Mosher, D.C., Erbacher, J., and Malone, M.J. (Eds.)
Proceedings of the Ocean Drilling Program, Scientific Results Volume 207

6. Data Report: Stable Isotope Ratios of Foraminifers from ODP Leg 207, Sites 1257, 1258, and 1260, and a Cleaning Procedure for Foraminifers in Organic-Rich Shales[1]

Karen L. Bice[2] and Richard D. Norris[3]

ABSTRACT

We report oxygen and carbon stable isotope analyses of foraminifers, primarily planktonic, sampled at low resolution in the Cretaceous and Paleogene sections from Sites 1257, 1258, and 1260. Data from two samples from Site 1259 are also reported. The very low resolution of the data only allows us to detect climate-driven isotopic events on the timescale of more than 500 k.y. A several million-year-long interval of overall increase in planktonic $\delta^{18}O$ is seen in the Cenomanian at Site 1260. Before and after this interval, foraminifers from Cenomanian and Turonian black shales have $\delta^{18}O$ values in the range –4.2‰ to –5.0‰, suggestive of upper ocean temperatures higher than modern tropical values. The $\delta^{18}O$ values of upper ocean dwelling Paleogene planktonics exhibit a long-term increase from the early Eocene to the middle Eocene.

During shipboard and postcruise processing, it proved difficult to extract well-preserved foraminifer tests from black shales by conventional techniques. Here, we report results of a test of procedures for cleaning foraminifers in Cretaceous organic-rich mudstone sediments using various combinations of soaking in bleach, Calgon/hydrogen peroxide, or Cascade, accompanied by drying, repeat soaking, or sonication. A procedure that used 100% bleach, no detergent, and no sonication yielded the largest number of clean, whole individual foraminifers with the

[1] Bice, K.L., and Norris, R.D., 2005. Data report: Stable isotope ratios of foraminifers from ODP Leg 207, Sites 1257, 1258, and 1260 and a cleaning procedure for foraminifers in organic-rich shales. *In* Mosher, D.C., Erbacher, J., and Malone, M.J. (Eds.), *Proc. ODP, Sci. Results,* 207, 1–23 [Online]. Available from World Wide Web: <http://www-odp.tamu.edu/publications/207_SR/VOLUME/CHAPTERS/104.PDF>. [Cited YYYY-MM-DD]
[2] Department of Geology and Geophysics, Woods Hole Oceanographic Institution, Mail Stop 23, 119 Clark Laboratory, Woods Hole MA 02543, USA. **kbice@whoi.edu**
[3] Scripps Institution of Oceanography, MS-0244, 308 Vaughan Hall, La Jolla CA 92093, USA

Initial receipt: 3 January 2005
Acceptance: 24 March 2005
Web publication: 22 July 2005
Ms 207SR-104

shortest preparation time. We found no significant difference in $\delta^{18}O$ or $\delta^{13}C$ values among sets of multiple samples of the planktonic foraminifer *Whiteinella baltica* extracted following each cleaning procedure.

INTRODUCTION

One of the principal objectives of Ocean Drilling Program (ODP) Leg 207 was to recover Cretaceous and Paleogene strata to reconstruct the history of low-latitude sea-surface temperatures. Studies of well-preserved Cenomanian and Turonian planktonic foraminifers from Deep Sea Drilling Project (DSDP) Site 144 (redrilled as Site 1257 during Leg 207) can be interpreted as evidence for unusually warm surface ocean temperatures, conservatively calculated between 30°C and 35°C (Norris et al., 2002; Wilson et al. 2002). However, the absence of continuously cored sequences at DSDP Site 144 prevented determination of the long-term record of tropical planktonic $\delta^{18}O$, particularly in the Turonian–Campanian. Accordingly, we report a low-resolution record from Leg 207 sites of the tropical Atlantic Ocean through the Cretaceous and Paleogene.

The combination of poor preservation of carbonates and the ubiquitous occurrence of several hiatuses prevented us from analyzing a complete isotopic record of Cenomanian–Eocene sediments. Nonetheless, during Leg 207 an expanded sequence of Eocene and Paleocene carbonates, as well as Cenomanian–Santonian black shales were recovered. Campanian and Maastrichtian sections were also recovered, but poor preservation of calcareous microfossils restricts their utility for studies of ancient seawater properties. Fortunately, very well preserved to excellently preserved microfossils could be obtained from the black shales and Paleogene. The only major impediment to producing the Cretaceous part of this record is the difficulty extracting well-preserved fossils from the organic-rich black shales. Conventional processing techniques of drying and crushing samples and soaking in deionized water prior to sieving proved to produce foraminifers coated with bits of clay and organic matrix. Therefore, we experimented with a variety of cleaning techniques and tested the cleaned foraminifers to determine whether these cleaning methods had any effect on our stable isotope results.

METHODS

Samples of carbonate sediments (one per core) were taken in the shipboard sediment laboratory. At Woods Hole Oceanographic Institution (WHOI; USA), these samples were oven dried, gently crushed, soaked for 2 hr in a 3% Calgon-hydrogen peroxide solution, and washed over a 63-µm sieve with warm tap water (pH = 6). Wet samples were dried in an oven at 45°C for 2 hr. Cretaceous organic-rich mudstone ("black shale") samples were cut from core on the *JOIDES Resolution* catwalk and frozen shipboard at –80°C. Black shale samples were later freeze dried and partly crushed using a porcelain mortar and pestle. Following a test of cleaning procedures (see the **"Appendix,"** p. 6), crushed black shale sediments were soaked in undiluted Clorox bleach for 2 hr, washed, and dried, and then soaked for 1 hr in undiluted bleach, washed, and dried.

For planktonics, single-species stable isotope analyses were made of 1 to 25 individual whole specimens. For benthics, 1 to 5 individuals of single species or mixed species were analyzed. Oxygen and carbon isotope ratios were measured on a Finnigan MAT 252 mass spectrometer with an automated Kiel carbonate device at WHOI. Instrument precision is ± 0.07‰ for $\delta^{18}O$ and ±0.03‰ for $\delta^{13}C$. Results are reported relative to the Vienna Peedee belemnite (VPDB) isotope standard and are given in Table T1.

We estimated the age of our sample set through a combination of shipboard biostratigraphy and shore-based magnetostratigraphy (Erbacher, Mosher, Malone, et al., 2004). The time-scale is that of the Leg 207 shipboard party (see references therein).

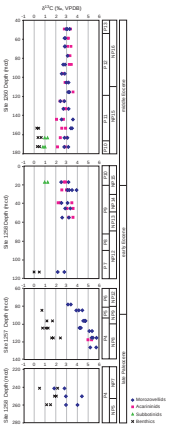
T1. Stable isotope results, p. 14.

MICROFOSSIL PRESERVATION AND SAMPLE TEMPORAL RESOLUTION

Foraminifer preservation quality in samples analyzed ranges from moderate to excellent in Paleocene and Eocene samples. All Paleogene planktonic foraminifers are white, rather than translucent, and most show (other than where noted) tabular microstructure and no obvious chamber infillings or overgrowths. At Site 1260, the frequent occurrence of sugary surface textures in middle Eocene foraminifers suggests some degree of recrystallization, but no infilling or encrusting secondary calcite was noted in samples picked for stable isotope measurements. The approximate temporal resolution of Paleogene samples with preservation deemed good enough for isotopic analyses is 500 k.y. from Sites 1257 and 1260 and 1 m.y. from Site 1258. For Cretaceous samples analyzed, resolution is much coarser, ranging from ~1 m.y. at Site 1260 to 4 m.y. at Site 1257.

Sample preservation in the Cretaceous samples analyzed was generally good to excellent. In two Cretaceous samples (207-1260B-37R-1, 10–16 cm, and 207-1260B-39R-1, 115–122 cm), spar infilling was observed in the outermost chamber of about 10% of *Hedbergella delrioensis*. These individuals were avoided when picking for stable isotope analyses. Foraminifers with excellent preservation display translucent tests without obvious overgrowths or recrystallization. Such foraminifers display surface features such as pores, keels, and pustules that look similar to those on modern foraminifers captured in plankton tows. In contrast, foraminifers with good preservation were mostly opaque (usually white) but without evidence of recrystallization, overgrowth, or infilling. Foraminifers with moderate preservation also show no evidence of infilling or recrystallization, but do display a sugary surface texture that may be evidence of minor overgrowth.

F1. Paleogene foraminifer carbon isotope ratios, p. 10.



STABLE ISOTOPE RESULTS

The Paleogene planktonic genera *Morozovella* and *Acarinina* have similar oxygen and carbon isotope values within samples, suggesting little or no relative depth stratification between these species. The most enriched $\delta^{13}C$ values occur in the upper Paleocene planktonic foraminifer Zone P4 (nannofossil Zone NP8) interval (Fig. F1). Here, planktonic $\delta^{13}C$ values range 5‰–6‰ VPDB. The Site 1257 data exhibit a decrease in $\delta^{13}C$ to near 3‰ in the upper Paleocene and upper Eocene. From

planktonic foraminifer Zone P9 (nannofossil Zone NP13) upward, the low-resolution carbon isotope record exhibits little structure and no overall trend.

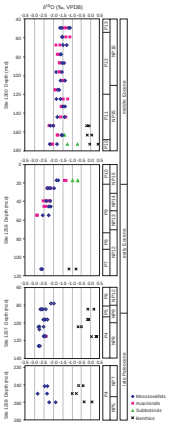
The low-resolution $\delta^{18}O$ record for the Paleogene (Fig. F2) has values from approximately –2.4‰ to –2.9‰ from planktonic foraminifer Zones P4–P9 (nannofossil Zones NP7–NP13). The oxygen isotope record of *Morozovella* species shows an overall increase of ~1‰ from Zone P9 to Zone P13, consistent with either a 1‰ increase in local water $\delta^{18}O$ or a 5°C decrease in upper ocean temperatures (Erez and Luz, 1983) over this interval.

The low-resolution Cretaceous $\delta^{13}C$ record (Fig. F3) exhibits no certain discernible trends. The Hole 1260B $\delta^{18}O$ record (Fig. F4) shows first an increase of values from approximately –4.3‰ to –4.5‰ low in the Cenomanian (Section 207-1260B-34R-2), to approximately –3.3‰ above (Section 37R-1). The lowest planktonic $\delta^{18}O$ values we obtained in the Cretaceous black shales were found in Sample 207-1260B-34R-2, 10–17 cm. Because of the extraordinarily low values obtained here, five analyses of the species *H. delrioensis* and *Heterohelix globulosa* were made in this sample. *H. delrioensis* has $\delta^{18}O$ ranging from –4.5‰ to –5.0‰ VPDB, with an average of –4.8‰. *H. globulosa* tests in the same sample have an average $\delta^{18}O$ of –4.7‰. The average for each species is more depleted than the lowest $\delta^{18}O$ values obtained by Wilson et al. (2002) from very well preserved Turonian samples taken previously on Demerara Rise. Data from DSDP Site 144 were interpreted by Wilson et al. (2002) as indicating upper ocean temperatures 3°–6°C higher than modern sea-surface temperatures. Given the same assumptions regarding water $\delta^{18}O$ values and pH, our Leg 207 $\delta^{18}O$ data suggest even higher tropical temperatures in the mid-Cretaceous.

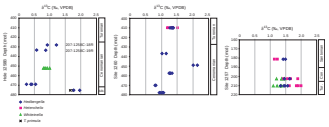
ACKNOWLEDGMENTS

This research used samples and/or data provided by the Ocean Drilling Program (ODP). ODP is sponsored by the U.S. National Science Foundation (NSF) and participating countries under management of Joint Oceanographic Institutions (JOI), Inc. Funding for this research was provided by the U.S. Science Support Program of JOI. Helen Sturt provided sections of frozen sediment samples taken for organic geochemical analyses. Lu Ping Zou assisted in sample preparation and cleaning procedure testing. This is WHOI publication number 11022.

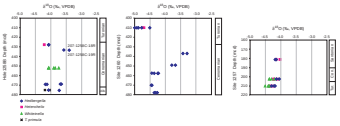
F2. Paleogene foraminifer oxygen isotope ratios, p. 11.



F3. Cretaceous foraminifer carbon isotope ratios, p. 12.



F4. Cretaceous foraminifer oxygen isotope ratios, p. 13.



REFERENCES

Erbacher, J., Mosher, D.C., Malone, M.J., et al., 2004. *Proc. ODP, Init. Repts.,* 207 [Online]. Available from World Wide Web: <http://www-odp.tamu.edu/publications/207_IR/207ir.htm>. [Cited 2004-12-13]

Erez, J., and Luz, B., 1983. Experimental paleotemperature equation for planktonic foraminifera. *Geochim. Cosmochim. Acta,* 47:1025–1031.

Gaffey, S.J., and Bronnimann, C.E., 1993. Effects of bleaching on organic and mineral phases in biogenic carbonates. *J. Sediment. Petrol.,* 63:752–754.

Norris, R.D., Bice, K.L., Magno, E.A., and Wilson, P.A., 2002. Jiggling the tropical thermostat in the Cretaceous hothouse. *Geology,* 30:299–302.

Pingitore, N.E., Jr., Fretzdorff, S.B., Seitz, B.P. Estrada, L.Y., Borrego, P.M., Crawford, G.M, and Love, K.M., 1993. Dissolution kinetics of $CaCO_3$ in common laboratory solvents. *J. Sediment. Petrol.,* 63:641–645.

Shipboard Scientific Party, 2004a. Site 1258. *In* Erbacher, J., Mosher, D.C., Malone, M.J., et al., *Proc. ODP, Init. Repts.,* 207 [Online]. Available from World Wide Web: <http://www-odp.tamu.edu/publications/207_IR/chap_05/chap_05.htm>. [Cited 2004-12-13]

Shipboard Scientific Party, 2004b. Site 1259. *In* Erbacher, J., Mosher, D.C., Malone, M.J., et al., *Proc. ODP, Init. Repts.,* 207 [Online]. Available from World Wide Web: <http://www-odp.tamu.edu/publications/207_IR/chap_06/chap_06.htm>. [Cited 2004-12-13]

Wilson, P.A., Norris, R.D., and Cooper, M.J., 2002. Testing the Cretaceous greenhouse hypothesis using glassy foraminiferal calcite from the core of Turonian tropics on Demerara Rise. *Geology,* 30:607–610.

APPENDIX

Procedure for Cleaning Cretaceous Organic-Rich Mudstones for Foraminiferal Stable Isotope Analysis

Introduction

Examination of Leg 207 black shales indicates that abundant material with very good to excellent preservation of mid-Cretaceous foraminifers can be analyzed for paleoceanographic data. However, past attempts to separate clean tests from the organic-rich mudstones using a Calgon-hydrogen peroxide solution have proved time consuming and generally unsatisfactory. We describe here a series of cleaning experiments performed to determine which solvents and steps are most effective at disaggregating organic mudstone clasts, separating foraminifer tests from the matrix, and removing dark matter from the surface of foraminifers. For our purposes, it was most important that the oxygen and carbon stable isotope ratios are not affected by the cleaning procedure.

Methodology

Experiments were performed on splits of a large (~60 g dry weight) black shale sample (Sample 207-1258B-51R-2, 13–20 cm) that remained after aliquots were taken for lipid analyses. Samples from nearby composite depths in this hole that were measured shipboard contained 9.5–12.4 wt% total organic carbon (Shipboard Scientific Party, 2004a). The sediments are Cenomanian in age and are assigned to planktonic foraminiferal Zone KS19. The freeze-dried, crushed sample was homogenized to minimize biases among subsequent splits. Cleaning tests were performed on ~5-g sample splits in 50–60 mL of solvent. The four cleaning procedures are given in Table AT1. Washing was done with warm tap water (pH = 6) through a 63-µm sieve. Wet samples were dried in an oven at 45°C for 1–2 hr.

AT1. Cleaning procedures, p. 21.

After final drying, 15–20 individual whole specimens of *Whiteinella baltica* were picked from the >150-µm size fraction from each procedure. In general, foraminiferal preservation in Sample 207-1258B-51R-2, 13–20 cm, is moderate to good. This particular interval does not exhibit the very good and excellent preservation that has been observed in other Demerara Rise Cenomanian and Turonian samples, but it was the only sample available to us that had sufficient volume for multiple procedures. An attempt was made to select the cleanest, best preserved examples of *W. baltica* from each procedure. These were then divided to yield five stable isotope measurements from each cleaning procedure. Oxygen and carbon isotope ratios were measured on a Finnigan MAT 252 mass spectrometer with an automated Kiel carbonate device at WHOI. Instrument precision is ±0.07‰ for $\delta^{18}O$ and ±0.03‰ for $\delta^{13}C$. The solvent in Procedure 1 is a 3% Calgon-hydrogen peroxide solution that has been used for soaking most Cretaceous and Paleogene carbonate sediment samples processed in the WHOI paleoceanography group's laboratory. For that reason, foraminifers picked from the Procedure 1 sample are taken as our control for comparison of isotopes.

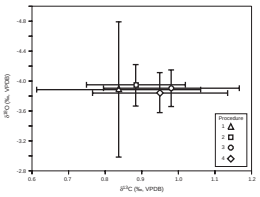
Results

Our qualitative assessment of the relative effectiveness of the cleaning procedures runs in reverse order to the procedure numbers: Procedure 4 yielded the cleanest sample; Procedure 1 retained the most organic matter. Procedure 1 also resulted in the most flakes of clay and organic compounds adhering to foraminifers and significantly higher dry weights than Procedure 4. Visual examination of the tests revealed no major difference in preservation quality, although sonicated samples tended to have a higher proportion of broken specimens and samples washed with the Calgon-peroxide solution were more prone to foraminifer breakage because of the need to physically rub the clay chips though the screen. In contrast, samples soaked in bleach did not require physical disaggregation during washing, particularly if two cycles of soaking, washing, and drying were employed.

Results of the 20 stable isotope measurements on specimens from Procedures 1–4 are given in Table AT2 and are reported relative to the Vienna Peedee belemnite (VPDB) isotope standard. The differences among the mean $\delta^{18}O$ or $\delta^{13}C$ values are not significant at the 95% confidence level (Fig. AF1). The variance in oxygen isotope values from Procedure 1 (our control) is an order of magnitude greater than that for Procedures 2, 3, and 4. However, additional tests would be required to determine if this is a robust observation and whether the difference in variance is significant.

AT2. Stable isotope results, p. 22.

AF1. *Whiteinella* sp. oxygen/carbon values, p. 19.



Discussion and Further Experiments

For Procedures 1 and 3, many large (>3 mm) grains remained even after the second soak (15 hr) and wash. A second sonication disaggregated most of these grains, but the final coarse fraction percentages for Procedures 1 and 3 (Table AT1) are lower than those for the procedures using bleach. This may be the result of fragmentation during sonication or the very long second soak. Although sonication quickly produced very clean foraminifers, it is our opinion that sonication should be avoided because of possible breakage. Under the binocular microscope, it appears that the smallest grains remaining in Procedure 1 are primarily individual chambers. In contrast, the smallest size fraction for Procedure 4 (no sonication) contains abundant whole shells. The only sample that disaggregated well without sonication was that in Procedure 4 (100% bleach).

In addition to the 2-hr/1-hr soak in 100% bleach (Procedure 4), we also tested a 1-hr/1-hr soak procedure with 100% bleach, in order to see if processing time could be decreased. However, cleaning results were not as satisfactory as those obtained with Procedure 4. We note that, regardless of the solvent used, the intermediate drying stage is critical in obtaining the greatest disaggregation possible: most of the disaggregation occurs in the second soak/wash cycle.

It should be noted that hydrogen peroxide, an ingredient in the solution used in Procedure 1, is strongly corrosive to calcium carbonate (Pingitore et al., 1993). While fragmentation during sonication may help account for the very low coarse fraction remaining in the Procedure 1 sample, it is also possible that the acidity (pH = 6) of the Calgon (sodium hexametaphosphate) and hydrogen peroxide solution causes dissolution of some grains. To better identify the primary cause of low final material weight and to test the procedures on a more carbonate rich black shale interval, we performed a set of experiments similar to

Procedures 1 and 4. The Calgon-peroxide and 100% bleach experiments were repeated. For both solvents, samples were soaked for 3 hr, washed, dried, soaked for 1 hr, washed, dried, and weighed (Table AT3). The black shale samples used in this case were taken from Hole 1259C, containing 6–9 wt% total organic carbon (Shipboard Scientific Party, 2004b). Sample 207-1259C-11R-4, 130–150 cm, is from the Santonian, near the top of the Leg 207 black shale sequence. Preservation is good to very good. Foraminifer tests are opaque, but there is no evidence of mineral infilling or overgrowths. Sample 207-1259C-17R-1, 116–136 cm, is Turonian in age and occurs below the distinctive glauconitic claystone seen in the Turonian interval at Sites 1259, 1260, and 1261. This Turonian sample contains several thin carbonate stringers and preservation of foraminifers ranges from poor to moderate. Calcite spar infilling is common in this sample.

AT3. Calgon-peroxide vs. bleach results, p. 23.

Again, the 100% bleach solvent produced a clean, well-disaggregated sample without sonication (Fig. AF2). Because many large mudstone grains still remained in the samples soaked in Calgon solution, these sediments were wetted with Calgon solution for 5 min, sonicated for 90 s, washed, dried, and reweighed. The reduction in coarse fraction weight percent (Table AT3) for the Calgon-peroxide solution in this second experiment was not nearly as great as that observed in Procedure 1 (Table AT1), either before or after sonication. This suggests that the very low coarse fraction in Procedure 1 may have resulted from soaking in the Calgon-peroxide solution overnight. To check the effect of long-term soaking in Calgon-peroxide, the Hole 1259C samples were soaked overnight (15 hr) in the solution, washed, dried, and weighed a third time (Table AT3). For both samples, the percent reduction in coarse fraction after the 15-hr soak was approximately equal to that caused by sonicating for 90 s.

AF2. Dried sediments, p. 20.

It is clear from our second set of experiments that different black shales will exhibit varying degrees of disaggregation with the same procedure. For the Hole 1259C Santonian sample, this final soak (15 hr) resulted in an acceptably clean sample, one from which many clean, well-preserved tests could be picked fairly easily. However, in the Hole 1259C Turonian sample, which had a higher percentage of secondary carbonate cement ("stringers"), the sample left was predominantly shale and cement fragments. Besides a few foraminifers visible encased within shale matrix, almost no tests remained. In fact, this Turonian sample did not disaggregate as well as either the Hole 1258B Cenomanian shale or the Hole 1259C Santonian shale in either the Calgon or bleach solvents. However, for all three shales, the bleach soaking procedure produced a more than sufficient number of loose, clean foraminifers for isotope work. We saw no samples in which it was necessary to soak overnight in bleach to obtain a well-disaggregated sample, perhaps because the intermediate drying stage we used is so effective at causing shale grains to fall apart. However, the very high pH of the bleach suggests that no loss of carbonate material is likely to occur through dissolution if samples are soaked overnight in bleach.

In some regions, Clorox brand regular bleach may not be available. In the United States, for example, many stores carry a less expensive bleach that is 5.25%, rather than 6% sodium hypochlorite. We therefore repeated Procedure 4 in a side-by-side test using the Hole 1258B Cenomanian sample with Clorox bleach (6% NaOCl), America's Choice bleach (5.25% NaOCl) and Stop & Shop bleach (5.25% NaOCl). The difference in cleaning power between the two concentrations of sodium hypochlorite is readily apparent, with the Clorox bleach producing a

cleaner sample. Because we do not know the composition of the 94.75% inert ingredients listed for the weaker bleaches, we can not say if it is the concentration of NaOCl or the inert ingredients themselves that is critical to obtaining a clean sample with bleach. While we recommend that the 6% NaOCl bleach be used, if it is available, the 5.25% bleach produced a sample that was acceptably clean for picking foraminifers.

There is reason to think that use of a 20% bleach solution (Procedure 2) should be avoided because diluting bleach lowers its pH. Gaffey and Bronnimann (1993) examined the textural effects of bleach on modern biogenic carbonates and determined that the increase in concentration of hypochlorous acid in dilute bleach solutions caused minor pitting of skeletal material. We did not bother to examine our Procedure 2 samples for pitting because this procedure yielded the second worst result in terms of organic matter removal and has no other advantages.

As with any chemical, laboratory personnel should be familiar with material safety information for bleach. If undiluted bleach is used in the laboratory, care should be taken to avoid reaction with agents that will produce chlorine gas. Having the beakers in a hood while soaking was sufficient to prevent bleach odors in the laboratory. Prolonged contact of bleach with some metals in the laboratory may cause pitting and discoloration. We noticed no effects of the beach on stainless steel (including sieves) in the laboratory when processing the black shale samples. However, we observed that ODP Leg 207 nannofossil chalks that contain small amounts of oxidized iron (such as Sample 207-1258B-39R-5, 115–135 cm) caused minor amounts of iron oxides to be deposited on the sieve wire mesh during drying in the oven. This occurred if the chalks were soaked in bleach but not when they were soaked in the Calgon-peroxide solution. We believe that the higher solubility of iron oxides in the high-pH (>11) bleach allowed dissolution of oxides in the beaker while soaking; these oxides were reprecipitated on the sieve while drying, even though the sediments were washed thoroughly. These nannofossil chalk samples disaggregated much better in the Calgon-peroxide solution than in the bleach, apparently because of dissolution of intergranular carbonate cements in the pH 6 solution. Based on these experiments, we caution that soaking in bleach should be performed only on organic-rich mudstone samples and not on chalk samples.

Conclusions

There are no significant differences among the oxygen or carbon isotope ratios obtained for *Whiteinella baltica* from the four cleaning procedures given in Table AT1. However, Procedure 4 yielded the best result in terms of cleaning foraminifer tests. Soaking in 100% bleach produced a well-disaggregated sample, with little organic matter adhered to tests and the lowest amount of fragmentation. The low mass of material remaining at the end of Procedure 1, as well as the near loss of all foraminifers in a second test, suggests that corrosion of biogenic carbonates may occur with prolonged soaking in a Calgon and hydrogen peroxide solution. Although frequently used for disaggregating carbonate-rich samples, this solution was the least effective in removing organic matter from the Cenomanian black shale sample.

Figure F1. Carbon isotope ratios in Paleogene foraminifers.

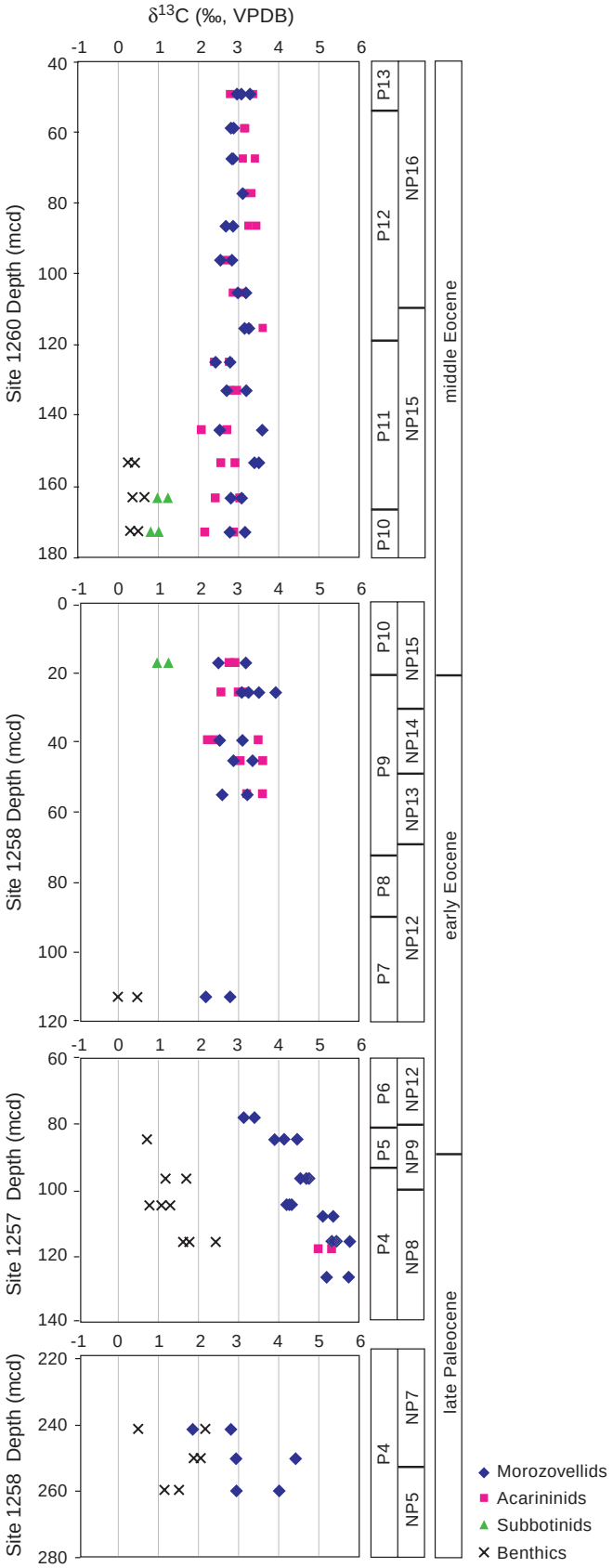
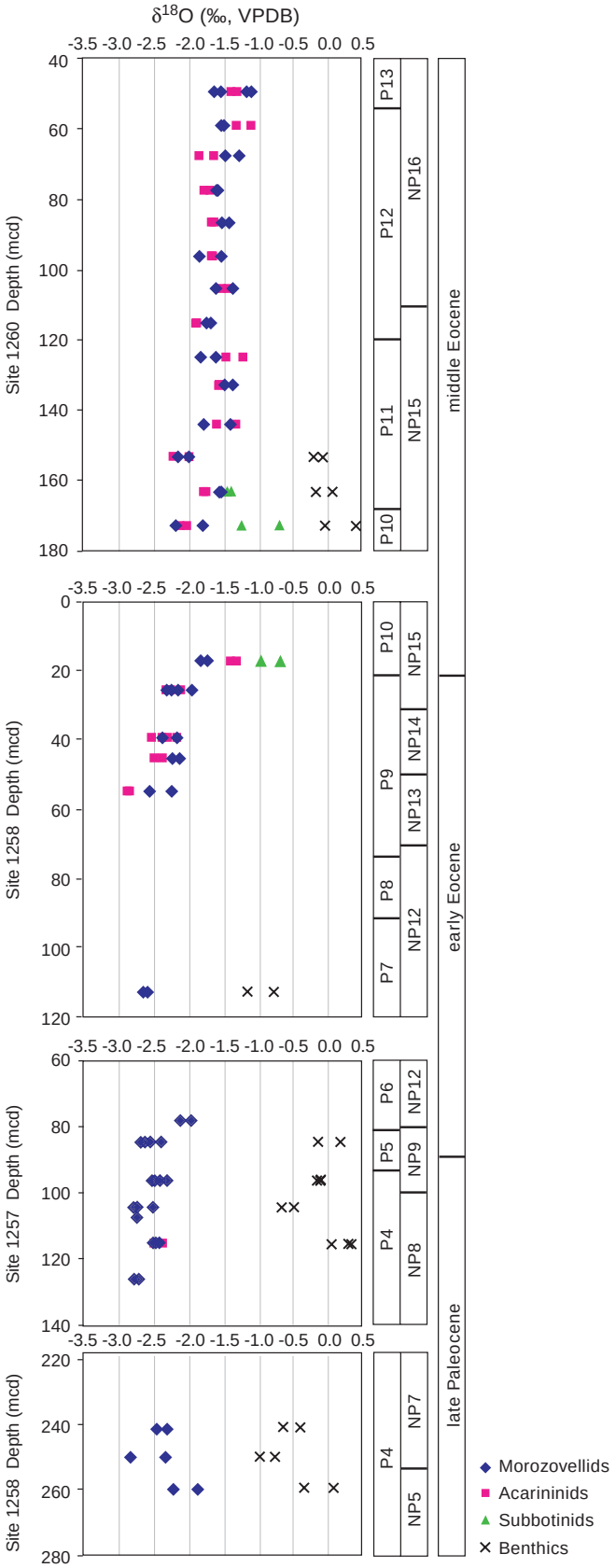


Figure F2. Oxygen isotope ratios in Paleogene foraminifers.



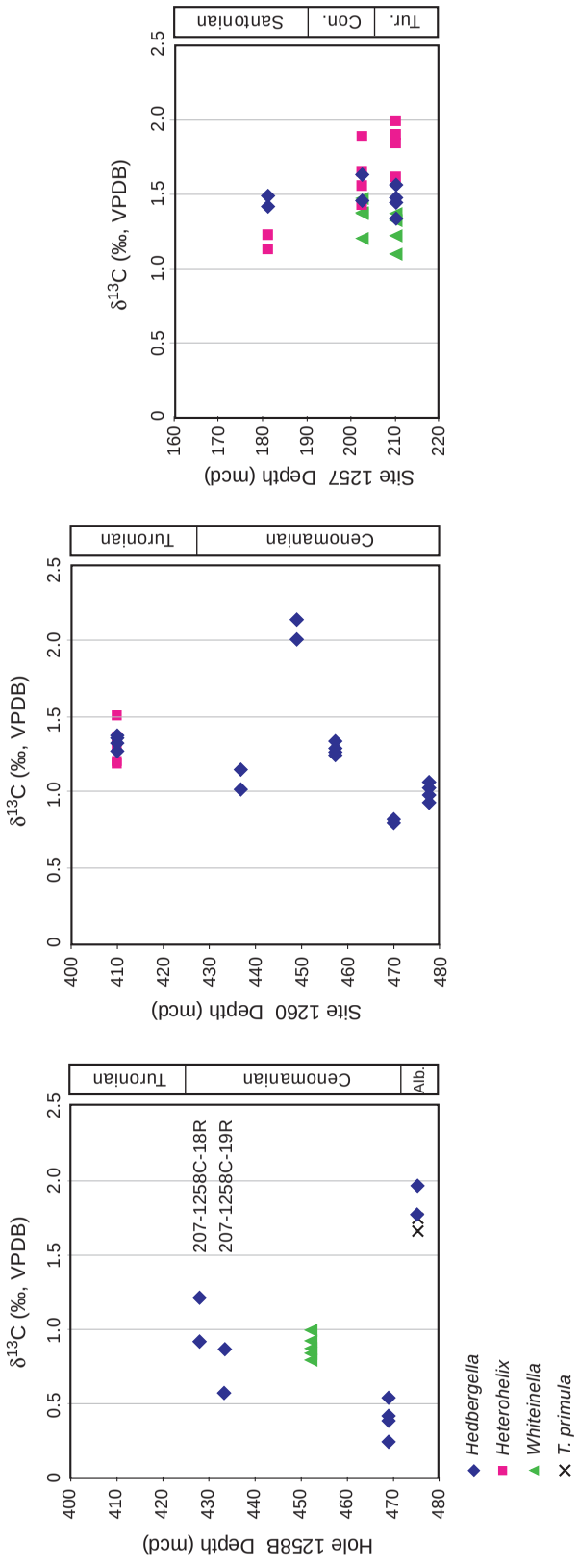


Figure F3. Carbon isotope ratios in Cretaceous foraminifers.

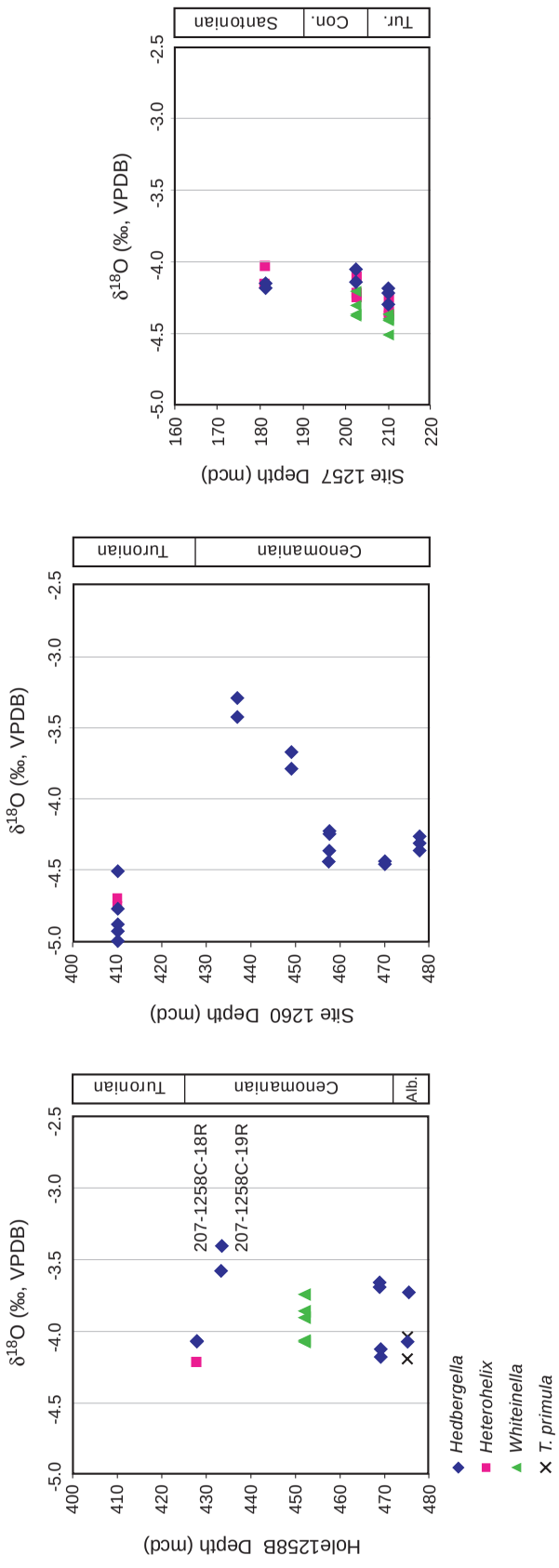


Figure F4. Oxygen isotope ratios in Cretaceous foraminifers.

Table T1. Stable isotope results. (See table notes. Continued on next four pages.)

Core, section, interval (cm)	Depth (mbsf)	Depth (mcd)	Foraminifer zone	Nannofossil zone	Age	Species	Size fraction (μ)	Preservation	$\delta^{103}C$ (VPDB) (‰)	$\delta^{18}O$ (VPDB) (‰)
207-1257A-										
10X-4, 39–41	78.20	78.20	P6	NP12	EEOC	*M. crassata*	>250	VG	3.4	–2.1
10X-4, 39–41	78.20	78.20	P6	NP12	EEOC	*M. crassata*	>250	VG	3.1	–2.0
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*M. crassata*	>250	VG	4.4	–2.7
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*M. crassata*	>250	VG	3.9	–2.4
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*M. velascoensis*	>250	VG	4.1	–2.6
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*M. velascoensis*	>250	VG	4.1	–2.5
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*Mixed benthics*	>250	VG	0.7	–0.1
11X-2, 15–17	84.50	84.50	P5	NP9	EEOC	*Mixed benthics*	>250	VG	0.7	0.2
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	*M. crassata*	>250	VG	4.7	–2.4
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	*M. crassata*	>250	VG	4.6	–2.3
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	*M. velascoensis*	>250	VG	4.5	–2.5
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	*M. velascoensis*	>250	VG	4.7	–2.5
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	*Cibicidoides* sp.	>250	VG	1.7	–0.1
12X-3, 62–64	96.20	96.20	P4	NP9	LPAL	Mixed benthics	>250	VG	1.2	–0.1
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	*M. velascoensis*	>250	G	4.3	–2.8
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	*M. velascoensis*	>250	G	4.2	–2.7
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	*M. velascoensis*	>250	G	4.2	–2.7
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	*M. velascoensis*	>250	G	4.2	–2.5
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	Mixed benthics	>250	G	0.8	–0.7
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	Mixed benthics	>250	G	1.3	–0.6
13X-2, 60–62	104.30	104.30	P4	NP8	LPAL	Mixed benthics	>250	G	1.0	–0.5
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*M. crassata*	>250	VG	5.8	–2.5
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*M. crassata*	>250	VG	5.3	–2.4
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*M. velascoensis*	>250	VG	5.4	–2.5
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*M. velascoensis*	>250	VG	5.3	–2.5
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	Mixed benthics	>250	VG	2.4	0.1
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	Mixed benthics	>250	VG	1.7	0.3
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	Mixed benthics	>250	VG	1.6	0.3
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*Acarinina* sp.	>250	VG	5.3	–2.5
14X-3, 38–40	115.20	115.20	P4	NP8	LPAL	*Acarinina* sp.	>250	VG	5.0	–2.4
207-1257C–										
3R-3, 130–150	105.50	107.50	P4	NP8	LPAL	*M. velascoensis*	>250	G	5.1	–2.7
3R-3, 130–150	105.50	107.50	P4	NP8	LPAL	*M. velascoensis*	>250	G	5.3	–2.7
5R-2, 130–150	123.30	126.00	P4	NP8	LPAL	*M. velascoensis*	>250	G	5.7	–2.8
5R-2, 130–150	123.30	126.00	P4	NP8	LPAL	*M. velascoensis*	>250	G	5.2	–2.7
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*H. delrioensis*	150–250	E	1.5	–4.2
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*H. delrioensis*	150–250	E	1.4	–4.2
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*H. globulosa*	212–250	E	1.2	–4.1
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*H. globulosa*	212–250	E	1.1	–4.0
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*Marginotruncana sinuosa*	>250	E	1.5	–4.2
11R-1, 84–89	178.84	181.40		CC15–16	SAN	*Marginotruncana sinuosa*	>250	E	1.9	–3.9
13R-2, 120–140	200.00	202.60		CC13	CON	*H. delrioensis*	150–250	E	1.5	–4.1
13R-2, 120–140	200.00	202.60		CC13	CON	*H. delrioensis*	150–250	E	1.6	–4.1
13R-2, 120–140	200.00	202.60		CC13	CON	*H. globulosa*	212–250	E	1.9	–4.2
13R-2, 120–140	200.00	202.60		CC13	CON	*H. globulosa*	212–250	E	1.7	–4.2
13R-2, 120–140	200.00	202.60		CC13	CON	*H. globulosa*	>250	E	1.4	–4.1
13R-2, 120–140	200.00	202.60		CC13	CON	*H. globulosa*	>250	E	1.6	–4.2
13R-2, 120–140	200.00	202.60		CC13	CON	*W. baltica*	>250	E	1.5	–4.4
13R-2, 120–140	200.00	202.60		CC13	CON	*W. baltica*	>250	E	1.4	–4.3
13R-2, 120–140	200.00	202.60		CC13	CON	*W. baltica*	>250	E	1.2	–4.2
13R-2, 120–140	200.00	202.60		CC13	CON	*W. baltica*	>250	E	1.4	–4.4
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. delrioensis*	150–250	E	1.6	–4.3
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. delrioensis*	150–250	E	1.4	–4.2
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. delrioensis*	150–212	E	1.5	–4.2
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. delrioensis*	150–212	E	1.3	–4.2
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. globulosa*	212–250	E	1.8	–4.3
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. globulosa*	212–250	E	1.9	–4.3
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. globulosa*	150–212	E	2.0	–4.3
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*H. globulosa*	150–212	E	1.6	–4.2
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*W. baltica*	>250	E	1.3	–4.5
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*W. baltica*	>250	E	1.2	–4.4
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*W. baltica*	>250	E	1.4	–4.4
14R-1, 76–82	207.76	210.30		CC10–11	TUR	*W. baltica*	>250	E	1.1	–4.4
207-1258A-										
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*M. aragonensis*	>300	G	2.5	–1.8
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*M. aragonensis*	>300	G	3.1	–1.7

Table T1 (continued).

Core, section, interval (cm)	Depth (mbsf)	Depth (mcd)	Foraminifer zone	Nannofossil zone	Age	Species	Size fraction (μ)	Preservation	$\delta^{103}C$ (VPDB) (‰)	$\delta^{18}O$ (VPDB) (‰)
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*G. nuttali*	>250	G	1.8	–0.8
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*G. nuttali*	>250	G	1.9	–0.7
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*S. boweri*	>250	G	1.2	–0.9
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*S. boweri*	>250	G	0.9	–0.7
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*A. bullbrooki*	>250	G	2.9	–1.4
3R-3, 6–8	17.30	17.30	P10	NP15	MEOC	*A. bullbrooki*	>250	G	2.7	–1.3
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*M. aragonensis*	250–300	G	3.2	–2.3
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*M. aragonensis*	250–300	G	3.5	–2.2
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*M. aragonensis*	>300	G	3.9	–2.1
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*M. aragonensis*	>300	G	3.1	–1.9
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*I. broedermanni*	250–300	G	3.3	–2.1
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*I. broedermanni*	250–300	G	3.0	–2.0
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*A. bullbrooki*	250–300	G	3.1	–2.3
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*A. bullbrooki*	250–300	G	2.5	–2.2
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*A. pentacamerata*	>300	G	3.1	–2.3
4R-2, 40–42	25.70	25.70	P9	NP15	EEOC	*A. pentacamerata*	>300	G	3.0	–2.1
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*M. aragonensis*	>300	G	2.5	–2.3
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*M. aragonensis*	>300	G	3.1	–2.2
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*A. pentacamerata*	>300	G	3.5	–2.5
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*A. pentacamerata*	>300	G	3.5	–2.4
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*A. soldadoensis*	>300	G	2.3	–2.3
5R-2, 18–20	34.88	39.40	P9	NP14	EEOC	*A. soldadoensis*	>300	G	2.4	–2.1
6R-2, 40–42	44.70	45.20	P9	NP14	EEOC	*M. aragonensis*	>250	VG	3.3	–2.2
6R-2, 40–42	44.70	45.20	P9	NP14	EEOC	*M. aragonensis*	>250	VG	2.8	–2.1
6R-2, 40–42	44.70	45.20	P9	NP14	EEOC	*A. pentacamerata*	>250	VG	3.5	–2.5
6R-2, 40–42	44.70	45.20	P9	NP14	EEOC	*A. pentacamerata*	>250	VG	3.0	–2.4
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*M. aragonensis*	>212	G	3.2	–2.5
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*M. aragonensis*	>212	G	2.6	–2.2
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*P. micra*	>212	G	1.9	–1.8
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*P. micra*	>212	G	1.7	–1.7
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*A. pentacamerata*	>212	G	3.1	–2.9
7R-2, 15–17	53.85	54.80	P9	NP13	EEOC	*A. pentacamerata*	>212	G	3.6	–2.8
13R-2, 20–22	112.20	112.81	P7	NP12	EEOC	*M. aragonensis*	>250	M	2.7	–2.6
13R-2, 20–22	112.20	112.81	P7	NP12	EEOC	*M. aragonensis*	>250	M	2.1	–2.6
13R-2, 20–22	112.20	112.81	P7	NP12	EEOC	Mixed benthics	>250	M	0.0	–1.1
13R-2, 20–22	112.20	112.81	P7	NP12	EEOC	Mixed benthics	>250	M	0.4	–0.8
24R-2, 21–23	218.01	241.44	P4	NP7	LPAL	*M. aragonensis*	>250	G	2.7	–2.4
24R-2, 21–23	218.01	241.44	P4	NP7	LPAL	*M. aragonensis*	>250	G	1.8	–2.3
24R-2, 21–23	218.01	241.44	P4	NP7	LPAL	*Cibicidoides* sp.	>250	G	0.4	–0.6
24R-2, 21–23	218.01	241.44	P4	NP7	LPAL	*Cibicidoides* sp.	>250	G	2.1	–0.4
25R-2, 20–22	227.70	250.15	P4	NP7	LPAL	*M. aragonensis*	>250	VG	4.4	–2.8
25R-2, 20–22	227.70	250.15	P4	NP7	LPAL	*M. aragonensis*	>250	VG	2.9	–2.3
25R-2, 20–22	227.70	250.15	P4	NP7	LPAL	*Cibicidoides* sp.	>250	VG	1.8	–0.7
25R-2, 20–22	227.70	250.15	P4	NP7	LPAL	Mixed benthics	>250	VG	1.9	–1.0
26R-2, 20–22	237.30	259.75	P4	NP5	LPAL	*M. aragonensis*	>250	G	3.9	–2.2
26R-2, 20–22	237.30	259.75	P4	NP5	LPAL	*M. aragonensis*	>250	G	2.9	–1.9
26R-2, 20–22	237.30	259.75	P4	NP5	LPAL	Mixed benthics	>250	G	1.4	–0.3
26R-2, 20–22	237.30	259.75	P4	NP5	LPAL	Mixed benthics	>250	G	1.1	0.1
207-1258B-										
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.8	–4.4
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–4.2
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.0	–4.1
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.0	–4.1
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–4.1
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.7	–4.0
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.2	–4.0
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–4.0
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.0	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.8	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.8	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–3.9
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.0	–3.8
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.8	–3.7
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–3.7
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.1	–3.7
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	1.0	–3.6

Table T1 (continued).

Core, section, interval (cm)	Depth (mbsf)	Depth (mcd)	Foraminifer zone	Nannofossil zone	Age	Species	Size fraction (μ)	Preservation	$\delta^{103}C$ (VPDB) (‰)	$\delta^{18}O$ (VPDB) (‰)
51R-2–1, 10–20	427.85	452.04	KS19		CEN	*W. baltica*	>150	G	0.9	–3.2
54R-3, 10–30	444.48	468.96			CEN	*H. delrioensis*	150–250	E	0.4	–3.7
54R-3, 10–30	444.48	468.96			CEN	*H. delrioensis*	150–250	E	0.2	–3.7
54R-3, 10–30	444.48	468.96			CEN	*H. delrioensis*	150–212	E	0.5	–4.1
54R-3, 10–30	444.48	468.96			CEN	*H. delrioensis*	150–212	E	0.4	–4.2
55R-3, 68–88	448.37	475.20		NC9a	LALB	*H. delrioensis*	150–250	E	1.9	–4.1
55R-3, 68–88	448.37	475.20		NC9a	LALB	*H. delrioensis*	150–250	E	1.8	–3.7
55R-3, 68–88	448.37	475.20		NC9a	LALB	*Ticinella primula*	150–250	E	1.7	–4.2
55R-3, 68–88	448.37	475.20		NC9a	LALB	*Ticinella primula*	150–250	E	1.7	–4.0
207-1258C-										
18R-1, 48–51	428.69	404.58		CC10b	CEN	*H. delrioensis*	>150	G	1.2	–4.1
18R-1, 48–51	428.69	404.58		CC10b	CEN	*H. delrioensis*	>150	G	0.9	–4.1
18R-1, 48–51	428.69	404.58		CC10b	CEN	*Heterohelix* sp.	100–150	G	0.1	–4.2
18R-1, 48–51	428.69	404.58		CC10b	CEN	*Heterohelix* sp.	100–150	G	0.4	–4.1
19R-1, 83–89	434.04	409.93		CC10b	CEN	*H. delrioensis*	>150	M	0.9	–3.6
19R-1, 83–89	434.04	409.93		CC10b	CEN	*H. delrioensis*	>150	M	0.6	–3.4
207-1259A-										
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	*M. acuta*	>250	G	5.3	–2.7
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	*M. acuta*	>250	G	5.2	–2.7
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	*M. velascoensis*	>250	G	4.9	–2.9
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	*M. velascoensis*	>250	G	5.5	–2.8
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	*Cibicidoides* sp.	212–250	G	2.9	–0.5
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	Mixed benthics	>250	G	2.3	–0.4
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	Mixed benthics	212–250	G	2.1	–0.3
44R-2, 20–22	412.10	413.27	P4	NP7–8	LPAL	Mixed benthics	>250	G	2.3	–0.1
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	*M. acuta*	>250	G	5.4	–3.0
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	*M. acuta*	>250	G	5.0	–2.9
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	*M. velascoensis*	>250	G	5.3	–2.9
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	*M. velascoensis*	>250	G	5.0	–2.6
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	Mixed benthics	>250	G	2.1	–0.1
45R-1, 20–22	420.20	421.68	P3b	NP7–8	LPAL	Mixed benthics	>250	G	2.0	0.3
207-1260A-										
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*M. crassata*	>250	G	3.3	–1.6
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*M. crassata*	>250	G	3.0	–1.5
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*M. lehneri*	>250	G	3.0	–1.1
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*M. lehneri*	>250	G	3.0	–1.1
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*A. rohri*	>250	G	3.3	–1.4
7R-2, 20–22	49.30	49.30	P13	NP16	MEOC	*A. rohri*	>250	G	2.8	–1.3
8R-2, 20–22	59.00	59.00	P12	NP16	MEOC	*M. crassata*	>250	G	2.8	–1.5
8R-2, 20–22	59.00	59.00	P12	NP16	MEOC	*M. crassata*	>250	G	2.8	–1.5
8R-2, 20–22	59.00	59.00	P12	NP16	MEOC	*A. rohri*	>250	G	3.1	–1.3
8R-2, 20–22	59.00	59.00	P12	NP16	MEOC	*A. rohri*	>250	G	3.1	–1.1
9R-2, 20–22	68.60	67.60	P12	NP16	MEOC	*M. lehneri*	>250	G	2.9	–1.5
9R-2, 20–22	68.60	67.60	P12	NP16	MEOC	*M. lehneri*	>250	G	2.8	–1.2
9R-2, 20–22	68.60	67.60	P12	NP16	MEOC	*A. rohri*	>250	G	3.4	–1.8
9R-2, 20–22	68.60	67.60	P12	NP16	MEOC	*A. rohri*	>250	G	3.1	–1.6
10R-2, 20–22	78.30	77.40	P12	NP16	MEOC	*M. lehneri*	>250	G	3.1	–1.6
10R-2, 20–22	78.30	77.40	P12	NP16	MEOC	*M. lehneri*	>250	G	3.1	–1.6
10R-2, 20–22	78.30	77.40	P12	NP16	MEOC	*A. rohri*	>250	G	3.1	–1.8
10R-2, 20–22	78.30	77.40	P12	NP16	MEOC	*A. rohri*	>250	G	3.3	–1.7
11R-2, 20–22	88.00	86.60	P12	NP16	MEOC	*M. lehneri*	>250	G	2.8	–1.5
11R-2, 20–22	88.00	86.60	P12	NP16	MEOC	*M. lehneri*	>250	G	2.7	–1.4
11R-2, 20–22	88.00	86.60	P12	NP16	MEOC	*A. rohri*	>250	G	3.4	–1.6
11R-2, 20–22	88.00	86.60	P12	NP16	MEOC	*A. rohri*	>250	G	3.3	–1.6
12R-2, 20–22	97.60	96.20	P12	NP16	MEOC	*M. lehneri*	>250	G	2.8	–1.8
12R-2, 20–22	97.60	96.20	P12	NP16	MEOC	*M. lehneri*	>250	G	2.5	–1.5
12R-2, 20–22	97.60	96.20	P12	NP16	MEOC	*A. rohri*	>250	G	2.7	–1.6
12R-2, 20–22	97.60	96.20	P12	NP16	MEOC	*A. rohri*	>250	G	2.7	–1.6
13R-2, 20–22	107.30	105.30	P12	NP16	MEOC	*M. lehneri*	>250	G	3.0	–1.6
13R-2, 20–22	107.30	105.30	P12	NP16	MEOC	*M. lehneri*	>250	G	3.2	–1.3
13R-2, 20–22	107.30	105.30	P12	NP16	MEOC	*A. rohri*	>250	G	2.9	–1.5
13R-2, 20–22	107.30	105.30	P12	NP16	MEOC	*A. rohri*	>250	G	3.1	–1.4
14R-2, 20–22	117.00	115.30	P12	NP15	MEOC	*M. lehneri*	>250	G	3.2	–1.7
14R-2, 20–22	117.00	115.30	P12	NP15	MEOC	*M. lehneri*	>250	G	3.2	–1.7
14R-2, 20–22	117.00	115.30	P12	NP15	MEOC	*A. rohri*	>250	G	3.6	–1.9
14R-2, 20–22	117.00	115.30	P12	NP15	MEOC	*A. rohri*	>250	G	3.3	–1.9
15R-2, 20–22	126.60	124.79	P11	NP15	MEOC	*M. lehneri*	>250	G	2.4	–1.8

Table T1 (continued).

Core, section, interval (cm)	Depth (mbsf)	Depth (mcd)	Foraminifer zone	Nannofossil zone	Age	Species	Size fraction (μ)	Preservation	$\delta^{103}C$ (VPDB) (‰)	$\delta^{18}O$ (VPDB) (‰)
15R-2, 20–22	126.60	124.79	P11	NP15	MEOC	*M. lehneri*	>250	G	2.8	–1.6
15R-2, 20–22	126.60	124.79	P11	NP15	MEOC	*A. rohri*	>250	G	2.7	–1.4
15R-2, 20–22	126.60	124.79	P11	NP15	MEOC	*A. rohri*	>250	G	2.4	–1.2
16R-1, 20–22	134.70	132.89	P11	NP15	MEOC	*M. spinulosa*	>250	G	3.2	–1.5
16R-1, 20–22	134.70	132.89	P11	NP15	MEOC	*M. spinulosa*	>250	G	2.7	–1.3
16R-1, 20–22	134.70	132.89	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.8	–1.5
16R-1, 20–22	134.70	132.89	P11	NP15	MEOC	*A. bullbrooki*	>250	G	3.0	–1.5
17R-2, 20–22	145.90	144.09	P11	NP15	MEOC	*M. aragonensis*	>250	G	3.6	–1.8
17R-2, 20–22	145.90	144.09	P11	NP15	MEOC	*M. aragonensis*	>250	G	2.5	–1.4
17R-2, 20–22	145.90	144.09	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.7	–1.6
17R-2, 20–22	145.90	144.09	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.1	–1.3
18R-2, 12–14	155.12	153.30	P11	NP15	MEOC	*M. aragonensis*	>250	G	3.4	–2.1
18R-2, 12–14	155.12	153.30	P11	NP15	MEOC	*M. lehneri*	>250	G	3.5	–2.0
18R-2, 12–14	155.12	153.31	P11	NP15	MEOC	*N. truempyi*	>250	G	0.2	–0.2
18R-2, 12–14	155.12	153.31	P11	NP15	MEOC	*N. truempyi*	>250	G	0.3	0.0
18R-2, 12–14	155.12	153.31	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.9	–2.2
18R-2, 12–14	155.12	153.31	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.6	–2.0
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*M. aragonensis*	>250	G	2.8	–1.5
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*M. aragonensis*	>250	G	3.1	–1.5
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*N. truempyi*	>250	G	0.3	–0.2
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*N. truempyi*	>250	G	0.6	0.1
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*S. boweri*	>250	G	1.0	–1.4
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*S. boweri*	>250	G	1.2	–1.4
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*A. bullbrooki*	>250	G	2.4	–1.8
19R-2, 22–24	164.92	163.11	P11	NP15	MEOC	*A. bullbrooki*	>250	G	3.0	–1.7
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*M. aragonensis*	>250	G	3.2	–2.2
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*M. aragonensis*	>250	G	2.8	–1.8
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*N. truempyi*	>250	G	0.5	0.0
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*N. truempyi*	>250	G	0.2	0.4
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*S. boweri*	>250	G	0.8	–1.2
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*S. boweri*	>250	G	1.0	–0.7
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*A. bullbrooki*	>250	G	2.9	–2.1
20R-2, 22–24	174.52	172.71	P10–11	NP15	MEOC	*A. bullbrooki*	>250	G	2.2	–2.0
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*M. velascoensis*	>250	E	4.8	–3.0
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*M. velascoensis*	>250	E	4.9	–2.9
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*C. pachyderma-like*	>250	E	1.8	–0.3
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*C. pachyderma*	>250	E	1.9	–0.3
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*N. truempyi*	>250	E	1.5	–0.4
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*N. truempyi*	>250	E	1.5	–0.3
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*S. triangularis*	>250	E	2.7	–2.8
34R-2, 20–22	309.10	308.02	P4	NP7	PAL	*S. triangularis*	>250	E	2.4	–1.3
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*M. velascoensis*	>250	E	5.1	–2.7
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*M. velascoensis*	>250	E	4.7	–2.5
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*C. pachyderma*	>250	E	2.3	–0.5
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*C. pachyderma*	>250	E	2.2	–0.4
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*N. truempyi*	>250	E	1.9	–0.5
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*N. truempyi*	>250	E	1.7	–0.4
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*A. soldadoensis*	>250	E	4.4	–2.9
35R-2, 20–22	318.80	317.72	P3	NP3	PAL	*A. soldadoensis*	>250	E	4.6	–2.9
37R-2, 20–22	338.10	338.35	KS31	CC25	MAAS	*Cibicidoides* sp.	>250	E	0.5	–1.7
37R-2, 20–22	338.10	338.35	KS31	CC25	MAAS	*Cibicidoides* sp.	>250	E	1.1	–0.5
37R-2, 20–22	338.10	338.35	KS31	CC25	MAAS	*N. truempyi*	>250	E	1.0	–0.6
37R-2, 20–22	338.10	338.35	KS31	CC25	MAAS	*N. truempyi*	>250	E	1.0	–0.5
207-1260B-										
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. delrioensis*	>250	E	1.4	–4.9
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. delrioensis*	>250	E	1.3	–5.0
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. delrioensis*	>250	E	1.3	–4.9
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. delrioensis*	>250	E	1.3	–4.8
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. delrioensis*	>250	E	1.3	–4.5
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. globulosa*	150–250	E	1.2	–4.7
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. globulosa*	150–250	E	1.5	–4.7
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. globulosa*	150–250	E	1.2	–4.8
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. globulosa*	150–250	E	1.4	–4.7
34R-2, 10–17	407.30	410.00	KS20/22	CC11	TUR	*H. globulosa*	150–250	E	1.3	–4.7
37R-1, 10–16	434.60	437.02		CC10	CEN	*H. delrioensis*	150–250	VG	1.0	–3.4
37R-1, 10–16	434.60	437.02		CC10	CEN	*H. delrioensis*	150–250	VG	1.1	–3.3
37R-1, 10–16	434.60	437.02		CC10	CEN	*Globigerinelloides*	150–250	VG	1.0	–3.5
38R-1, 84–86	444.94	449.01		CC10	CEN	*H. delrioensis*	150–250	E	2.0	–3.8

Table T1 (continued).

Core, section, interval (cm)	Depth (mbsf)	Depth (mcd)	Foraminifer zone	Nannofossil zone	Age	Species	Size fraction (μ)	Preservation	$\delta^{103}C$ (VPDB) (‰)	$\delta^{18}O$ (VPDB) (‰)
38R-1, 84–86	444.94	449.01		CC10	CEN	*H. delrioensis*	150–250	E	2.1	–3.7
39R-1, 115–122	454.85	457.63		CC10	CEN	*H. delrioensis*	150–250	VG	1.3	–4.2
39R-1, 115–122	454.85	457.63		CC10	CEN	*H. delrioensis*	150–250	VG	1.3	–4.2
39R-1, 115–122	454.85	457.63		CC10	CEN	*H. delrioensis*	150–250	VG	1.2	–4.4
39R-1, 115–122	454.85	457.63		CC10	CEN	*H. delrioensis*	150–250	VG	1.3	–4.4
40R-2, 97–103	465.57	470.15		CC10	CEN	*H. delrioensis*	150–250	M	0.8	–4.5
40R-2, 97–103	465.57	470.15		CC10	CEN	*H. delrioensis*	150–250	M	0.8	–4.4
41R-1, 114–120	473.84	478.00		CC10	CEN	*H. delrioensis*	150–250	E	1.1	–4.4
41R-1, 114–120	473.84	478.00		CC10	CEN	*H. delrioensis*	150–250	E	1.0	–4.3
41R-1, 114–120	473.84	478.00		CC10	CEN	*H. delrioensis*	150–250	E	1.0	–4.3
41R-1, 114–120	473.84	478.00		CC10	CEN	*H. delrioensis*	150–250	E	0.9	–4.3

Notes: Preservation key: E = Excellent; VG = Very good; G = Good; M = Moderate. Age key: MEOC = Middle Eocene; EEOC = Early Eocene; LPAL = Late Paleocene; MAAS = Maastichtian; SAN = Santonian; CON = Coniacian; TUR = Turonian; CEN = Cenomanian; LALB = Late Albian. VBPD = Vienna Peedee belemnite.

Figure AF1. Cross plot of the average oxygen and carbon isotope values for *Whiteinella* sp. from the four cleaning procedures given in Table AT1, p. 21. Error bars indicate the ±2σ range based on five analyses for each procedure.

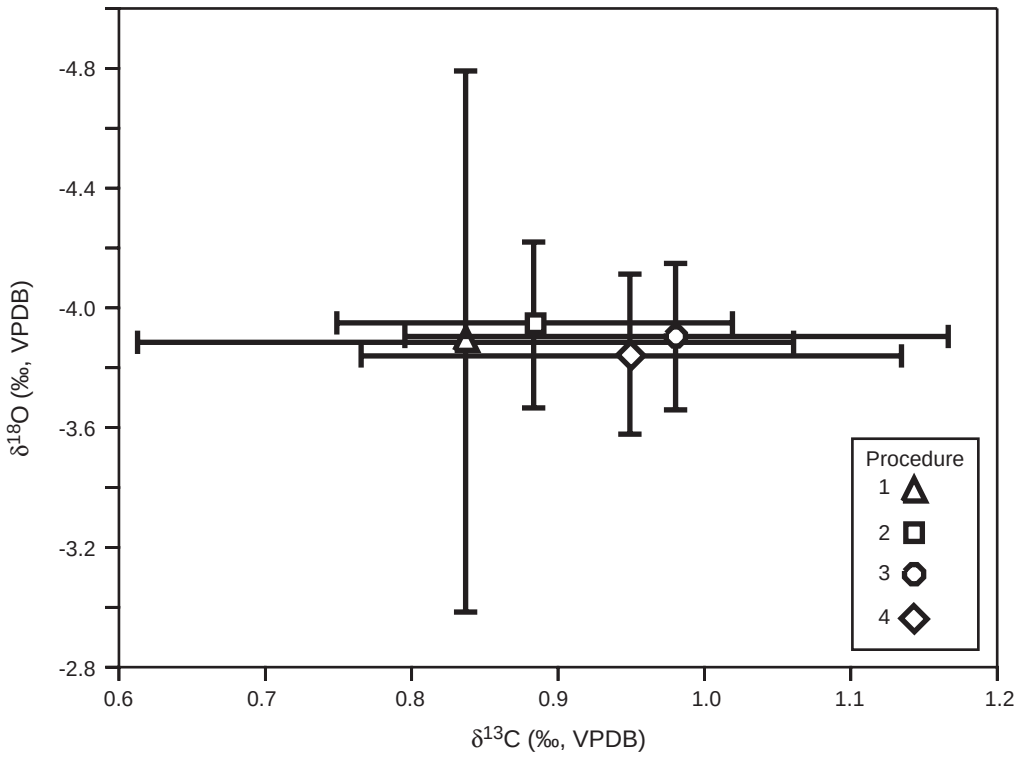


Figure AF2. Dried sediments from Sample 207-1259C-11R-4, 130–150 cm, remaining after soaking in Calgon-peroxide solution (left sieve, 2.3 g sediment) and 100% Clorox bleach (right sieve, 0.3 g sediment). The white grains in the bleach sample are predominantly loose foraminifers. Photograph was taken before the Calgon-peroxide sample was sonicated and then soaked overnight.

Table AT1. Black shale sample cleaning procedures for black shale sample splits from Sample 207-1258B-51R-2, 13–20 cm (Cenomanian).

Procedure	Treatment Steps						Coarse fraction (%)*
1	Soak 2 hr Calgon sol.†	Sonicate 40 s	Wash/dry	Soak 15 hr Calgon sol.	Sonicate 90 s	Wash/dry	5.0
2	Soak 2 hr 20% bleach‡	Sonicate 40 s	Wash/dry	Soak 1.5 hr 75% bleach		Wash/dry	13.1
3	Soak 2 hr 20% Cascade**	Sonicate 40 s	Wash/dry	Soak 15 hr 75% Cascade	Sonicate 90 s	Wash/dry	9.0
4	Soak 2 hr 100% bleach		Wash/dry	Soak 1 hr 100% bleach		Wash/dry	10.9

Notes: * = Coarse fraction is the % (by weight) of the initial ~5 g of material that was left at the end of the procedure. † = Calgon-peroxide solution (pH = 6) prepared by mixing 175 g sodium hexametaphosphate, 2 liters of ~35% hydrogen peroxide (pH < 4) and 18 liters deionized water (pH = 5). ‡ = Ultra Clorox regular bleach manufactured by The Clorox Company. This is 94% water, 6% sodium hypochlorite, and trace sodium hydroxide (pH = 11.4 for the undiluted product). ** = Cascade Pure Rinse Gel dishwasher detergent manufactured by Procter and Gamble. This is 90% water, 5%–10% sodium silicate, and 1%–5% of each of the following: sodium hypochlorite, potassium hydroxide, sodium hydroxide (pH = 11.7 for the undiluted product).

Table AT2. Stable isotope results for four cleaning procedures.

Procedure:	1(‰)		2 (‰)		3 (‰)		4 (‰)	
Stable isotope results:	$\delta^{13}C$	$\delta^{18}O$	$\delta^{13}C$	$\delta^{18}O$	$\delta^{13}C$	$\delta^{18}O$	$\delta^{13}C$	$\delta^{18}O$
207-1258B-51R-2, 12–20 cm								
W. baltica	0.9	–4.2	0.9	–3.9	0.9	–4.0	1.0	–3.6
	0.7	–4.0	0.9	–4.1	0.9	–4.0	0.8	–3.9
	0.8	–4.4	1.0	–4.1	1.1	–3.7	0.9	–3.7
	0.9	–3.2	0.8	–3.7	1.0	–3.9	0.9	–3.9
	1.0	–3.8	0.8	–3.9	1.0	–4.1	1.2	–4.0
Average:	0.9	–3.9	0.9	–3.9	1.0	–3.9	1.0	–3.8
Standard deviation:	0.11	0.42	0.07	0.13	0.09	0.12	0.1	0.15

Note: permil relative to Vienna Peedee Belemnite.

Table AT3. Results of a test of Calgon-hydrogen peroxide solution and 100% bleach on two different shale samples.

Solvent	Coarse fraction (weight %)		
	Before sonication	After sonication	After overnight soak
Sample 207-1259C-11R-4, 130–150 cm (Santonian)			
Calgon-peroxide	44.7	11.8	3.6
100% bleach	5.9	NA*	NA
Sample 207-1259C-17R-1, 116–136 cm (Turonian)			
Calgon-peroxide	66.9	45.5	32.4†
100% bleach	12.1	NA	NA

Notes: * = No further cleaning steps were needed after the 2-hr and 1-hr soaks.
† = Very few foraminifers remained at this point.