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A. Trentesaux, Philippe Recourt, Viviane Bout-roumazeilles, Nicolas Tribovillard

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# 1 **Carbonate grain-size distribution in hemipelagic sediments from a laser** 2 **particle sizer**

3

4 Trentesaux, A., Recourt, P., Bout-Roumazelles, V., Tribovillard, N.

5

6 Laboratoire de Sédimentologie et de Géodynamique, UMR 8577, Université des Sciences et  
7 Technologies de Lille, SN5, F-59655 Villeneuve d'Ascq Cedex, France

8 e-mail : alain.trentesaux@univ-lille1.fr

9

## 10 **Abstract**

11 Laser grain-sizer instruments provide the opportunity to study the grain size  
12 distribution of sediments across a wide size range in a short time. Automatic  
13 Tapez une équation ici.measurements can therefore be made, on a routine basis, for a great  
14 number of samples. Oceanic studies have proved the utility of these methods in characterizing  
15 both climatic changes and changes in sediment provenances. In addition, carbonate content is  
16 estimated either directly by CaCO<sub>3</sub> measurement, by visual observations, or by proxies such  
17 as sediment color reflectance. Nevertheless, the grain size distribution of the carbonate  
18 fraction is still a matter of speculation, and only optical observations can distinguish the  
19 nature of each carbonate fraction. Here we present the improvements on a method to study  
20 rapidly, with a high resolution, the grain size distribution of the carbonate fraction by use of a  
21 laser grain-sizer. We describe the basic methodology and apply it to an example from the  
22 Pleistocene of the Northern Atlantic Ocean.

23

## 24 **Introduction**

25 In paleoceanographic studies, grain size distribution and carbonate content are often  
26 examined. The first parameter is derived by a number of methods, each having some  
27 advantages and drawbacks. It provides valuable information on the depositional mechanisms  
28 (Clemens and Prell 1990; Rea and Hovan 1995; Prins 1997; Kaiho 1999; Wang et al. 1999)  
29 and sea-floor currents (Pudsey 1992; Faugères and Stow 1993; McCave et al. 1995a; McCave  
30 et al. 1995b; Diekmann and Kuhn 1997; Michels 2000). Carbonate content is usually obtained  
31 by chemical digestion, X-ray diffraction, and, more recently, the use of sediment color  
32 reflectance as a proxy for carbonate content (Blum 1997; Balsam et al. 1999).

33 In this paper, we discuss a method that facilitates rapid measurement of the two  
34 parameters with a good degree of accuracy, including the grain size distribution of the  
35 carbonate fraction. The method uses a wide-range laser diffraction size analyzer. A brief  
36 application is presented on samples from a Pleistocene climatic transition in Northern Atlantic  
37 sediments from an Ocean Drilling Program core. This method offers a good alternative to  
38 some classical measurements performed through smear-slide observations or differential  
39 chemical digestions and microscopic observations (Diekmann and Kuhn 1997).

40

## 41 **Method**

### 42 *Measurement Principle*

43 For this study, grain size parameters were determined using a Malvern Mastersizer X  
44 apparatus. Other manufacturers exist, and in all cases the instruments use the diffraction of a  
45 laser light in a cell filled with the sample in suspension in a dispersant (usually deionized  
46 water). The diffraction angle is inversely proportional to the grain size and is measured with a  
47 photoelectric cell. A series of algorithms are then used to calculate a grain size distribution  
48 curve assuming a spherical shape of the particles. For these instruments, manufacturers  
49 indicate that typical accuracy is less than 2% for grain sizes between 900 and 5  $\mu\text{m}$  and  
50 decreases to about 6% for finer grain sizes. Manufacturers' reported precision is 2%. The  
51 measurement principle is well explained in McCave et al. (1986) and Agrawal et al. (1991).

52 The relative advantages or drawbacks of this technique and other grain-size analyzers  
53 have been well documented (McCave and Syvitski 1991; Loizeau et al. 1994; Kench and  
54 McLean 1997; Konert and Vandenberghe 1997; Beuselinck et al. 1998). Among the  
55 advantages that led us to choose this technique instead of others is the good precision and the  
56 rapidity of the measurement, allowing the study of large sample sets. Four lenses are  
57 available, depending on the estimated grain size distribution: 45, 100, 300, and 1000 mm,  
58 respectively for 0.1 80, 0.5 180, 1.2 600, and 4.0 2000  $\mu\text{m}$ , respectively. The 100 mm lens  
59 was selected because it was the most suitable for our tests.

60

### 61 *Sample Preparation*

62 In the case of a coarse fraction, the sediment can be gently sieved under deionized  
63 water. Samples were first put in suspension in deionized water and then gently shaken for two  
64 hours to achieve disaggregation. For organic-rich sediments, it might be interesting to use  
65 H<sub>2</sub>O<sub>2</sub> to oxidize organic matter and to break up the floccules; this has not been tested in our  
66 studies. In contrast to the treatment proposed by Loizeau et al. (1994), sonication was not

67 used to complete the sediment dispersion. Measurements showed that after more than two  
68 hours of gentle shaking, the suspended sediment is well disaggregated. Moreover, the use of  
69 ultrasonic dispersion has a dramatic effect on some particles containing trapped water  
70 inclusions, such as foraminifers or vesicular volcanic glass. Repeated measurements after  
71 increasing periods of ultrasonic dispersion clearly demonstrate the disintegration of the  
72 foraminifer tests and vesicular glass particles, leading to a decrease in the mean sediment  
73 grain size. Broken foraminifer tests can subsequently be observed in residues on smear slides.

74 The suspension is then gently poured into the fluid module of the granulometer, filled  
75 with tap water. Tap water was chosen rather than degassed water for reasons of simplicity.  
76 The background noise introduced by the degassing water (Loizeau et al. 1994) is nevertheless  
77 small and is included in the measured background that is later subtracted from the total  
78 measurement. Ultrasonics is used before pouring the sediment in order to decrease the  
79 degassing time. The final suspension has a concentration varying from  $10^{-4}$  to 0.1%  
80 (expressed as a relative volume concentration). Measurement begins after two minutes of  
81 continuous stirring to allow most of the air bubbles created by the sediment introduction to  
82 escape.

83

#### 84 *Measurement and Calculations*

85 Grain-size distribution is calculated using the refractive index of quartz (1.56) for the  
86 sediment and 1.0 for the water. After measurement, the data are saved in a file containing all  
87 percentiles and a variety of calculated parameters. Each size class  $I$  is characterized by an  
88 amount noted  $T(i)$ . The computer software also computes a cumulative and frequency  
89 histogram for each sample. The data are transferred to a spreadsheet using a macro that  
90 samples the data at increments chosen by the user (in our case,  $\#/6$ ).  $\#$  ( $\phi$ ) is the negative  
91 logarithm to the base 2 of the particle diameter in millimetres.

92

#### 93 *Distribution of the Carbonate Fraction.*

94 The major advantage of a laser diffraction grain-size analyzer is the ability to measure  
95 a sediment suspended in water. This allows the addition of the necessary amount of  
96 hydrochloric acid (HCl) to dissolve the calcium carbonate contained in the sediment, an  
97 approach already used by McCave et al. (1986) to characterize the non carbonate fraction.  
98 HCl is added in excess to the suspension, verified by stabilization of the measured pH at a  
99 value of 1 for more than one minute. The instrument does not suffer from the acid conditions,  
100 because the main parts are made of Teflon, glass, or stainless steel. A repeat measurement is

101 performed after acid digestion to obtain a “non carbonate fraction” grain-size distribution.  
102 For each size class  $i$ , the decarbonated fraction is expressed as  $D(i)$ .

103 After transferring data to the spreadsheet, the carbonate fraction  $C(i)$  is calculated by  
104 the formula

$$C(i) = T(i) - D(i) \times (1 - CaCO_3) \quad (4)$$

106 derived from McCave et al. (1995b), where  $C(i)$  is the calcium carbonate fraction of the size  
107 class  $i$ ,  $T(i)$  is the total sediment fraction of size class  $i$ ,  $D(i)$  is the decarbonated sediment  
108 fraction of size class  $i$ , and  $CaCO_3$  is the calcium carbonate content in the sediment, as  
109 described next.

110

### 111 Calculation of the $CaCO_3$ Percentage.

112 Although it is possible to measure the  $CaCO_3$  content by classical chemical  
113 techniques, we found that the Malvern Mastersizer was able to calculate automatically the  
114 carbonate content of a sediment measured first as a total sediment and then as a decarbonated  
115 sediment. Among the different parameters measured by the instrument, “obscuration”  
116 corresponds to the loss of transmitted light intensity due to the presence of particles in  
117 suspension. For a monodispersed sample, obscuration is a direct function of grain size,  
118 concentration, and path length (width of the measurement cell) according to the following  
119 formula

$$V = \frac{-d \times \ln(L-O)}{3.1} \quad (2)$$

121 (Malvern Instruments Ltd. Reference manual), where  $V$  is the volume concentration as  
122 relative percentage,  $d$  is the particle diameter in microns,  $L$  is the path length illuminated  
123 expressed in millimeters, and  $O$  is the obscuration.

124 It follows from this relationship that the decrease in concentration between the two  
125 grain size measurements (before and after acid digestion) will cause a decrease in the  
126 obscuration directly related to the loss of particles dissolved by hydrochloric acid.

127 The  $CaCO_3$  content can therefore be directly expressed by the formula

$$CaCO_3 = \frac{ObsT - ObsD}{ObsT} \quad (3)$$

129 where  $CaCO_3$  is  $CaCO_3$  the calcite content, ObsT is the obscuration measured when the total  
130 sample is in suspension, and ObsD is the obscuration measured after acid digestion  
131 (decarbonated sample).

132 This was tested by measuring the carbonate content both by chemical digestion and by  
133 obscuration difference using Equation 3 (Fig. 1). Although the correlation is very good  
134 (correlation coefficient  $r = 0.9877$ ), it is notable that the correlation line is not 1:1, indicating  
135 that the carbonate content measured with the obscuration difference has to be calibrated to  
136 better represent the carbonate content. This is most likely because obscuration also depends  
137 on the grain size (Malvern Instruments Ltd., Reference Manual). We therefore suggest that a  
138 calibration should be made with samples from each new location.

139 Substitution of Equation 3 into Equation 1 gives

$$140 \quad C(i) = T(i) - D(i) \times \frac{ObsD}{ObsT} \quad (4)$$

141 Equation 4 facilitates drawing of the cumulative or frequency distribution curve of the  
142 carbonate fraction from the sediment, permitting identification of the mode, mean, sorting  
143 index, or skewness. Figure 2 illustrates the three curves for a sample from the Northern  
144 Atlantic Ocean.

145

#### 146 **Application to pelagic sediments**

147 To test the method on natural samples, we selected 16 samples from the ODP leg 162,  
148 site 980, in the Northern Atlantic Ocean, on the western slope of the Rockall Trough at a  
149 depth of 2180 m (Jansen et al. 1996). To both emphasize the possible changes in the  
150 carbonate fraction of the sediment and to scan a wide variety of  $\text{CaCO}_3$  content, we used  
151 samples dated at the transition between the Pleistocene isotope stages 6 and 5e, between  
152 25.26 and 20.56 m below sea floor (146.5 to 119.0 ka, respectively). The sediment consists of  
153 calcareous oozes and muds. It is rich in foraminifers of diameter greater than the  
154 measurement range. For that reason, it was decided to sieve the sediment at 80  $\mu\text{m}$  very  
155 gently under deionized water to remove the large foraminifer fraction. The 16 samples were  
156 then prepared as described, and their grain-size distribution was measured on both total and  
157 decarbonated fraction.

158 A number of analyses have been performed onboard and in the shore-based  
159 laboratory: grain-size distribution,  $\delta^{18}\text{O}$  composition, and reflectance. Values of mean grain  
160 size measured on the bulk sediment range between 6.5 and 20  $\mu\text{m}$  with no trend in the depth  
161 distribution (Fig. 3A). After acid digestion (decarbonated sample in Fig. 3A), the usually  
162 higher values of the mean grain size indicate that the terrigenous fraction is slightly coarser  
163 than the carbonate fraction that is probably rich in nannofossils. The carbonate-fraction mean

164 grain size varies between 4.5 and 16.5 $\mu\text{m}$ , with variations similar to those observed in the  
165 other fractions. It is clear from Fig. 3A that the grain-size distribution of the bulk sediment in  
166 this example is not an informative parameter, even if it is sometimes interesting to illustrate  
167 variations in the sediment regime related to climatic change (Wang et al. 1999). The age  
168 model has been established from the  $\delta^{18}\text{O}$  stratigraphy measured on *Cibicides* sp. (Fig. 3B,  
169 Jerry McManus, unpublished data). The isotope stage 6–5e transition is especially well  
170 pronounced at this location because of its proximity to the polar front in glacial stages but  
171 distance from it during interglacial stages. Using the technique described above,  $\text{CaCO}_3$   
172 content ranges from 11.5 to 84% (Fig. 3C). There is a clear increase in carbonate at the  
173 transition, interpreted to be related to an increase in carbonate productivity during isotope  
174 stage 5e (considering no change in the dissolution conditions (Milliman and Droxler 1996) or  
175 winnowing removal of the carbonate (Michels 2000)) with a time lag that can be evaluated as  
176 0.3 to 0.4 ka between the change in  $\delta^{18}\text{O}$  composition and the change in  $\text{CaCO}_3$ . The duration  
177 of this interval might be explained, in part, by the sampling interval. Reflectance measured  
178 onboard (Fig. 3D, Ortiz et al. 1999), on the contrary, is directly related to C content as  
179 observed in numerous oceanic locations (e.g., Blum 1997).

180 The  $\text{CaCO}_3$  content estimated from the obscuration difference (Equation 3) is used to  
181 produce the 16 carbonate grain-size distributions (Fig. 4). The surface of the histograms is  
182 directly related to the general carbonate content and emphasizes the climatic transition around  
183 130 ka. The carbonate-fraction distribution indicates two modal sizes centred at 2 and 32  $\mu\text{m}$ .  
184 Smear-slide observations indicate that these modes correspond to nannofossils and planktonic  
185 foraminifers, respectively. During the end of glacial stage 6, there is a clear dominance of  
186 nannofossils over foraminifers in most samples. This is not the case during the early part of  
187 stage 5e (samples at 130.5 and 126.5 ka) when the foraminifer modal class is of the same  
188 order of magnitude as that of the nannofossil, indicating that foraminifers are a major  
189 contributor (in volume or in weight) to the  $\text{CaCO}_3$  sedimentation. After this transition,  
190 nannofossil abundance increases rapidly to assume the main mode while the foraminifer  
191 mode increases slowly. The reasons for this two-phase transition between the glacial and an  
192 interglacial stage are not discussed here but indicate a phase lag that has to be confirmed by  
193 studies on other transitions or over a longer time period.

194

## 195 **Discussion**

196 The technique described has great potential utility, making possible a fast  
197 determination of the carbonate content with a good accuracy and yielding the carbonate

198 distribution over the studied grain size range. Nevertheless, there is always a vital need to  
199 verify the interpretations by visual observations, especially due to some uncertainties, such as  
200 sample quality and instrumental limitations.

201

### 202 *Sample Quality*

203 Detrital Calcite.—Calcite can be present in oceanic sediments as a contributor to the detrital  
204 fraction. Although calcite is easily distinguished on smear slides, the method we describe  
205 cannot differentiate between biogenic and detrital calcite. For that reason, we recommend  
206 closer examination of some samples to avoid misinterpretations. At site 980 in the Northern  
207 Atlantic Ocean, smear-slide observations indicate the absence of such a contribution along the  
208 study interval.

209 Diagenetic Calcite.—Grain size analyses can easily be performed on non cemented samples.  
210 The technique described here must be applied to sediments that have not suffered from any  
211 carbonate diagenesis. None of the present-day existing methods for measuring grain size can  
212 make the distinction between diagenetic calcite and biogenic calcite.

213

### 214 *Instrument Limitations*

215 High Carbonate Content.—The accuracy of the CaCO<sub>3</sub> content measurement decreases with  
216 increasing carbonate concentration, as indicated by the increased scatter in Figure 1 for values  
217 greater than 60%. Equation 3 indicates that carbonate content is directly related to the  
218 obscuration parameter. The obscuration values have to fall within the grain-sizer range. The  
219 instrument can measure with obscurations that are higher or lower (>30% or <10%,  
220 respectively; Malvern instrument Ltd. reference manual) than the optimal case, but there is an  
221 associated loss of accuracy.

222 Using these values of obscuration, the maximum carbonate content can be calculated  
223 by substituting obscuration values in Equation 3

224

$$225 \quad CaCO_{3Max} = \frac{ObsT_{Max} - PbsD_{Min}}{ObsT_{Max}} \times 100 = \frac{30-10}{30} \times 100 = 66.6\% \quad (5)$$

226

227 If a deterioration of the measurement is acceptable (replacing Obs<sub>TMax</sub> and Obs<sub>DMin</sub> by  
228 50 and 5, respectively; Malvern instrument Ltd. reference manual), then a maximum value of  
229 90% of carbonate content is obtained using Equation 3. This means that above 66.6% of  
230 carbonate, the carbonate content accuracy decreases rapidly to be non-measurable above 90



231 % ObsT is fixed by the instrument user. It is therefore recommended to introduce a large  
232 quantity of bulk sediment when a high carbonate content is expected, to maintain enough  
233 material after acid digestion.

234 Low Carbonate Content.—When the carbonate content is too low (approaching 15%), the  
235 measurements approach the accuracy limit of the instrument. Note in Figure 1 that the  
236 intercept at the origin is not zero, indicating that in the case of very low carbonate  
237 concentration a higher volume is calculated. It also indicates that incorporating acid in the  
238 sampling cell could affect the grain-size distribution even if no carbonate is present in the  
239 sediment.

240 Measurement Accuracy of Carbonate Content.—The two previous sections indicate that in  
241 some cases, measurement of carbonate content can suffer from a decrease in analysis  
242 accuracy if carbonate content is calculated using obscuration parameters. To avoid this  
243 problem, it is more suitable to use data on carbonate content from other methods as acid  
244 digestion and use Equation 1. However, this causes extra laboratory work and is slower.  
245 Furthermore, some problems can occur because the CaCO<sub>3</sub> analysis is not performed on  
246 strictly the same sample, leading to a loss of precision in calculating the size distribution of  
247 the carbonate fraction.

248

## 249 **Conclusions**

250 With the ability to measure the concentration and grain size distribution first on total  
251 sediment and then on the same, but decarbonated, sample by the laser diffraction size  
252 analyzer, we demonstrate that by using simple mathematical formulae and a spreadsheet  
253 program it is possible to calculate the carbonate content and the grain size distribution of the  
254 carbonate fraction. A first application on samples from the Stage 5e–6 Pleistocene climatic  
255 transition in the Northern Atlantic Ocean with variable carbonate content indicates the  
256 reliability of the method. Thus, use of a laser diffraction size analyzer can be of great value in  
257 paleoceanographic studies. This rapid method must still take into account the sample quality,  
258 to avoid mistakes in interpreting the carbonate distribution.

259

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268

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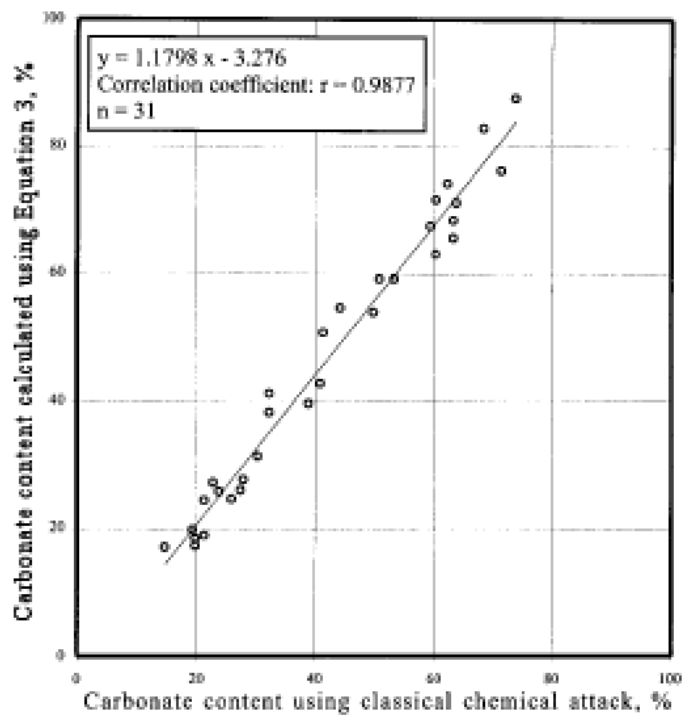
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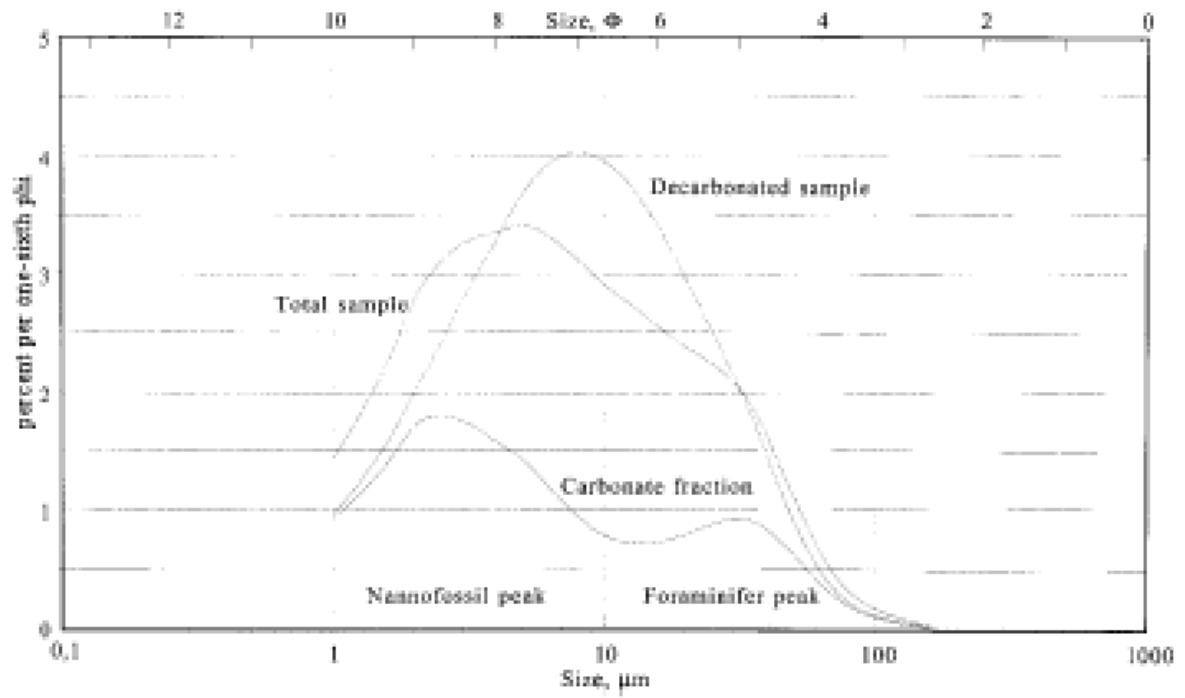
337 **Figure captions**



338

339 Fig. 1. Relationship between carbonate content measured by acid digestion and calculated  
340 using obscuration parameters on 31 Pleistocene samples from the Northern Atlantic Ocean  
341 (ODP Site 980). Values in %.

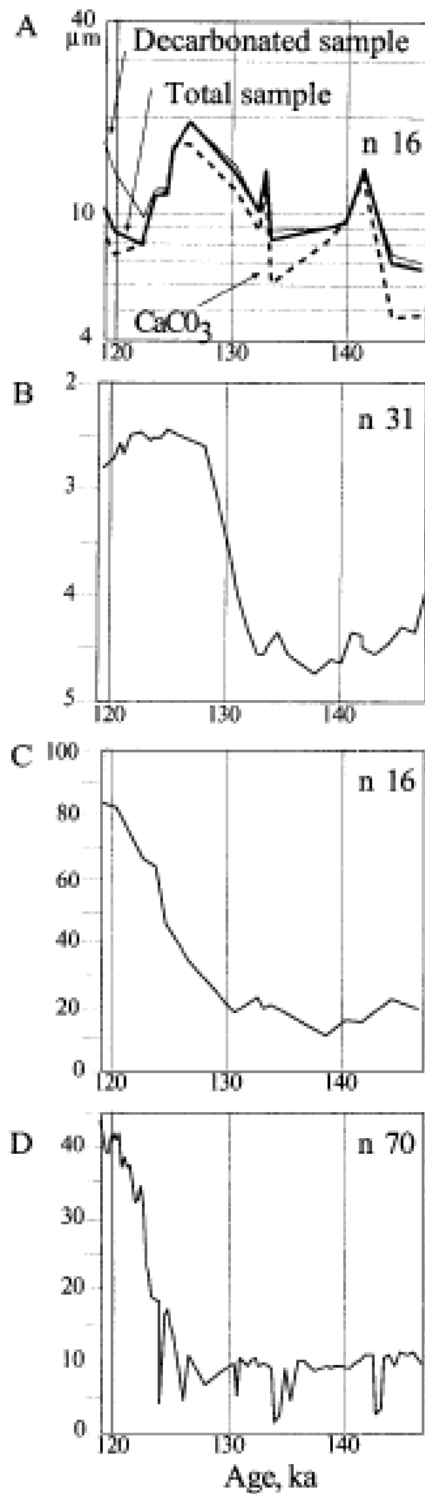
342



343

344 Fig. Grain-size distribution of the sediment before acid digestion (total sediment size  
 345 distribution: open squares), after acid digestion (decarbonated sediment size distribution:  
 346 distribution: black circles) and calculated carbonate size distribution (carbonate fraction size distribution:  
 347 distribution: black triangles). Sample: 980-C-3H03 016–018, 124.5 ka.

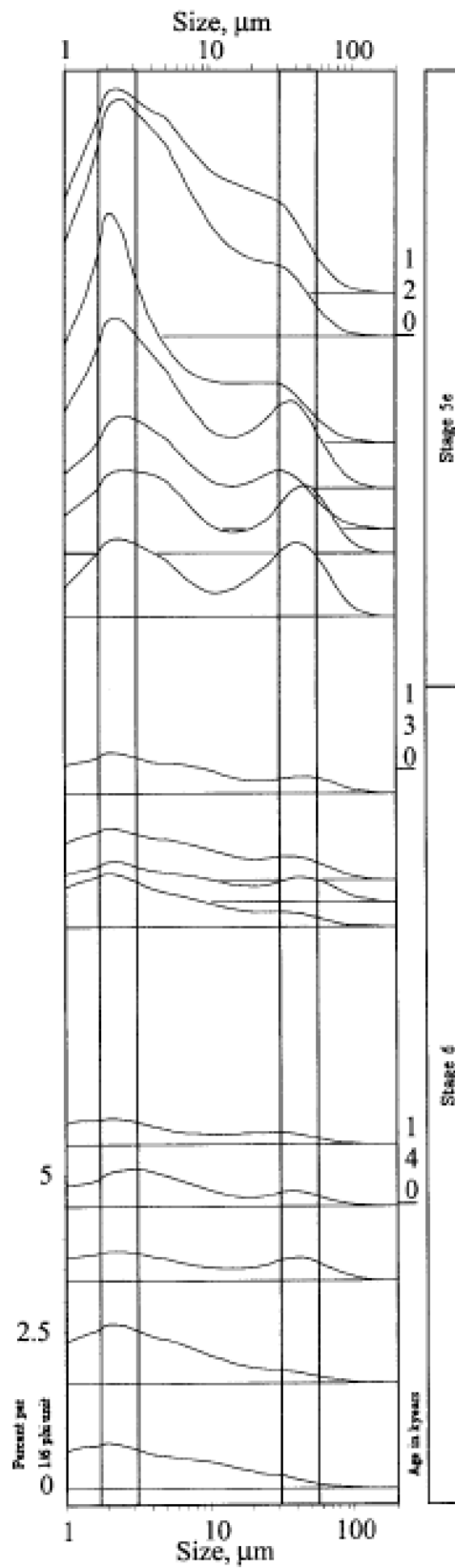
348



349

350 Fig 3. Variation of different parameters at the transition between the Pleistocene isotope stage  
 351 6 and 5e. **A)** Mean grain size measured on total sediment, decarbonated sediment, and  
 352 calculated carbonate fraction; **B)**  $\delta^{18}\text{O}$  variation (Jerry McManus, unpublished data, in ‰); **C)**  
 353  $\text{CaCO}_3$  content; **D)** Reflectance measured on the red (650 700 nm) band (Ortiz et al. 1999); *n*  
 354 = number of measurements.

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357 Fig 4. Grain size distribution of the carbonate fraction in the 16 samples from ODP Site 980.

358 The grey bars located at 2 to 3 microns and 30 to 60 microns represent the two modes,

359 nannofossils and foraminifers.