

## **2. DATA REPORT: TERRIGENOUS GRAIN-SIZE DISTRIBUTIONS AT SITES 1263 AND 1267: TESTING THE APPLICABILITY OF LEG 208 SEDIMENTS FOR EOLIAN ANALYSIS<sup>1</sup>**

Micah J. Nicolo<sup>2</sup> and Gerald R. Dickens<sup>2</sup>

### **ABSTRACT**

To date, the only Southern Hemisphere eolian grain-size record constructed for the early Paleogene comes from Deep Sea Drilling Project Site 215. Ten early Paleogene sediment samples from Site 215 were collected and processed to show that the existing eolian grain-size record at this site can be reproduced. Five samples each from Ocean Drilling Program Sites 1263 and 1267 were similarly examined to test the possibility of generating new Southern Hemisphere eolian grain-size records for the early Paleogene. Our results indicate that an eolian grain-size signal can be constructed at Walvis Ridge, although the record will be complicated by hemipelagic terrigenous inputs. Further, we assert that a record generated at a site located on the deep flanks of Walvis Ridge is particularly susceptible to hemipelagic influence.

### **INTRODUCTION**

The median grain size of eolian material extracted from deep-sea sediment can be used to constrain past zonal wind intensity (e.g., Janecek and Rea, 1983; Rea et al., 1985; Miller et al., 1987; Hovan and Rea, 1992; Rea, 1994). Sediment sequences from several locations in the Northern Hemisphere have been examined for changes in eolian grain size over the Cenozoic and specifically the early Paleogene. These

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<sup>2</sup>Department of Earth Science, Rice University, 6100 Main Street, Houston TX 77005, USA. Correspondence author: [micahn@rice.edu](mailto:micahn@rice.edu)

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records show a major fining in eolian material from the Paleocene to the Eocene, an observation interpreted as a relative decrease in the intensity of zonal winds and, more generally, atmospheric circulation (Rea and Janecek, 1983; Rea et al., 1985; Miller et al., 1987; Rea, 1994). To date, one eolian grain-size record has been generated using lower Paleogene sediment deposited in the Southern Hemisphere. This record from Deep Sea Drilling Project (DSDP) Site 215 adjacent to Ninetyeast Ridge in the central Indian Ocean also shows a significant drop in grain size across the Paleocene/Eocene (P/E) transition (Hovan and Rea, 1992). More records from the Southern Hemisphere, however, are clearly needed to confirm these results and further substantiate broad zonal interpretations.

One major hurdle facing studies of eolian material is site selection. Sediment sequences must contain biogenic carbonate, which can be dated, and cannot contain chert, which limits the chemical isolation of all siliciclastic material, including eolian grains. Appropriate sites also must be located far away from continents so that the terrigenous fraction of sediment is not completely dominated by noneolian (riverine/hemipelagic) material. Often, the principal concern is hemipelagic material (Rea, 1994). At locations beyond the continental shelf, reworked riverine material settles to water depths of supportable density (deeper than ~2000 meters below sea level [mbsl]) and travels as a hemipelagic cloud hundreds of kilometers from the margin (Honjo, 1982; Rea, 1994).

Walvis Ridge is located in the South Atlantic Ocean and extends west from Africa to the mid-ocean ridge. The sites of interest to this study were all drilled on Walvis Ridge >1200 km from Namibia. DSDP Site 527, cored in 1980 at ~4430 mbsl, recovered a thick Cenozoic sediment sequence on a deep flank of this ridge (Moore, Rabinowitz, et al., 1984). Preliminary work at this site indicated that hemipelagic material heavily dominates the terrigenous component of the lower Paleogene section (Rea and Hovan, 1995), making it problematic for studies of eolian material.

Ocean Drilling Program (ODP) Leg 208 returned to Walvis Ridge, recovering good lower Paleogene sediment sequences at five proximal sites that span a depth transect of ~2 km (Zachos, Kroon, Blum, et al., 2004). Site 1267 was drilled close to Site 527 and presumably holds a Paleogene terrigenous record dominated by hemipelagic material. Site 1263, however, was drilled near the crest of the ridge at ~2700 mbsl and could contain a terrigenous record with significant amounts of eolian material. The object of this work is to evaluate whether the newly acquired lower Paleogene sequence at Site 1263 is appropriate for eolian grain-size studies. Our results suggest that a high-age resolution eolian grain-size record could be generated at Site 1263, providing long-desired information regarding early Paleogene atmospheric circulation in the Southern Hemisphere.

## **SITES AND SAMPLES**

### **Site 215**

Site 215 is located at ~5300 mbsl on the western flank of Ninetyeast Ridge far from any continents in the central Indian Ocean (von der Borch, Sclater, et al., 1974). The existing early Paleogene eolian grain-size record for the Southern Hemisphere (Hovan and Rea, 1992) was

generated using sediment samples recovered between 83 and 130 meters below seafloor (mbsf). This record (Fig. F1) shows a significant  $\sim 6 \mu\text{m}$  decrease in the median grain size of eolian material near the P/E boundary ( $\sim 103.3$  mbsf), from  $\sim 9.6 \mu\text{m}$  in upper Paleocene sediment (109.37 mbsf) to  $\sim 3.8 \mu\text{m}$  in lower Eocene sediment (84.83 mbsf).

The isolation and analysis of eolian material from marine sediment is a labor-intensive process involving many steps (see the “Appendix,” p. 8). The subsequent analysis of the extracted component requires reliable measurements. Our first goal, therefore, was to test whether we could reproduce the published eolian grain-size record at Site 215. Ten samples were collected between 85 and 107 mbsf, with most samples taken within 5 cm of those examined by Hovan and Rea (1992).

### **Leg 208**

All Leg 208 sites currently lie well off the continental shelf more than 1200 km away from the African continent and likely were a greater distance in the early Paleogene as a result of a higher sea level. Drilling at Sites 1263 and 1267 recovered lower Paleogene sediment sequences on Walvis Ridge but at much different water depths, both now and in the past. Site 1263 is presently at  $\sim 2700$  mbsl, whereas Site 1267 is presently at  $\sim 4350$  mbsl. Near the P/E boundary at  $\sim 55$  Ma, however, water depths were probably  $\sim 1500$  m for Site 1263 and  $\sim 3200$  m for Site 1267 (Zachos et al., 2005).

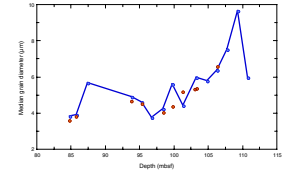
The P/E boundary has been identified at both Sites 1263 and 1267 (Zachos et al., 2005). Close to this time horizon, ages can be assigned to sediment depths fairly accurately using a model (Zachos et al., 2005) that ties changes in the carbon-isotope composition of carbonate to those observed at ODP Site 690, which has been calibrated using cyclostratigraphy (Rohl et al., 2000). Thus, “time-coincident” sediment samples can be taken from Sites 1263 and 1267 around the P/E boundary. Five such samples were collected from each site. Time-coincidence was emphasized so that potential differences in the delivery of eolian and hemipelagic components to the two sites at similar times could be explored.

## **METHODS**

### **Terrigenous Sediment Isolation**

Bulk marine sediment comprises multiple components; therefore, the terrigenous component (eolian and hemipelagic material) needs to be isolated for study. Previous work (e.g., Mehra and Jackson, 1960; Rea and Janecek, 1981; Hovan and Rea, 1992; Olivarez Lyle and Lyle, 2002) has developed a method to chemically extract the nonterrigenous components. The general procedure involves removing carbonate with weak acetic acid, removing metalliferous oxides and hydroxides with a buffered sodium citrate–sodium hydrosulfite mixture, and removing biogenic silica with one of several sediment-dependent steps. In this study, we adopted a specific procedure for extracting the terrigenous component, which is outlined in the “Appendix,” p. 8.

**F1.** Median grain size, p. 10.



## Grain-Size Distributions

Once extracted, the terrigenous component was homogenized and the grain-size distribution of all 20 samples was examined using a Coulter Multisizer II housed in the Civil and Environmental Engineering Department at Rice University (Houston, Texas). This device measures the electronic resistance of individual particles and divides results into 256 channels over a size range applicable to the material being analyzed, in this case 1–30  $\mu\text{m}$ . Previous work (Poppe et al., 2000) and repeat analyses conducted for this study indicate that the precision of this instrument is within  $\pm 10\%$ .

Nominally, 0.1 g of isolated terrigenous material and 15 mL of 0.008-M sodium hexametaphosphate were placed into a clean, labeled sample vial. The solution was sonicated for 5 min and mixed overnight in a hand-action shaker. After running clean electrolyte solution through the Coulter Multisizer II for 3 min to obtain a background count,  $\sim 2$  mL of stirred solution was slowly added to clean electrolyte solution until a desired sample density was met. Following subtraction of background counts, grain-size measurements were collected by averaging three 3-min sample runs on the Coulter Multisizer II. This method typically allows counting of  $\sim 100,000$ – $200,000$  individual particles.

## RESULTS

### Site 215

The median grain size of the extracted terrigenous component ranges from 6.6 to 3.6  $\mu\text{m}$  for the 10 samples collected from Site 215 (Table T1; Fig. F1). As highlighted by Hovan and Rea (1992), we find a general drop in median grain size from upper Paleocene sediment to lower Eocene sediment, although this change is not as great as previously recorded. We note, however, that our samples are not true replicates and do not span the entire depth interval examined by Hovan and Rea (1992). For the nine samples collected within 5 cm (and on average 3 cm) of those analyzed by Hovan and Rea (1992), we measured an average difference in grain size of  $\sim 0.4$   $\mu\text{m}$  (Fig. F1), which is approximately equal to the precision of the instrument for particles of this size.

### Site 1267

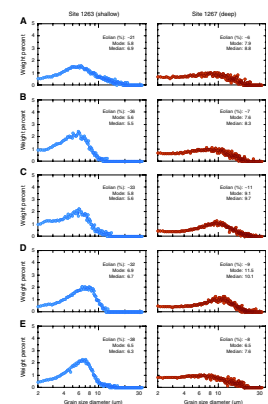
All five samples analyzed at Site 1267 show a relatively flat grain-size distribution over a broad size range (Fig. F2). The median grain sizes range from 7.6 to 10.1  $\mu\text{m}$ , whereas the modal grain size varies from 6.5 to 11.5  $\mu\text{m}$  (Table T2). The overall shapes of the grain-size patterns are similar to those observed at Site 527 by Rea and Hovan (1995).

### Site 1263

In contrast to samples from Site 1267, grain-size distributions for the five samples from Site 1263 show a distinct mode (Fig. F2). Similar to the samples measured at Site 1267, however, Site 1263 samples record a broad grain-size distribution indicative of hemipelagic terrigenous material influence. The median grain sizes range from 5.5 to 6.9  $\mu\text{m}$ , whereas the modal grain size varies from 5.6 to 6.9  $\mu\text{m}$  (Table T2).

T1. Median grain size, p. 12.

F2. Grain-size distributions, p. 11.



T2. Grain-size median and mode, p. 13.

## DISCUSSION

### Robust Grain-Size Records

Previous work on terrigenous material in deep-sea sediment indicates that given similar extraction procedures and analytical methods, measurements of grain size are reproducible (e.g., Rea, 1994). For closely spaced lower Paleogene sediment samples at Site 215, the median grain size determined for the extracted terrigenous component is similar to that reported by Hovan and Rea (1992). We therefore conclude that our approach for isolating and examining the terrigenous component will render robust grain-size information.

### Terrigenous Component on Walvis Ridge: a Mixture of Hemipelagic and Eolian Material

The grain-size distributions of the terrigenous component extracted from upper Paleocene–lower Eocene sediment at Site 1267 are similar to those observed for the terrigenous component extracted from contemporaneous sediment at nearby Site 527 (Rea and Hovan, 1995). In both cases, the isolated component has a flat pattern characterized by low weight percents of material spread across a wide grain-size range (Fig. F2). This pattern suggests that hemipelagic material dominates the terrigenous component (Rea and Hovan, 1995; Boven and Rea, 1998).

Grain-size analyses of the terrigenous component in time-coincident samples at Site 1263 clearly vary from those at Site 1267 (Fig. F2). Isolated samples from Site 1263, the shallower location, contain a pronounced grain-size mode centered between 5 and 7  $\mu\text{m}$ . Such a grain-size distribution is consistent with a large fraction of eolian material in the terrigenous component (Boven and Rea, 1998).

Even in open-ocean settings, the terrigenous component of deep-sea sediment may comprise a mixture of eolian and hemipelagic material (Rea, 1994; Rea and Hovan, 1995). Using extensive data sets of terrigenous grain-size distributions from Pacific Ocean sediments, Boven and Rea (1998) developed a two-component mixing model to determine the relative proportion of each component. This approach is based on the observation that hemipelagic grain-size distributions are broad with peak weight percents typically low near 1%, whereas eolian grain-size distributions depict a dominant mode with greater peak weight percents near 5% (Boven and Rea, 1998). The model compares the peak weight percent of a sample's grain-size distribution to end-member eolian and hemipelagic distributions. According to this model, our results suggest that the terrigenous component of sediment deposited near the P/E boundary contains ~6%–11% eolian material at the relatively deep Site 1267 but as much as 20%–40% eolian material at the relatively shallow Site 1263. This indicates that the shallow site received a higher concentration of eolian material than did the deep sites. However, sites 1263 and 1267 have likely received a similar flux of eolian material through time because they are proximal. This indicates that the deep flank of Walvis Ridge received a higher flux of hemipelagic sediment than the shallow crest. This finding conforms to general views of hemipelagic deposition, considering the paleodepths of Sites 1267 (~3200 mbsl at ~55 Ma) and 1263 (~1500 mbsl at ~55 Ma). Hemipelagic material should have settled and moved at >2000 m water depth, such that

greater amounts of hemipelagic material would have accumulated at Site 1267 (and Site 527).

### **Site 1263 Eolian Grain-Size Record: Mode vs. Median**

An eolian grain-size record can probably be gleaned from the lower Paleogene sequence recovered at Site 1263 on Walvis Ridge. However, there is an issue regarding measurement because, even at this shallow location, the terrigenous component contains a mixture of hemipelagic and eolian material. Large amounts of hemipelagic material broaden the grain-size distribution so that the median and modal grain size of the terrigenous component differs significantly (Fig. F2). Whereas previous studies of eolian grain size have generally used the median for paleoenvironmental reconstructions, we suggest that the mode more accurately measures the grain size of the eolian fraction in mixed hemipelagic-eolian systems (where an eolian signal is detectable). This is particularly evident in samples with lower eolian percents, such as the Site 1263 sample illustrated in Figure F2A. Figure F2A shows the lowest Site 1263 eolian percent and the highest mode-median offset of 1.1  $\mu\text{m}$ , whereas samples shown in Figure F2B–F2E have eolian percents ranging from 32% to 38% and a mode-median difference of only 0.1–0.2  $\mu\text{m}$ . Further, samples collected from Site 215 that were shown to be of eolian origin (Hovan and Rea, 1992) record a mode-median average difference of 0.1  $\mu\text{m}$ .

## **CONCLUSIONS**

The terrigenous component of lower Paleogene marine sediment can be isolated and subsequently analyzed for precise grain-size measurements. It is probable that an early Paleogene Southern Hemisphere eolian grain-size record can be constructed at Site 1263 on the crest of Walvis Ridge. Interpretation of such a record must consider the mixed eolian-hemipelagic nature of the terrigenous component. Modal grain size may be a more important measurement than median grain size for eolian proxies at this site, as it appears to retain a grain-size distribution signal characteristic of eolian sediments. Sites at greater water depth and on the flank of Walvis Ridge contain a higher abundance of hemipelagic material, which limits their utility for analyses of the eolian component. This probably reflects the general process of hemipelagic deposition, where material settles in waters of sufficiently high density (often >2000 mbsl) to support and transport mineral grains.

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## APPENDIX: EXTRACTION FOR ISOLATING TERRIGENOUS MATERIAL

A series of papers have discussed the isolation of terrigenous material from deep-sea sediment (e.g., Rea and Janecek, 1981; Hovan and Rea, 1992; Olivarez Lyle and Lyle, 2002). In general, these works have used a similar overall procedure that involves sequential extraction to remove carbonate, metalliferous oxides and hydroxides, and biogenic silica. Rarely, however, are the actual steps presented. The detailed steps used in this study to extract terrigenous material are presented here, modified somewhat from notes kindly provided to us by Dr. Leah Joseph.

### Carbonate Removal

1. Place weighed masses of sample (~10 bulk g) into clean, labeled 500 mL Erlenmeyer flasks.
2. Slowly add ~125 mL of 4.5-N (25% by volume) acetic acid to each flask in 10-mL increments to avoid loss by bubbling.
3. Shake flasks with mechanical wrist-action shaker for 2 hr.
4. Pour solutions from flasks into labeled 250-mL centrifuge bottles.
5. Centrifuge for 13 min at ~4500 rpm and decant supernatant.
6. Add ~125 mL of 4.5-N (25% by volume) acetic acid to each 250-mL centrifuge bottle.
7. Sonicate for 5 min.
8. Pour sample solutions back into corresponding Erlenmeyer flasks and shake again with mechanical wrist-action shaker for 2 hr.
9. Pour solutions from flasks into corresponding 250-mL centrifuge bottles.
10. Centrifuge for 13 min at ~4500 rpm and decant supernatant.

### Metalliferous Oxyhydroxide Removal

1. Fill a water bath with tap water to about three-quarters full and heat on hot plate to ~85°C.
2. Fill one 1000-mL beaker full and one 600-mL beaker with mixture of half ultrapure deionized (DI) water and half 0.3-M sodium citrate (50/50) and place on separate hot plate (solution hot plate) at a slightly lower setting.
3. Add 40 mL of 0.3-M sodium citrate into each 250-mL centrifuge bottle.
4. Slowly add 5 mL of 1.0-M sodium bicarbonate to each 250-mL centrifuge bottle.
5. Place bottles in hot water bath and leave for 10–15 min. While waiting, fill one 1000-mL beaker full and one 600 mL beaker with 0.3-M sodium citrate and place on solution hot plate. Only fill the sodium citrate beakers during the first of two repeats (i.e., Step 9).
6. Add ~1 g sodium dithionate (sodium hydrosulfite) to each 250-mL centrifuge bottle, stir continuously for 30 s, and return to hot water bath.
7. After 5–10 min, add 100 mL of 50/50 solution to each centrifuge bottle and stir thoroughly.



8. Centrifuge for 13 min at ~4500 rpm and decant supernatant.
9. Repeat oxide/hydroxide extraction Steps 2–8.
10. Add 100 mL of hot 0.3-M sodium citrate and stir thoroughly.
11. Centrifuge for 13 min at ~4500 rpm and decant supernatant.
12. Repeat Steps 3–11 so that each sample has received two extractions and hot rinses. Samples should now be a grayish green. If they are still reddish orange, another oxyhydroxide removal sequence may be necessary.
13. Centrifuge for 13 min at ~4500 rpm and decant supernatant.

### **Opal Removal**

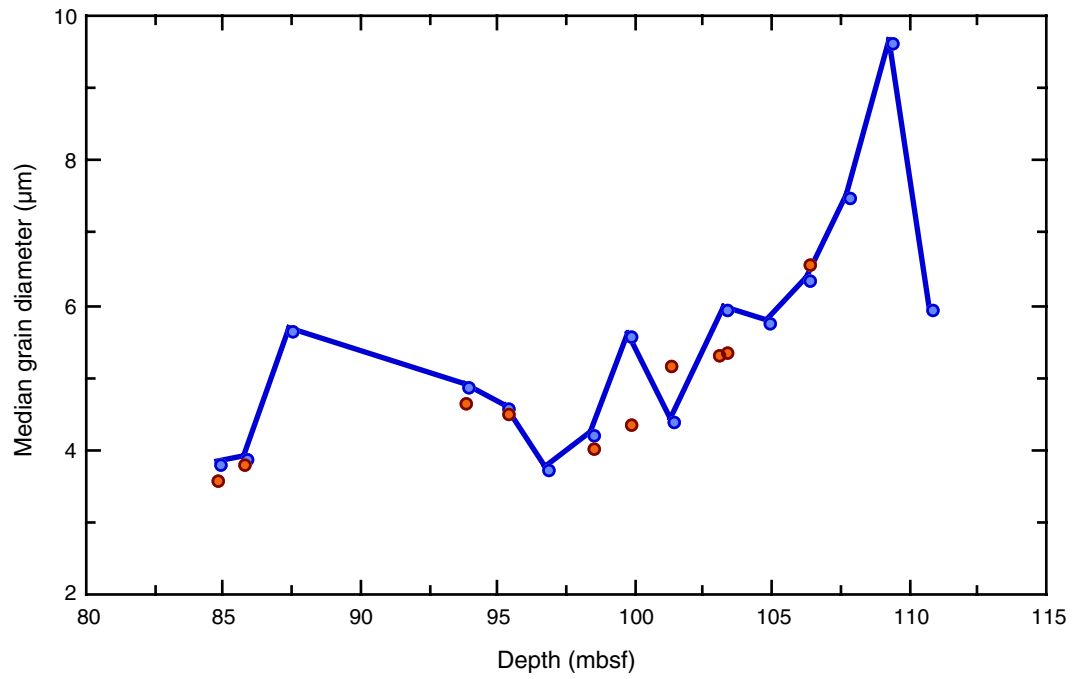
1. Wash each sample into a clean, labeled 600-mL beaker with ultrapure DI water.
2. Fill beakers with ultrapure DI water to 400 mL and cover with watchglass.
3. Place beakers directly on hot plate and bring to ~85°C.
4. Add 24 g sodium hydroxide (NaOH) to each beaker (1.5-M NaOH), stir thoroughly, and replace watchglass.
5. Leave beakers on hot plate for 2 hr at ~85°C. Stir solution after 1 hr.
6. Remove beakers from hot plate and let cool for several minutes.
7. Pour sample/solution into corresponding 250-mL centrifuge bottle, centrifuge for 13 min at ~4500 rpm, and decant supernatant.

Note: The opal removal step was not conducted on sediment samples from Leg 208 sites because they lack radiolarians and diatoms (Zachos, Kroon, Blum, et al., 2004).

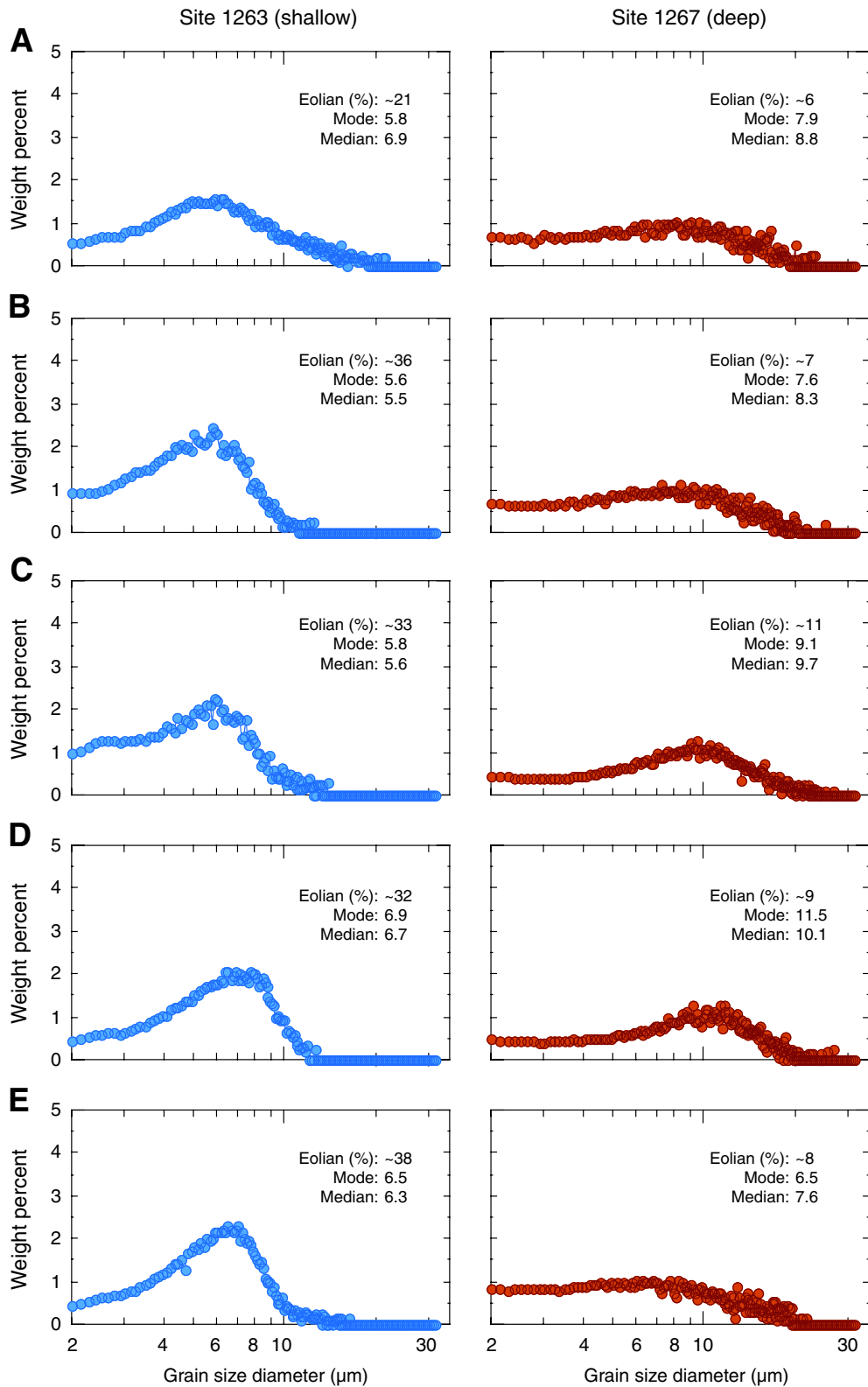
### **Final Rinse**

1. Fill each 250-mL centrifuge bottle with ~40 mL of hot, ultrapure DI water and stir thoroughly.
2. Wash solution and remnant sample into corresponding clean, dry, labeled, and preweighed 50-mL centrifuge tube.
3. Centrifuge for 13 min at ~4500 rpm and decant supernatant.
4. Repeat Steps 1–3 four times.
5. Freeze the residue in the centrifuge tube for 12 hr.
6. Freeze-dry the residue in the centrifuge tube for 48 hr.
7. Tare the dried residue.

**Figure F1.** Median grain size of lower Paleogene terrigenous sediment component at Site 215. Blue circles = data published by Hovan and Rea (1992), red circles = results generated for this study.



**Figure F2.** A–E. Site 1263 and Site 1267 grain-size distributions. Five sets of time coincident (less than ~16 k.y. difference) grain-size distributions are illustrated, such that each part contains one distribution from the shallower Site 1263 and one distribution from the deeper Site 1267. Eolian percent has been calculated after the two-component mixing model of Boven and Rea (1998).



**Table T1.** Median grain size of eolian material, Site 215.

Core, section, interval (cm)	Mean depth (mbsf)	Median grain size ( $\mu\text{m}$ )
22-215-		
10R-1, 128-130	84.79	3.6
10R-1, 131-134	84.83	3.8
10R-2, 75-78	85.77	3.8
10R-2, 80-83	85.82	3.9
10R-3, 94-97	87.46	5.6
11R-1, 83-85	93.84	4.7
11R-1, 85-88	93.87	4.9
11R-2, 81-84	95.33	4.5
11R-2, 85-88	95.37	4.6
11R-3, 85-88	96.87	3.7
11R-4, 92-95	98.44	4.0
11R-4, 95-98	98.47	4.2
11R-5, 80-82	99.81	4.4
11R-5, 82-85	99.84	5.6
11R-6, 82-85	101.34	5.2
11R-6, 85-88	101.37	4.4
12R-1, 50-57	103.04	5.3
12R-1, 83-85	103.34	5.4
12R-1, 85-88	103.37	6.0
12R-2, 85-88	104.87	5.8
12R-3, 83-86	106.35	6.6
12R-3, 85-88	106.37	6.3
12R-4, 83-86	107.85	7.5
12R-5, 85-88	109.37	9.6
12R-6, 85-88	110.87	6.0

Note: Shaded rows represent Hovan and Rea (1992) results.

**Table T2.** Terrigenous grain-size median and mode and percent eolian, Leg 208.

Grouping	Core, section, interval (cm)	Mean depth (mbsf)	Grain size ( $\mu\text{m}$ )		Eolian (%)
			Median	Mode	
A	208-1263C-14H-2, 21–23	284.12	6.9	5.8	21
	208-1267B-23H-3, 57–58	205.28	8.8	7.9	6
B	208-1263C-14H-2, 50–52	284.41	5.5	5.6	36
	208-1267B-23H-3, 82–83	205.53	8.3	7.6	7
C	208-1263C-14H-2, 60–62	284.51	5.6	5.8	33
	208-1267B-23H-3, 87–88	205.58	9.7	9.1	11
D	208-1263C-14H-2, 110–112	285.01	6.7	6.9	32
	208-1267B-23H-3, 102–103	205.73	10.1	11.5	9
E	208-1263A-34X-1-36–40	284.48	6.3	6.5	38
	208-1267B-23H-3, 107–109	205.78	7.6	6.5	8

Notes: Grouping corresponds to Parts A–E of Figure F2, p. 11. Percent eolian calculated using the model of Boven and Rea (1998).