
Data report: foraminiferal stable isotope and percent calcium carbonate analysis from IODP Expedition 318 Hole U1361A¹

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Abstract

A newly recognized carbonate preservation event (CPE) dated between 11 and 12 Ma was identified in Hole U1361A during Integrated Ocean Drilling Program Expedition 318 in the Southern Ocean based on CaCO₃ content and the preservation of foraminiferal tests. High carbonate content occurs within the interval between 311.60 and 349.99 meters below seafloor, whereas CaCO₃ content was typically near 1 wt% for the rest of the core. In addition, benthic and planktonic foraminifers were relatively abundant. However, stable isotope measurements carried out within this interval contained unrealistic values, which we ascribe to postdepositional diagenetic alteration by the release of methane. This prohibited quantification of temperature, ice volume, and salinity.

The CPE identified at Hole U1361A was also observed at Ocean Drilling Program Site 689, which is correlated to the Mi5 glaciation. We hypothesize that this Southern Ocean CPE and the diagenetic alteration of foraminifers were due to both the Mi5 glaciation and reorganization of ocean circulation.

Introduction

Integrated Ocean Drilling Program (IODP) Expedition 318 Hole U1361A (64.2457°S, 143.5320°E) is located on the George V Land continental rise (3466 meters below sea level) ~280 km from the coast (Fig. F1). The purpose of drilling this site was to refine the history of climatic and oceanic changes from the middle Miocene to the Pleistocene and to determine the relative stability of the East Antarctic Ice Sheet (EAIS), especially during warm intervals (e.g., Pliocene) (see the “[Site U1361](#)” chapter [Expedition 318 Scientists, 2011b]).

The state of the EAIS after the middle Miocene is a topic of controversy that created two schools of thought among paleoclimate researchers (Miller and Mabin, 1998). “Stabilists” cite evidence that a polar Antarctic ice sheet was established by 14 Ma. In contrast, a considerable amount of evidence suggests a polythermal ice sheet existed from ~33 to 2.5 Ma (Wilson et al., 2002). Current research indicates that the thermal regime of the EAIS varied by location during the Miocene, with a polar EAIS grounded in the Dry Valleys (Marchant et al., 1996; Sugden, 1996) and a polythermal EAIS occupying Wilkes Land and Prydz Bay (Whitehead et al.,

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2006). These differences in thermal regimes are likely due to the EAIS being grounded below sea level at George V Land and Prydz Bay, making them more susceptible to climactic warming (see the “[Site U1361](#)” chapter [Expedition 318 Scientists, 2011b]; Whitehead et al., 2006).

In this study, moderately high resolution stable isotopic ratio (from planktonic and benthic foraminifers) and CaCO₃ content records were developed from samples taken from Cores 318-U1361A-34X through 37X (311.60–349.99 meters below seafloor [mbsf]). The purpose of these data were to evaluate cryospheric and Southern Ocean changes during the middle to late Miocene. The CaCO₃ content helped identify a new carbonate preservation event (CPE) in the Southern Ocean (DeCesare, 2014). However, diagenesis affected the stable isotope records, limiting our interpretation of cryospheric and oceanographic changes during this study interval.

Methods

Sample preparation and foraminifer identification

Sediment samples from Hole U1361A were weighed and then dried in an oven at 50°C for 24 h. The dried samples were soaked in a “Calgon solution” (5.5 g of Fisher Scientific laboratory grade sodium metaphosphate (NaPO₃)₆ mixed with 1 L H₂O) to help break down the sediment. The slurry was decanted into a 63 μm sieve and gently washed with water to flush out silt- and clay-sized particles. The sediment was then separated into 425–355, 355–250, 250–125, and 125–63 μm size fractions by dry sieving. Planktonic and benthic specimens >63 μm were picked under a reflected light binocular microscope with a fine paintbrush and separated by species. Visual inspection of the foraminiferal tests indicated excellent preservation, often with a glassy appearance. The following species were picked for isotopic analysis: *Oridorsalis umbonatus*, *Gyroidinoides soldani*, *Epistominella exigua*, *Cibicidoides wuellerstorfi*, *Cibicidoides bradyi*, and *Globigerina bulloides*. Specimens were placed in double distilled water and sonicated for 5–10 s using a Branson ultrasonic cleaner prior to stable isotope analysis.

Stable isotopes

The cleaned benthic and planktonic foraminifers were first sent to the stable isotope laboratory at Lamont-Doherty Earth Observatory (USA) for δ¹⁸O and δ¹³C analysis. A VG Optima isotope-ratio mass spec-

trometer equipped with a multiprep carbonate preparation module with long-term precision of ±0.07‰ for δ¹⁸O and ±0.05‰ for δ¹³C was used to collect stable isotope data. Foraminifers were also analyzed at the Stable Isotope Laboratory at Rutgers University (USA) using a Micromass Optima mass spectrometer with an attached multiprep device with analytical error (1σ) of ±0.08‰ for δ¹⁸O and ±0.05‰ for δ¹³C.

Foraminifers were reacted in 100% phosphoric acid at 90°C for 10 min, and the evolved CO₂ was collected in a liquid nitrogen cold finger. Ratios are reported in standard delta notation in parts per thousand (per mil; ‰) $d = [(R_{\text{sample}}/R_{\text{standard}}) - 1] \times 1000$ where $R = {}^{18}\text{O}/{}^{16}\text{O}$ or ${}^{13}\text{C}/{}^{12}\text{C}$, relative to Vienna-Pee Dee Belemnite (δ¹⁸O and δ¹³C_{VPDB}) through the analysis of an in-house laboratory standard (RGF1). This standard is routinely calibrated to National Bureau of Standards NBS-19 to ensure consistency in reported values.

Age model

The age model for Hole U1361A was constructed using the geomagnetic Chron C5n.2n (o), at 11.04 Ma and the biostratigraphic datum the first occurrence of *Denticulopsis dimorpha* s.l. at 12.4 Ma (Fig. F2). This resulted in an age range of 11.0–12.0 Ma for the study interval between 311.60 and 349.99 mbsf. In addition, the carbonate preservation event was correlated to a similar preservation event at Ocean Drilling Program Site 689 in the Weddell Sea (DeCesare, 2014), which has a robust magnetostratigraphic age model (Speiß, 1990). This enabled a more precise dating of the event to 11.9–10.5 Ma (DeCesare, 2014).

Calcium carbonate weight percent and methane concentration

For discussion of calcium carbonate weight percent and methane concentration, see the “[Methods](#)” chapter (Expedition 318 Scientists, 2011a).

Results

Calcium carbonate content

During Expedition 318, four Cores (34X–37X; 311.60–349.99 mbsf), averaging 100% recovery, were collected from Hole U1361A. These cores contained the only significant calcium carbonate (CaCO₃) preservation within the Miocene section (Fig. F3). Carbonate preservation was typically <1% for sediments

from Hole U1361A. From 11.7 to 11.0 Ma, CaCO_3 increased to a maximum of 24%, averaging 16.3% (Table T1).

Methane concentration

Methane concentration in Hole U1361A is elevated during the middle to late Miocene with an average concentration of 11,632 ppmv, which is 3.2 times greater than the whole core average (Fig. F3, Table T2). The highest methane concentrations are 14,739 ppmv in Core 35X (325.7 mbsf), 13,826 ppmv in Core 36X (335.3 mbsf), and 15,362 ppmv in Core 38X (353.5 mbsf; Core 38X is not included in this study). Concentrations reach negligible amounts by 162.7 mbsf and remain low to the core top (see the “Site U1361” chapter [Expedition 318 Scientists, 2011b]).

Hole U1361A foraminiferal data

Foraminiferal abundances range from absent to common, and 76% of the 90 samples examined contain foraminifers. Planktonic and benthic foraminifers exhibit good to excellent preservation (Fig. F4), with the majority of benthic foraminifers having a glassy appearance. Benthic foraminifers were identified down to the species level to evaluate paleoenvironmental changes in the middle to late Miocene Southern Ocean (Table T3). Dominant species include *Nuttallides umbonifera*, *Pullenia bulloides*, *O. umbonatus*, *Melonis pompilioides*, *G. soldani*, *E. exigua*, *C. wuellerstorfi*, and *C. bradyi*. Specimens from the genus *Oolina* sp., *Fissurina* sp., *Lagena* spp., and *Sphaeroidina* sp. were also observed.

E. exigua is the most dominant benthic species throughout the majority of the study interval (Table T3); it inhabits cold and well-oxygenated waters with abundance positively correlated with seasonal phytodetritus (Smart, 2008). *Cibicidoides* spp. mainly occurs in the upper portion of the core from 325.35 to 313.23 mbsf and prefers cold waters with low organic matter flux (Poli et al., 2012).

Stable isotopes

Planktonic (*G. bulloides*) and benthic (*O. umbonatus*, *G. soldani*, *E. exigua*, *C. wuellerstorfi*, and *C. bradyi*) foraminifers were first run at Lamont-Doherty Earth Observatory for stable isotopes. The $\delta^{18}\text{O}$ values range from -0.3‰ to 2.4‰ for planktonic foraminifers and 1.3‰ to 3.3‰ for benthic foraminifers (Table T4; Fig. F5), and $\delta^{13}\text{C}$ values range from -15.5‰ to 0.4‰ for planktonic foraminifers and -9.3 to 1.0‰ for benthic foraminifers (Table T5; Fig. F5). A second set of samples were sent to Rutgers University for analysis and yielded similar results, with $\delta^{18}\text{O}$ val-

ues from benthic foraminifers ranging from 0.8‰ to 3.4‰ (Table T4; Fig. F5) and $\delta^{13}\text{C}$ values ranging from -12.1‰ to 1.1‰ (Table T5; Fig. F5).

The extremely negative $\delta^{13}\text{C}$ values suggest that foraminiferal calcite was diagenetically altered by the incorporation of ^{13}C -depleted inorganic carbonate, likely caused by methane seeps at the core site (Martin et al., 2004; Torres et al., 2003; Rathburn et al., 2000). Foraminiferal $\delta^{13}\text{C}$ values decrease to -16‰ at Hole U1361A, whereas Southern Ocean values typically reach 0.4‰ (Wright and Miller, 1993).

Benthic foraminiferal $\delta^{18}\text{O}$ values vary more than 2‰ , which suggests unrealistically extreme changes in ice volume and temperature over short timescales (<100 ky). The dissociation of methane hydrates has been observed to cause alterations in $\delta^{18}\text{O}$ of pore waters (Torres et al., 2003) and likely caused the alteration of foraminiferal $\delta^{18}\text{O}$. Interestingly, these diagenetically altered foraminifers, most of which had a glassy texture, showed little to no indication of overgrowth during visual inspection with a binocular light microscope.

A list of sample requests is found in Table T6.

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Figure F1. Location map of Site U1361 ~280 km off the coast of George V Land (64.2457°S, 143.5320°E) and ODP Site 689 on the Maud Rise in the Weddell Sea (64.5170°S, 3.0999°E).

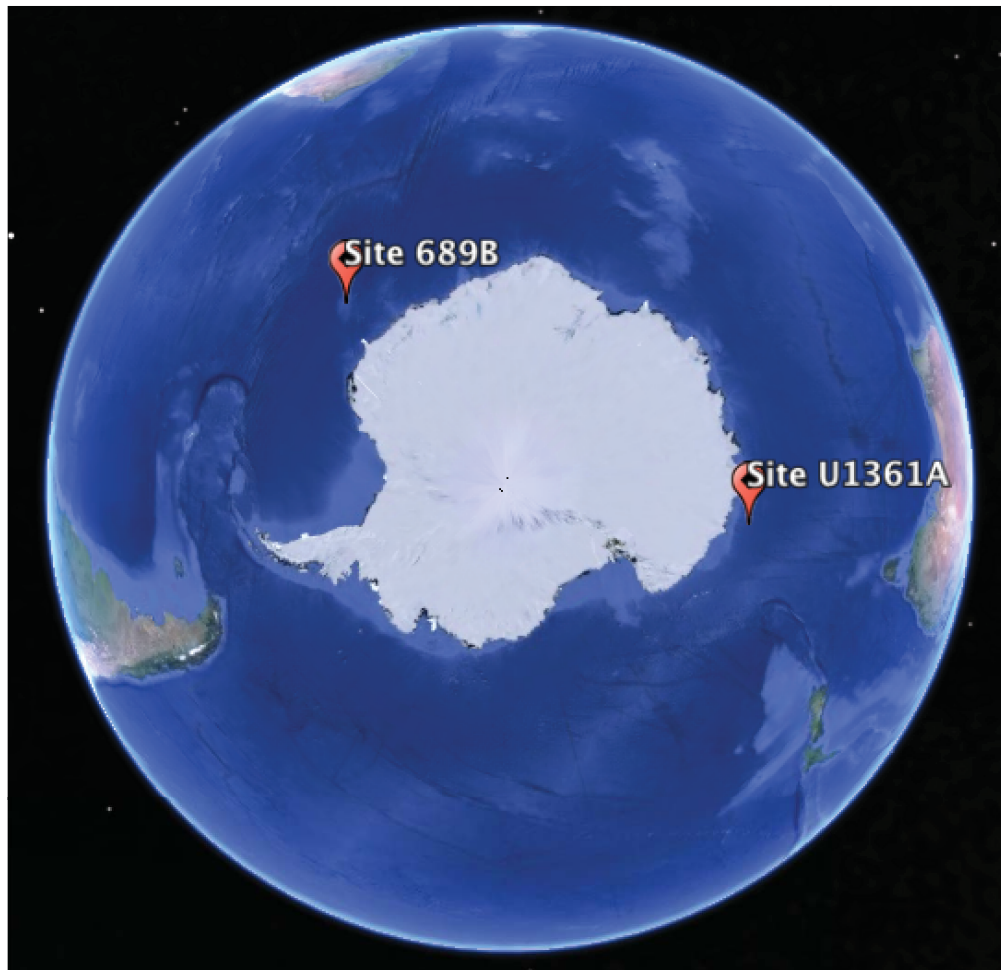


Figure F2. Age model for Hole U1361A from Tauxe et al. (2012) (Copyright 2012 American Geophysical Union), with expanded age model of the study interval using the geomagnetic tie point, Chron C5n.2n (o), at 11.04 Ma and biostratigraphic tie point and first occurrence of *Denticulopsis dimorpha* s.l. at 12.4 Ma.

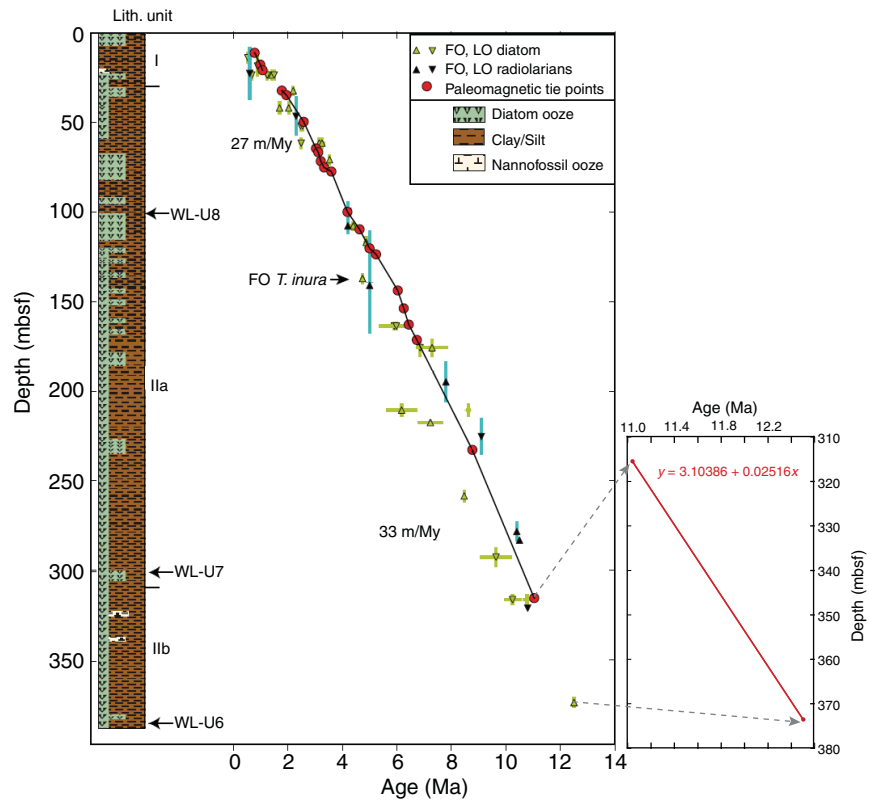


Figure F3. Methane concentrations in ppmv ($\times 10^3$) and CaCO_3 , Hole U1361A. Gray box = study interval. Data from IODP LIMS database.

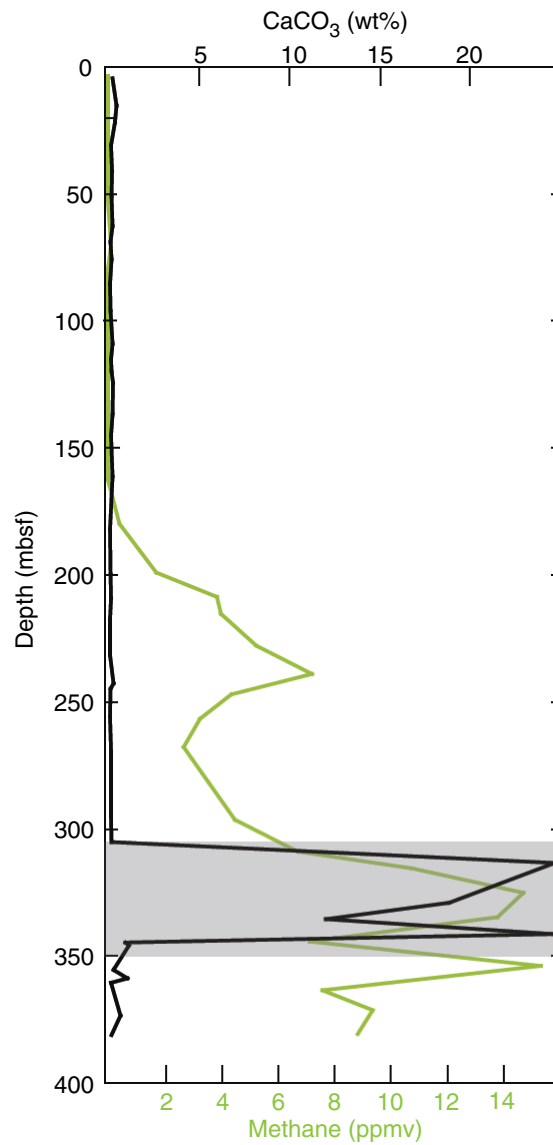


Figure F4. Light microscope images. A. *G. bulloides*. B. *E. exigua*. C. *O. umbonatus*. D. *Melonis* sp. Scale bar = 200 μm .

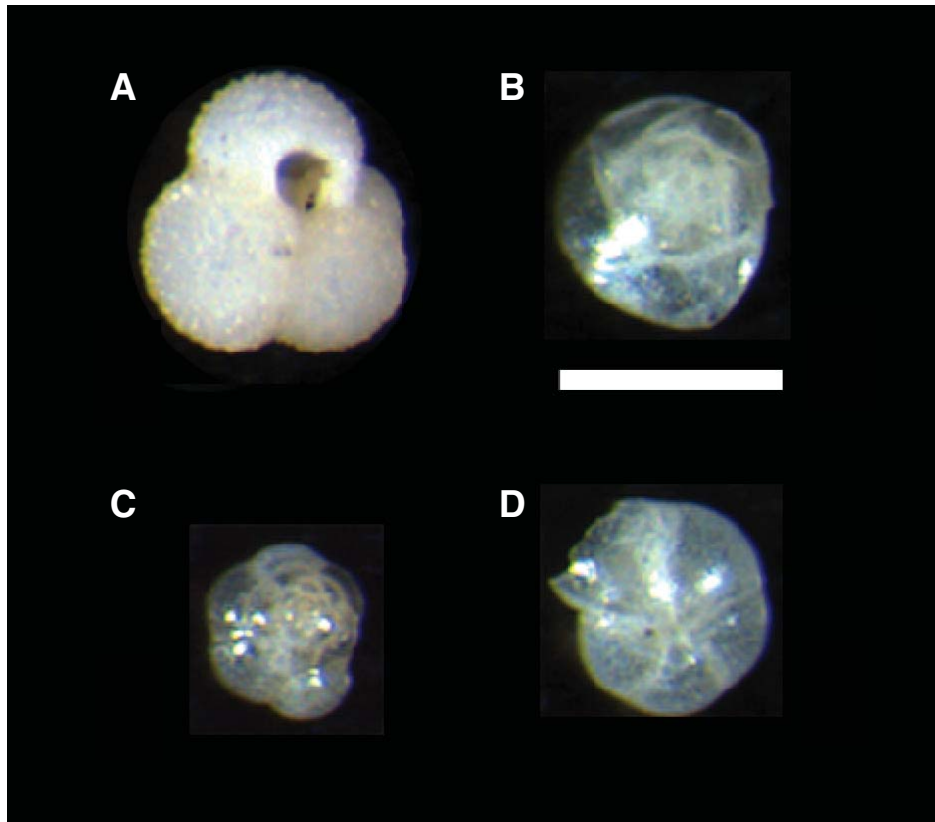


Figure F5. Stable isotope time series (raw data), Hole U1361A. Oxygen isotopes for benthic (red) and planktonic (blue) foraminifers are on the left, and carbon isotopes are on the right.

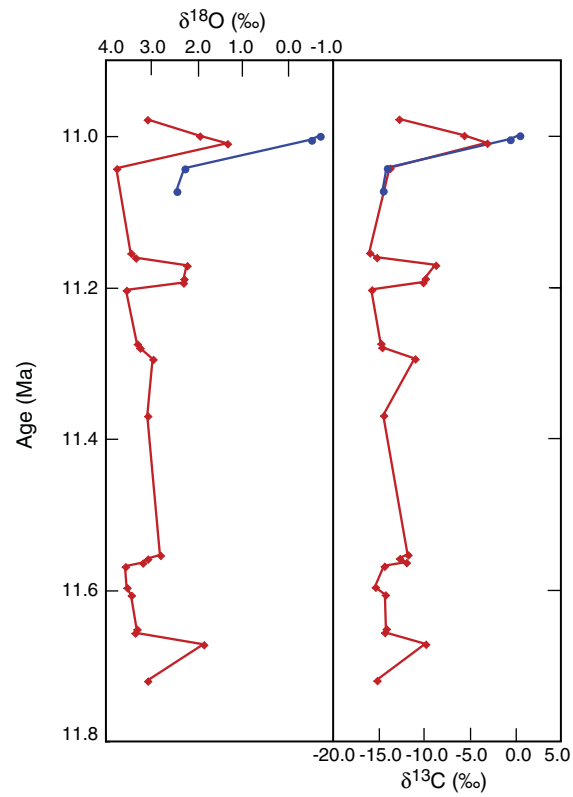


Table T1. CaCO₃, Hole U1361A.

Age (Ma)	Top depth (mbsf)	CaCO ₃ (wt%)
	4.9	0.3
	15.8	0.5
	22.2	0.4
	31.5	0.2
	41.7	0.2
	51.6	0.2
	63.0	0.3
	69.4	0.2
	76.3	0.2
	86.0	0.1
	96.4	0.2
	109.7	0.3
	115.6	0.2
	120.3	0.2
	125.0	0.3
	137.4	0.3
	145.9	0.2
	161.9	0.3
	182.7	0.1
	200.3	0.2
	209.9	0.2
	218.3	0.1
	232.2	0.1
	243.3	0.3
	245.4	0.2
	256.5	0.1
	270.0	0.2
	295.8	0.2
	305.7	0.2
11.00	314.0	24.8
11.40	329.7	18.9
11.56	336.2	12.1
11.71	342.0	24.7
11.79	345.4	1.0
11.82	346.4	1.2
12.06	356.1	0.3
12.14	359.4	1.1
	361.3	0.2
	374.1	0.7
	381.8	0.2

Age control is for the middle to late Miocene. CaCO₃ and depth data are from the IODP LIMS database.

Table T2. Methane (CH₄) concentrations, Hole U1361A.

Age (Ma)	Depth (mbsf)	Methane (ppmv)
	4.5	3
	13.5	3
	23.0	3
	31.0	3
	42.0	3
	51.5	0
	70.5	126
	80.0	8
	89.5	4
	99.0	3
	107.8	4
	118.0	4
	127.5	3
	137.0	3
	146.5	3
	156.0	3
	162.7	3
	180.4	413
	199.6	1,715
	209.2	3,880
	215.8	4,016
	228.4	5,273
	239.5	7,248
	247.6	4,387
	257.2	3,278
	268.3	2,695
	297.0	4,512
	309.5	6,739
	316.1	10,807
11.2	325.7	14,739
11.5	335.3	13,827
11.8	344.9	7,153
12.1	354.5	15,363
	364.1	7,612
	371.9	9,403
	381.4	

Age control is for the middle to late Miocene. CH₄ and depth data are from the IODP LIMS database.



Table T3. Benthic foraminifers, Hole U1361A.

Depth (mbsf)	Age (Ma)	DS1 (%)	DS1	DS2 (%)	DS2	DS3 (%)	DS3	Specimens (N)	Spp. (N)	Weight (g)	Specimens/gram
313.23	11.00	50	<i>Epistominella exigua</i>	33	<i>Cibicidoides</i> spp.	17	<i>Melonis pompilioides</i>	6	3	15.97	0.38
313.43	11.00	48	<i>Epistominella exigua</i>	28	<i>Sphaeroidina</i> sp.			60	9	16.74	3.58
313.63	11.01	43	<i>Epistominella exigua</i>	29	<i>Cibicidoides</i> spp.	14	<i>Oridorsalis umbonatus</i>	7	4	17.25	0.41
313.83	11.01	12	<i>Lagena</i> spp.	9	<i>Cibicidoides</i> spp.			114	13	19	6.00
314.05	11.02	7	<i>Lagena</i> spp.	6	<i>Cibicidoides</i> spp., <i>Epistominella exigua</i>			165	16	18.53	8.90
319.89	11.16	15	<i>Nuttallides umbonifera</i>	12	<i>Oridorsalis umbonatus</i>	11	<i>Lagena</i> spp.	105	13	15.52	6.77
320.65	11.18	15	<i>Cibicidoides</i> spp.	13	<i>Nuttallides umbonifera</i> , <i>Pullenia bulloides</i> , <i>Fissurina</i> sp.			86	13	15.4	5.58
321.93	11.22	24	<i>Epistominella exigua</i>	19	<i>Cibicidoides</i> spp.	14	<i>Sphaeroidina</i> sp., <i>Melonis pompilioides</i>	21	8	15.08	1.39
322.85	11.24	100	<i>Epistominella exigua</i>					1	1	14.8	0.07
323.62	11.26							0	0	15.32	0.00
324.55	11.28	18	<i>Melonis pompilioides</i> , <i>Fissurina</i> sp.	14	<i>Pullenia bulloides</i> , <i>Epistominella exigua</i>			21	10	14.41	1.46
325.35	11.30	39	<i>Cibicidoides</i> spp.	12	<i>Pullenia bulloides</i> , <i>Oridorsalis umbonatus</i>			98	16	16.66	5.88
325.63	11.31							0	0	14.83	0.00
327.04	11.34							0	0	15.55	0.00
327.88	11.36	20	<i>Pullenia bulloides</i>	19	<i>Oolina</i> spp.	13	<i>Lagena</i> spp., <i>Fissurina</i> sp., <i>Oridorsalis umbonatus</i>	16	8	17.58	0.91
328.89	11.39							0	0	17.71	0.00
329.58	11.41							0	0	18.72	0.00
336.05	11.57	29	<i>Oridorsalis umbonatus</i>	15	<i>Nuttallides umbonifera</i> , <i>Sphaeroidina</i> sp.	13	<i>Cibicidoides</i> spp.	192	17	15.67	12.25
336.9	11.59							0	0	16.86	0.00
337.75	11.61	29	<i>Epistominella exigua</i>	14	<i>Oridorsalis umbonatus</i> , <i>Gyroidinoides soldani</i> , <i>Fissurina</i> sp.			21	8	18.03	1.16
338.67	11.64	29	<i>Epistominella exigua</i> , <i>Melonis pompilioides</i>	14	<i>Fissurina</i> sp.			7	4	16.56	0.42
339.56	11.66	52	<i>Epistominella exigua</i>	24	<i>Melonis pompilioides</i>	14	<i>Pullenia bulloides</i>	21	4	19.86	1.06
340.55	11.68	55	<i>Epistominella exigua</i>	13	<i>Oridorsalis umbonatus</i>			91	13	14.23	6.39
341.35	11.70	25	<i>Oridorsalis umbonatus</i> , <i>Nuttallides umbonifera</i> , <i>Melonis pompilioides</i> , <i>Oolina</i> sp.					4	4	13.55	0.30
342.25	11.73	52	<i>Epistominella exigua</i>	18	<i>Oridorsalis umbonatus</i>	10	<i>Fissurina</i> sp.	33	8	14.72	2.24

DS1, DS2, DS3 = benthic foraminifers categorized by the highest percentage of dominant species. *E. exigua* is highly abundant through the study interval. Abundance of *Cibicidoides* spp. increases toward upper half of the study interval.

Table T4. Oxygen isotope ($\delta^{18}\text{O}$, raw data) separated by benthic and planktonic foraminifers, Hole U1361A.

Species	$\delta^{18}\text{O}$ (‰)	Depth (mbsf)
Benthic		
<i>Oridorsalis umbonatus</i>	3.08	312.95
<i>Cibicoides bradyi</i>	1.93	313.85
<i>Gyroidinoides soldani</i>	1.22	314.23
<i>Cibicoides wuellerstorfi</i>	1.42	314.23
<i>Cibicoides bradyi</i>	3.75	315.55
<i>Cibicoides wuellerstorfi</i>	3.43	320.01
<i>Cibicoides</i> spp.	3.33	320.25
<i>Cibicoides wuellerstorfi</i>	2.24	320.65
<i>Cibicoides wuellerstorfi</i>	2.14	320.65
<i>Cibicoides wuellerstorfi</i>	2.25	320.65
<i>Cibicoides bradyi</i>	2.40	321.35
<i>Cibicoides wuellerstorfi</i>	2.15	321.35
<i>Oridorsalis umbonatus</i>	2.34	321.53
<i>Oridorsalis umbonatus</i>	2.45	321.53
<i>Epistominella exigua</i>	2.07	321.53
<i>Cibicoides wuellerstorfi</i>	3.54	321.93
<i>Cibicoides wuellerstorfi</i>	3.30	324.75
<i>Cibicoides wuellerstorfi</i>	3.24	324.95
<i>Cibicoides wuellerstorfi</i>	2.96	325.55
<i>Cibicoides wuellerstorfi</i>	3.07	328.54
<i>Oridorsalis umbonatus</i>	2.79	335.85
<i>Oridorsalis umbonatus</i>	3.05	336.05
<i>Oridorsalis umbonatus</i>	3.08	336.05
<i>Gyroidinoides soldani</i>	3.34	336.26
<i>Oridorsalis umbonatus</i>	2.98	336.26
<i>Cibicoides wuellerstorfi</i>	3.21	336.26
<i>Cibicoides wuellerstorfi</i>	3.49	336.45
<i>Cibicoides bradyi</i>	3.62	336.45
<i>Epistominella exigua</i>	3.54	337.55
<i>Cibicoides wuellerstorfi</i>	3.50	337.55
<i>Epistominella exigua</i>	3.59	337.95
<i>Oridorsalis umbonatus</i>	3.26	337.95
<i>Epistominella exigua</i>	3.47	339.76
<i>Oridorsalis umbonatus</i>	3.14	339.76
<i>Epistominella exigua</i>	3.34	339.96
<i>Epistominella exigua</i>	1.84	340.55
<i>Cibicoides bradyi</i>	3.07	342.46
Planktonic		
<i>Globigerina bulloides</i>	-0.72	313.85
<i>Globigerina bulloides</i>	-0.24	314.03
<i>Globigerina bulloides</i>	-0.81	314.03
<i>Globigerina bulloides</i>	2.26	315.55
<i>Globigerina bulloides</i>	2.44	316.72

Table T5. Carbon isotope ($\delta^{13}\text{C}$, raw data) separated by benthic and planktonic foraminifers, Hole U1361A.

Species	$\delta^{13}\text{C}$ (‰)	Depth (mbsf)
Benthic		
<i>Oridorsalis umbonatus</i>	-2.19	312.95
<i>Cibicoides bradyi</i>	-9.31	313.85
<i>Gyroidinoides soldani</i>	-12.11	314.23
<i>Cibicoides wuellerstorfi</i>	-11.61	314.23
<i>Cibicoides bradyi</i>	-1.19	315.55
<i>Cibicoides wuellerstorfi</i>	1.02	320.01
<i>Cibicoides</i> spp.	0.26	320.25
<i>Cibicoides wuellerstorfi</i>	-5.98	320.65
<i>Cibicoides wuellerstorfi</i>	-6.59	320.65
<i>Cibicoides wuellerstorfi</i>	-6.02	320.65
<i>Cibicoides bradyi</i>	-5.12	321.35
<i>Cibicoides wuellerstorfi</i>	-5.00	321.35
<i>Oridorsalis umbonatus</i>	-4.21	321.53
<i>Oridorsalis umbonatus</i>	-4.23	321.53
<i>Epistominella exigua</i>	-6.00	321.53
<i>Cibicoides wuellerstorfi</i>	0.82	321.93
<i>Cibicoides wuellerstorfi</i>	-0.21	324.75
<i>Cibicoides wuellerstorfi</i>	-0.33	324.95
<i>Cibicoides wuellerstorfi</i>	-3.93	325.55
<i>Cibicoides wuellerstorfi</i>	-0.48	328.54
<i>Oridorsalis umbonatus</i>	-3.17	335.85
<i>Oridorsalis umbonatus</i>	-2.10	336.05
<i>Oridorsalis umbonatus</i>	-2.37	336.05
<i>Gyroidinoides soldani</i>	-4.76	336.26
<i>Oridorsalis umbonatus</i>	-3.06	336.26
<i>Cibicoides wuellerstorfi</i>	-1.16	336.26
<i>Cibicoides wuellerstorfi</i>	-0.43	336.45
<i>Cibicoides bradyi</i>	-0.69	336.45
<i>Epistominella exigua</i>	-0.30	337.55
<i>Cibicoides wuellerstorfi</i>	1.11	337.55
<i>Epistominella exigua</i>	-0.18	337.95
<i>Oridorsalis umbonatus</i>	-1.19	337.95
<i>Epistominella exigua</i>	-0.44	339.76
<i>Oridorsalis umbonatus</i>	-1.14	339.76
<i>Epistominella exigua</i>	-0.60	339.96
<i>Epistominella exigua</i>	-5.10	340.55
<i>Cibicoides bradyi</i>	0.23	342.46
Planktonic		
<i>Globigerina bulloides</i>	-15.45	313.85
<i>Globigerina bulloides</i>	-13.38	314.03
<i>Globigerina bulloides</i>	-15.35	314.03
<i>Globigerina bulloides</i>	-0.86	315.55
<i>Globigerina bulloides</i>	-0.43	316.72

