	–24.11
18R-2, 30–33	III	162.80	0.22	0.01	22.0	0.18	–24.89
23R-1, 45–46	III	210.00	0.18	—	—	—	–23.26
26R-2, 82–84	III	240.22	0.38	0.02	19.0	0.22	–23.57
28R-1, 39–41	III	275.00	6.64	0.07	94.9	4.65	–23.93
30R-1, 7–8	IV	277.00	3.75	0.12	31.3	5.10	–23.78
31R-1, 47–48	IV	287.00	2.91	0.09	32.3	2.15	–23.84
32R-1, 28–32	IV	295.58	3.81	0.13	29.3	2.07	–24.29
32R-1, 60–64	IV	295.90	4.88	0.10	48.8	2.71	–24.42
32R-1, 90–95	IV	296.20	3.91	0.13	30.1	1.93	–24.53
32R-1, 120–124	IV	296.50	5.03	0.16	31.4	2.25	–24.40
32R-2, 30–34	IV	297.10	5.13	0.17	30.2	2.23	–24.50
32R-2, 60–64	IV	297.40	4.88	0.14	34.9	2.22	–24.55
32R-2, 90–94	IV	297.70	5.88	0.18	32.7	2.23	–24.46
32R-2, 117–121	IV	297.97	4.41	0.14	31.5	1.49	–24.41
32R-CC, 15–19	IV	298.24	4.27	0.12	35.6	2.09	–24.51
33R-CC, 0–5	IV	313.00	4.34	0.08	54.3	1.67	–24.03
34R-1, 30–34	IV	314.20	3.59	0.09	39.9	1.78	–24.12
34R-1, 60–64	IV	314.50	6.70	0.15	44.7	2.36	–24.31
34R-1, 68–69	IV	314.58	4.09	0.10	40.9	1.87	–23.97
34R-1, 90–94	IV	314.80	4.22	0.12	35.2	6.88	–24.42

Notes: — = not analyzed. TOC = elemental total organic carbon, TN = elemental total nitrogen, $\delta^{13}C_{org}$ = stable isotopic organic carbon, PDB = Peedee belemnite standard.

Table T2. *N. pachyderma* (s.) $\delta^{13}C$ and $\delta^{18}O$ stable isotopic results, Site 1166.

Core, section, interval (cm)	Depth (mbsf)	$\delta^{13}C_{PDB}$ (‰)	$\delta^{18}O_{PDB}$ (‰)
188-1166A-			
8R-1, 9–11	65.5	–0.69	4.29
9R-1, 50–53	75.5	–1.61	3.88
10R-1, 17–20	84.6	–2.44	3.93
10R-CC, 9–14	85.1	–0.97	4.51
11R-1, 20–25	94.2	–0.96	3.85
11R-1, 55–60	94.6	–1.29	3.40
11R-1, 80–82	94.8	–2.05	3.47
11R-CC, 9–14	95.5	–2.57	3.84
12R-1, 50–55	104.0	–1.06	3.82
12R-1, 110–115	105.0	–2.53	3.82
12R-1, 19–21	105.4	–1.03	3.88
12R-CC, 5–10	106.0	–1.18	4.16

Note: PDB = Peedee belemnite standard.