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4. Pliocene–Pleistocene Opal Records off Southwest Africa, Sites 1082 and 1084: a Comparison of Analytical Techniques[1]

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ABSTRACT

Two sites of the Ocean Drilling Program Leg 175 (Sites 1082 and 1084) in the Benguela Current system were investigated for their biogenic opal content in upper Pliocene and lower Pleistocene sediments. Both sites show a remarkable opal maximum, which corresponds to a distinct diatom maximum. The maximum spans the lower half of the Matuyama reversed polarity chron. Two different methods were used to measure opal abundance; similar variations in amplitude are found despite an offset in absolute values. We show that the different data sets can be merged, even though sample spacing differs. The results from the opal determinations are quite similar to the overall diatom abundance. In these sediments, therefore, diatoms are the chief contributors to the opal fraction.

INTRODUCTION

Ocean Drilling Program (ODP) Leg 175 shipboard and shore-based diatom studies, based on smear-slide analysis, showed evidence of a pronounced and prolonged maximum in diatom export production during the late Pliocene and early Pleistocene at Walvis Ridge (Site 1081) and in Walvis (Sites 1082 and 1083) and Cape Basins (Sites 1084 and 1085) (Wefer, Berger, Richter, et al., 1998). This prolonged maxi-

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mum is reached within the early Matuyama Chron; it is centered at ~2.2 Ma and follows a rapid increase in diatom deposition near 3.1 Ma (Lange et al., 1999).

In order to precisely define the Matuyama diatom maximum (MDM) for future comparison with other records in the region and elsewhere (e.g., equatorial Atlantic: Ruddiman and Janecek, 1989; subantarctic region: Froelich et al., 1991), we selected Sites 1082 and 1084 for the measurements of biogenic opal content (Fig. F1).

The goals of the present work are threefold: (1) to find ways to merge opal data from different laboratories for time series analysis, (2) to compare the Pliocene–Pleistocene opal records at Sites 1082 and 1084, and (3) to establish whether overall diatom abundance constitutes a reliable proxy for opal content.

Several techniques have been used to measure the biogenic opal content in marine sediments. The wet alkaline methods are the most widely used. Advantages and limitations of the most common extraction procedures were discussed by DeMaster (1991), illustrating the variations in the methodology and showing that different methods do not necessarily give the same results. More recently, Conley (1998) made an interlaboratory comparison that confirmed a wide range of variability in the measurement of biogenic opal, though no significant differences were found between the two most widely used techniques: (1) DeMaster (1981) and (2) Mortlock and Froelich (1989). In this study, we demonstrate that these two methods delivered results that can be merged.

F1. Location of Site 1082 opal data, p. 11.

STUDY AREA

The Angola-Benguela Current system is one of the major upwelling regions of the world, intermediate in intensity between the systems off Peru and California. It is characterized by organic-rich sediments that provide an excellent record of productivity history, which in turn is closely related to regional dynamics of circulation, mixing, and upwelling (Wefer, Berger, Richter, et al., 1998).

The Benguela Current (BC) originates near the Cape of Good Hope. On its way northward and at ~28°S, the BC divides into an oceanic (Benguela Oceanic Current) and a coastal branch (Benguela Coastal Current [BCC]) (Stramma and Peterson, 1989). The cool and nutrient-rich upwelled waters of the BCC are fed from the thermocline underlying South Atlantic central water, which in turn originates at the subtropical-subantarctic front by mixing and sinking of subtropical and subantarctic surface water (Lutjeharms and Valentine, 1987).

The Benguela upwelling area can be divided into a true coastal upwelling regime, averaging ~150–200 km wide, that is composed of several distinct upwelling cells (Lutjeharms and Meeuwis, 1987) and a filamentous mixing domain streaming offshore, with filaments that may exceed 1000 km in length (Lutjeharms et al., 1991).

Site 1084 (25°30.8′S, 13°1.7′E; water depth 1991.9 m) lies close to the largest and most active coastal upwelling center, the Lüderitz cell. Upwelling in this area is characterized by year-round high primary production, and phytoplankton assemblages are generally dominated by diatoms (Shannon and Pillar, 1986).

Site 1082 (21°5.6′S, 11°49.2′E; water depth 1279.3 m) is located at a crucial latitude where the BC turns westward into the open ocean.

Thus, the sediments from this site represent an important key for reconstructing the history of the BC.

METHODOLOGY AND STRATIGRAPHY

Sediment samples were taken at about every 50–100 cm of the Pliocene–Pleistocene level of Hole 1082A. The biogenic opal content (see "Appendix," p. 9) was determined by two methods based on wet chemical extraction techniques:

1. "OLin" data: At the National Sun Yat-Sen University (Taiwan), Lin analyzed 80 samples over a depth interval from 121.86 to 261.96 meters below sea floor (mbsf), following the basic leaching method of Mortlock and Froelich (1989). This method involves a single extraction of silica with an alkaline solution at 85°C for 5 hr and the measurement of the dissolved silicon concentration in the extract by molybdate-blue spectrophotometry. Instead of the 2-N Na_2CO_3 suggested by Mortlock and Froelich (1989), 0.5-N NaOH was employed as the basic leaching chemical to ensure complete dissolution. The HCl was replaced by a milder 0.5-M glacial acetic acid solution to help prevent the breakdown of silicates, which would result in overextraction of silicate from the sediment (Murray et al., 1995).
2. "OPer" data: At the University of Bremen (Federal Republic of Germany), Pérez analyzed 142 samples from 100.25 to 250.47 mbsf and 104 samples from 298.6 to 399.5 mbsf using the automated leaching method of Müller and Schneider (1993), which is a modification of the manual sequential leaching method of DeMaster (1981). While stirred constantly, the opal is extracted with 1-M NaOH at 85°C in a stainless steel vessel. The extraction time varies according to the sample type (e.g., sediment type, opal concentration, and presence of clay minerals), and the increase in dissolved silica is continuously monitored as a minor portion of the leaching solution is cycled to an autoanalyzer and analyzed for dissolved silicon by molybdate-blue spectrophotometry. The resulting absorbance vs. time plot is then evaluated following the extrapolation procedure of DeMaster (1981). The weak point in the manual technique is that the slope—for example, the increase in dissolved silica extracted with time—and the extrapolated intercept value are based only on a few measurements. The automated technique of Müller and Schneider (1993) reduces the degree of uncertainty in the determination of the intercept.

Sediment samples from Hole 1084A were taken at about every 150 cm throughout the uppermost 476 mbsf, which represents approximately the last 3 m.y. For this site, the biogenic opal content was determined by the modified "OLin" method only.

Overall diatom abundance and species composition were determined based on smear-slide analysis with phase-contrast illumination at a magnification of 400×. A diatom abundance index (DAI) was established using the following convention: 6 (very abundant or dominant) = >50%; 5 (abundant) = 35%–50%; 4 (common) = 20%–35%; 3 (few) = 5%–20%; 2 (rare) = 1%–5%; 1 (trace) = <1%; 0 (barren) = no diatoms in sample (Lange et al., 1999).

After converting depths (mbsf) to meters composite depth (mcd), age models at Sites 1082 and 1084 were derived according to shipboard magnetostratigraphy and nannofossil datums (Shipboard Scientific Party, 1998a, 1998b).

RESULTS

In Figure F2 we present the opal values (in weight percent) from 137.52 to 266.13 mcd at Site 1082, the interval analyzed by the two laboratories. The two records show the same general pattern but show offsets in absolute numbers. OLin values tend to be higher, averaging 23.6% (N = 50), whereas OPer values have an average of 18.3% (N = 50), comparing the closest neighbors (<4 cm offset) in the overlapping portion of the record.

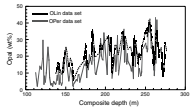
Reproducibility in the two procedures was estimated from both duplicate and replicate determinations. The pooled precision was ±1.37% opal for duplicate determinations of 23 OLin samples and ±0.6% opal for duplicate determinations of 42 OPer samples. Replicate determinations of nine OLin samples with an average content of ~15%–20% opal yielded a standard deviation of ±1.42% opal; replicate measurements of three OPer samples with ~8%–15% opal gave a standard deviation of ±0.55% opal. Table T1 and Figure F3 show the differences between the two techniques for selected OLin and OPer samples (opal-poor and opal-rich samples). The error is larger for samples with the lower opal content.

We tested whether a regression of OPer data points vs. OLin data points was sufficiently precise to transform OPer to OLin values and vice versa. Because OLin and OPer opal measurements were not done on aliquots from the same samples, we followed two approaches: the closest neighbor method and the linear interpolation method. For the closest neighbor method, only those samples taken at nearby depth intervals (<4 cm offset) were considered in the correlation (N = 50). Alternatively, estimates of the OPer opal content for depth intervals corresponding to OLin samples were obtained by linear interpolation from the OPer values (N = 70) (Fig. F4A, F4B). The data points outside the 95% confidence interval were removed for the calculation of the regression. We found that a linear regression had the highest correlation coefficient of several possible transforms in both approaches (r^2 = 0.84 and r^2 = 0.86, respectively).

By using the linear regression equation given by the linear interpolation method (Fig. F4B), OPer data were converted to OLin data. Subsequently, the estimated OPer data set and the measured OLin data set were merged to obtain a more detailed and extended opal record of Site 1082. The merged data set could then be compared to the opal record of Site 1084, which was analyzed with only the OLin method. Figure F5A shows a similar pattern at both sites with a distinct opal maximum during the late Pliocene and early Quaternary. Because of the location of Site 1084, close to the Lüderitz upwelling cell, opal values were higher at this site than at Site 1082.

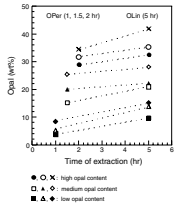
DAI, as estimated in Lange et al. (1999), yielded the same patterns at both sites (Fig. F5B, F5C). The diatom maximum is characterized by high abundances of *Thalassiothrix* and other pelagic species in addition to the *Chaetoceros* spores indicating coastal upwelling (Lange et al., 1999).

F2. Comparison of Site 1082 opal data from two laboratories, p. 12.

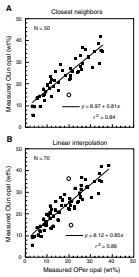


T1. Mean opal concentrations and distance to the mean of OLin and OPer samples, p. 16.

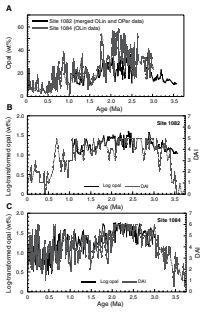
F3. Amount of opal extracted with time for selected OLin and OPer samples, p. 13.



F4. Relationships between OLin and OPer opal values, p. 14.



F5. Opal records at Sites 1082 and 1084, p. 15.



DISCUSSION AND CONCLUSIONS

Little can be said about the relative reliability of the two chemical methods used because of the absence of absolute sediment standards for determining the accuracy of any biogenic opal technique. In any case, most of the differences could be due to inhomogeneities among samples rather than reflecting the accuracy of the techniques. Paradoxically, OLin values were consistently higher, even though a weaker digestion solution (0.5-N NaOH vs. 1-N NaOH in OPer method) was used.

One source of the discrepancy between OLin and OPer results may be the extraction of silica from coexisting marine clays and authigenic silicates in the OLin technique. Experimental and field studies (Berner, 1981; DeMaster et al., 1983) have reported the preferential dissolution of ultrafine particles, such as clays, during the initial extraction period. Therefore, excessive grinding of the samples might have erroneously increased the opal content in OLin samples, especially those with low opal contents. In addition, OLin samples were extracted for 5 hr, whereas in OPer samples, the extraction time was dependent on the sample type (Fig. **F3**). Opal-poor samples were dissolved within 1 hr, whereas 2 hr were required for opal-rich samples. The 5-hr dissolution in OLin samples could have resulted in silica being leached from clay minerals. However, we cannot rule out the possibility of incomplete digestion of diatoms in OPer data. Extraction curves show an early spectral absorbance increase due to the preferential dissolution of opal followed by a slower, linear increase representing the dissolution rates of the silicate minerals present. Although dissolution curves appeared linear after 1 and 2 hr, longer periods might have been required to extract 100% of the biogenic opal in the samples.

Despite all difficulties, the overall patterns between OLin and OPer data are strikingly similar. The results from the two techniques are comparable and provide the opportunity to combine the two records by applying a linear transform (Fig. **F4A, F4B**). Thus, longer and more detailed opal records for subsequent time series analysis could be obtained by splitting samples among laboratories and merging the results after a proper calibration and statistical treatment.

Interestingly, the substantial increase in the concentration of opal at 2.6 Ma (Fig. **F5A**) is similar to that found at the northern and southern boundaries of the BC system. In the equatorial Atlantic (ODP Sites 662, 663 and 664), Ruddiman and Janecek (1989) reported abrupt upper Pliocene increases in opal and terrigenous dust fluxes near 2.5 Ma. Also, in the subantarctic region (ODP Site 704), Froelich et al. (1991) showed a marked increase in the accumulation rates of biogenic silica at ~2.5 Ma. Cieselski and Grinstead (1986) identified the latest Gauss/early Matuyama (2.67–2.47 Ma) as the time of greatest change in Neogene climate in the northern antarctic and subantarctic regions.

The comparison of opal content with DAI at both sites (Fig. **F5B, F5C**) indicates that the DAI is a good proxy for opal concentration in sediments, at least in coastal upwelling areas. Studies on sediment traps at Walvis Ridge also showed that diatoms closely parallel variations in opal fluxes (Treppke et al., 1996). On the other hand, it is noteworthy that DAI has a logarithmic nature, which derives from the convention used in the smear-slide analysis (see **"Methodology and Stratigraphy,"** p. 3).

The evidence of high opal and diatom deposition in the upper Pliocene and the specific composition of the diatom assemblage lead us

to suggest that for the area of the Benguela Current system, a combination of increased upwelling and intensified advection of silica-rich subsurface waters must have existed at MDM time. The merging of different data sets describing the variations of opal deposition in and around the MDM will make more detailed studies of this feature possible.

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APPENDIX

Percentage of opal (OLin and OPer data) in sediment samples from Hole 1082A. (Continued on next page.)

Depth (mbsf)	Opal (wt%)
OLin data	
121.86	9.87
123.10	10.39
123.88	15.09
124.86	16.21
125.86	16.26
126.88	10.27
127.86	12.19
128.85	9.17
130.29	9.60
132.28	18.06
133.88	22.76
134.72	25.59
138.40	13.67
140.38	19.04
142.36	16.45
144.36	5.65
149.98	12.52
157.66	17.31
159.35	27.59
161.31	13.50
167.26	20.91
168.11	36.43
171.41	19.51
176.96	22.27
178.07	27.88
179.98	24.13
181.80	27.50
187.50	32.52
189.48	23.90
191.03	31.34
192.58	9.71
196.26	26.94
198.28	26.13
200.22	20.67
205.96	35.07
207.96	23.61
211.79	25.79
212.77	24.22
215.56	25.95
216.56	23.74
217.59	20.62
222.35	29.63
218.32	21.88
219.32	14.33
220.33	32.12
221.35	25.56
225.26	14.87
226.30	22.08
227.26	20.39
228.24	17.75
229.24	19.82
230.25	26.55
234.86	35.81
235.86	36.72
236.87	31.76
237.86	35.00
238.86	41.87
239.88	42.34
240.86	38.66
241.86	33.31
242.88	28.05
243.86	25.75
244.46	14.46
245.46	14.75
246.46	22.52
247.46	28.83
248.46	35.40
249.72	29.34
250.43	26.78
251.43	19.15
254.06	25.31
255.06	22.12
256.08	7.58
257.06	11.27
258.06	16.45
259.08	28.53
259.96	26.02
209.13	32.13
260.96	23.26
261.96	23.13
OPer data	
100.25	10.02
101.25	6.03
102.25	2.05
103.24	14.02
104.24	13.05
105.11	12.14
106.44	18.80
107.29	29.67
108.29	3.24
109.29	8.09
110.29	4.40
111.29	10.06
112.30	25.33
113.40	17.79
114.40	7.21
115.52	3.20
116.23	2.53
117.23	3.64
118.21	1.74
119.26	4.92
121.900	3.06
122.44	3.81
123.90	8.51
124.45	5.68
125.40	14.32
126.40	3.14
127.40	8.84
128.32	4.27
129.20	2.58
130.32	3.97
131.32	16.43
132.30	11.17
133.42	23.47
134.25	21.89
138.40	5.29
139.40	7.91
140.41	13.00
142.39	9.23
143.39	2.54
143.90	1.52
144.40	3.29
148.00	6.05
149.00	5.13
150.00	8.68
151.00	9.00
152.11	4.44
157.7.0	15.14
158.71	14.64
159.86	17.02
160.85	7.28
161.85	16.85
162.81	12.05
164.11	20.89
167.30	15.18
168.61	22.72
169.61	25.87
170.45	17.55
171.45	10.05
172.40	9.06
177.00	20.13
178.09	30.57
179.02	20.85
180.02	18.38
181.34	17.85
182.32	33.94
186.60	8.96
187.52	28.88
188.52	31.10
189.50	20.47
190.52	26.58
191.57	24.67
192.60	6.74
193.10	9.37
196.30	20.57
197.30	10.97
198.30	17.21
199.42	32.45
200.42	11.42
200.92	8.82
206.00	31.74
207.50	36.10
208.16	16.56
208.66	16.98
209.16	29.10
209.66	25.49
210.66	16.37
211.16	15.46
211.31	18.18
211.82	21.51
212.31	17.11
215.60	13.94
216.10	13.10
216.60	11.24
217.11	12.53
217.61	11.21
218.11	16.20
218.36	13.43
218.86	10.53
219.36	6.51
219.86	30.09
220.86	27.21
221.39	23.83
221.89	13.83
222.39	26.00
225.30	20.12
225.80	7.83
226.28	12.03
226.78	12.67
228.28	8.28
228.78	8.52
229.28	9.94
229.78	10.67
230.76	24.10
234.90	27.79
235.40	24.73
235.90	26.02

Appendix (continued).

Depth (mbsf)	Opal (wt%)	Depth (mbsf)	Opal (wt%)
236.40	24.61	332.79	10.53
236.90	22.78	333.29	11.18
237.40	24.37	333.79	8.85
237.90	35.73	334.29	13.56
238.40	40.04	334.79	13.53
238.90	34.37	335.29	10.35
239.40	40.72	335.79	12.79
240.90	33.71	336.29	5.68
241.40	30.01	336.78	8.34
241.90	25.23	341.20	9.58
242.40	24.73	341.70	9.88
242.91	25.40	343.20	7.86
243.40	24.97	343.70	13.34
243.90	24.33	344.21	11.54
244.50	11.86	344.70	17.91
245.00	21.46	345.20	16.74
245.50	8.30	345.70	18.16
246.00	11.77	346.20	11.44
246.50	20.36	346.70	12.73
247.00	43.24	347.20	13.41
247.50	15.88	347.70	12.04
248.00	27.64	348.20	13.91
248.50	34.89	348.70	11.99
249.00	41.84	350.90	3.68
249.70	34.11	351.90	8.14
250.47	25.89	352.90	5.02
298.60	13.09	353.72	7.60
299.08	14.99	354.72	3.10
302.70	11.21	355.54	4.93
303.20	14.07	356.02	2.58
303.7	11.61	360.92	5.73
304.20	17.66	361.92	7.93
304.70	16.28	362.79	8.35
305.20	23.30	363.79	8.82
306.21	25.02	364.79	4.86
306.74	29.62	365.79	7.22
307.24	35.20	369.60	8.39
307.74	37.41	370.60	8.75
308.24	46.65	371.60	1.94
308.74	39.86	372.60	0.81
309.24	31.28	373.60	0.54
312.30	16.64	374.60	0.80
312.80	12.20	375.60	1.90
313.30	12.87	376.60	2.34
313.80	15.61	377.60	3.67
314.30	17.61	379.80	3.56
314.82	12.69	380.80	4.06
315.32	16.23	381.80	3.56
315.82	12.38	383.80	4.32
316.28	15.42	384.80	6.74
316.78	15.23	385.80	4.14
322.00	3.91	386.30	3.09
323.00	10.06	388.90	3.51
323.50	4.75	389.90	3.66
326.00	7.33	390.90	4.32
326.50	7.26	391.59	2.40
327.00	4.97	392.63	1.00
327.50	12.54	393.59	0.82
328.00	6.71	394.59	0.50
331.29	8.88	395.59	0.59
331.79	6.08	398.50	1.12
332.29	9.70	399.50	0.56

Figure F1. Locations of Sites 1081–1085 on the southwest African margin (modified from Wefer, Berger, Richter, et al., 1998).

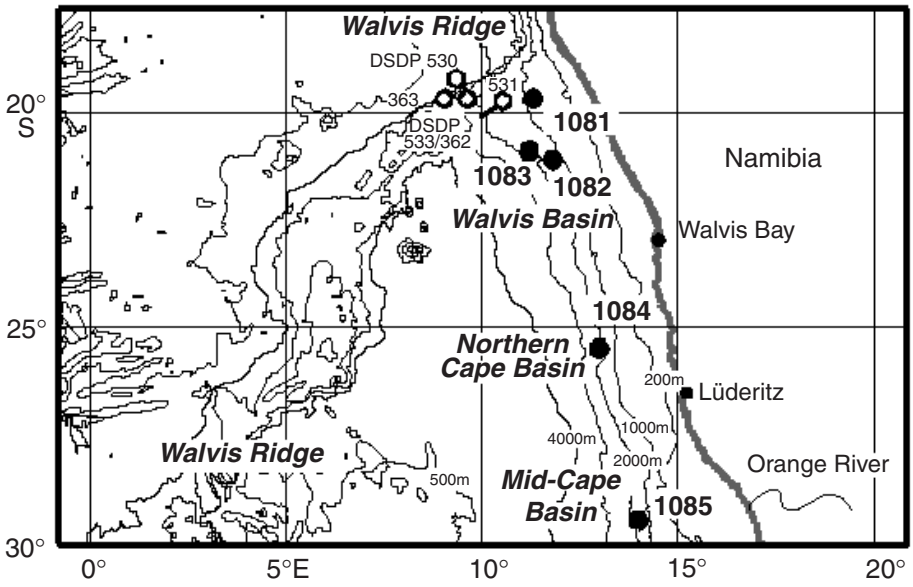

Figure F2. Comparison of Site 1082 opal data from two laboratories, using two different wet alkaline techniques (OLin = modified Mortlock and Froelich, 1989; OPer = Müller and Schneider, 1993).

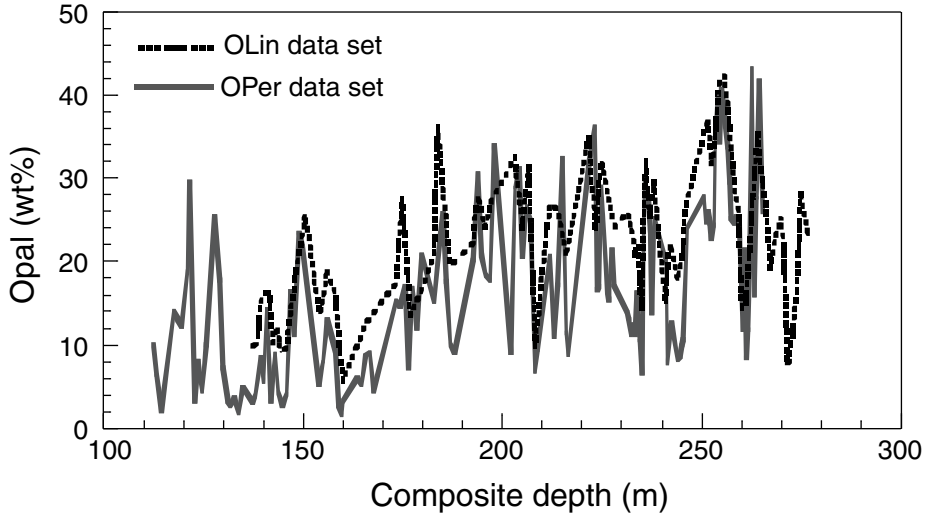

Figure F3. Amount of opal extracted with time for selected OLin and OPer samples. OLin data points are the result of discrete analysis after 5 hr of extraction, whereas OPer data points are determined from the linear part of the continuous leaching diagram after 1–2 hr of extraction.

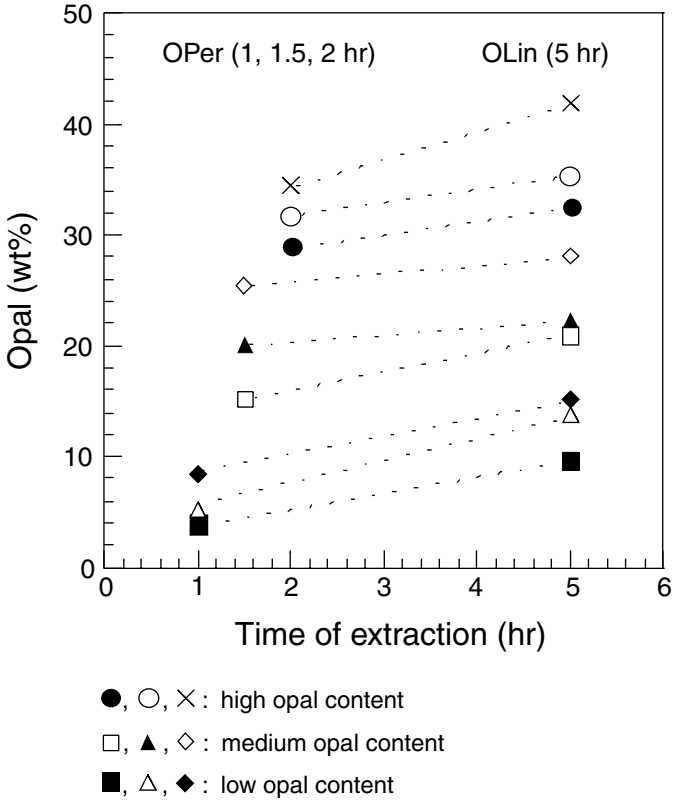

Figure F4. A. Relationship between measured OLin and OPer opal values over the same sedimentary interval. **B.** Relationship between measured OLin and linearly interpolated OPer opal values. Outlying points (open circles) were dismissed for the calculation of the regression line (see "Results," p. 4).

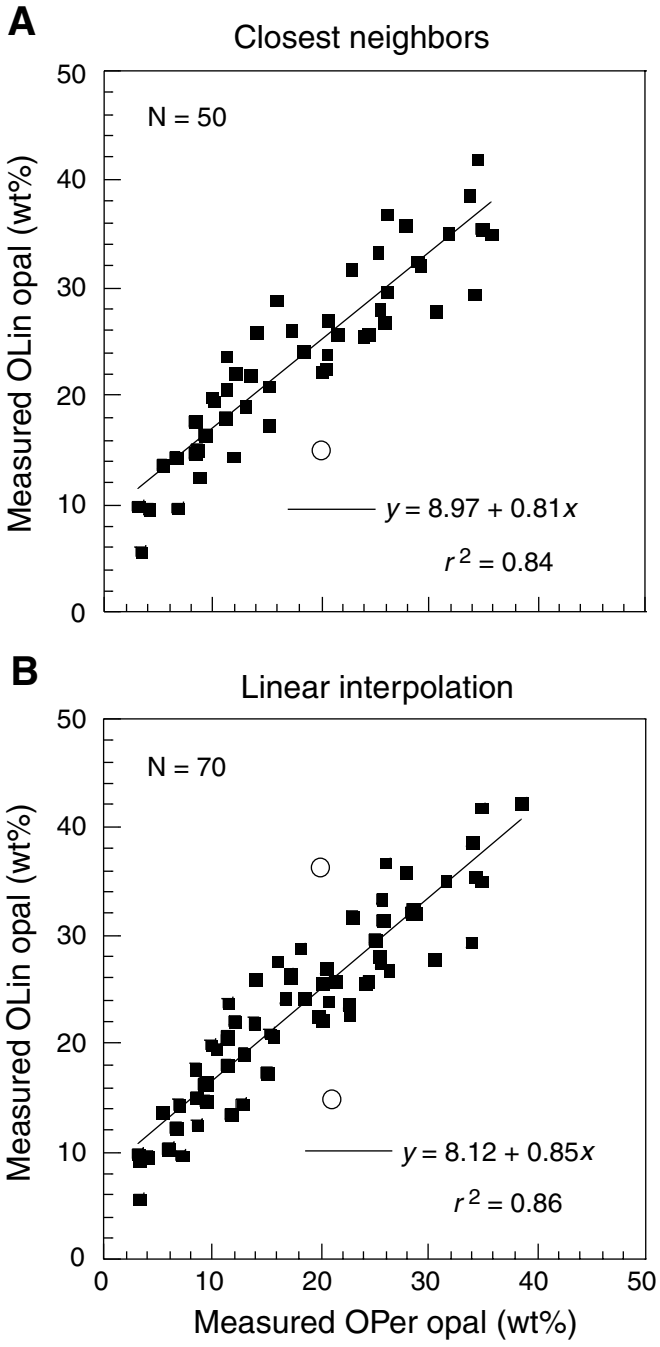

Figure F5. **A.** Opal record at Sites 1082 and 1084. **B, C.** Deposition of opal transformed to log and diatoms at Sites 1082 and 1084. DAI = diatom abundance index.

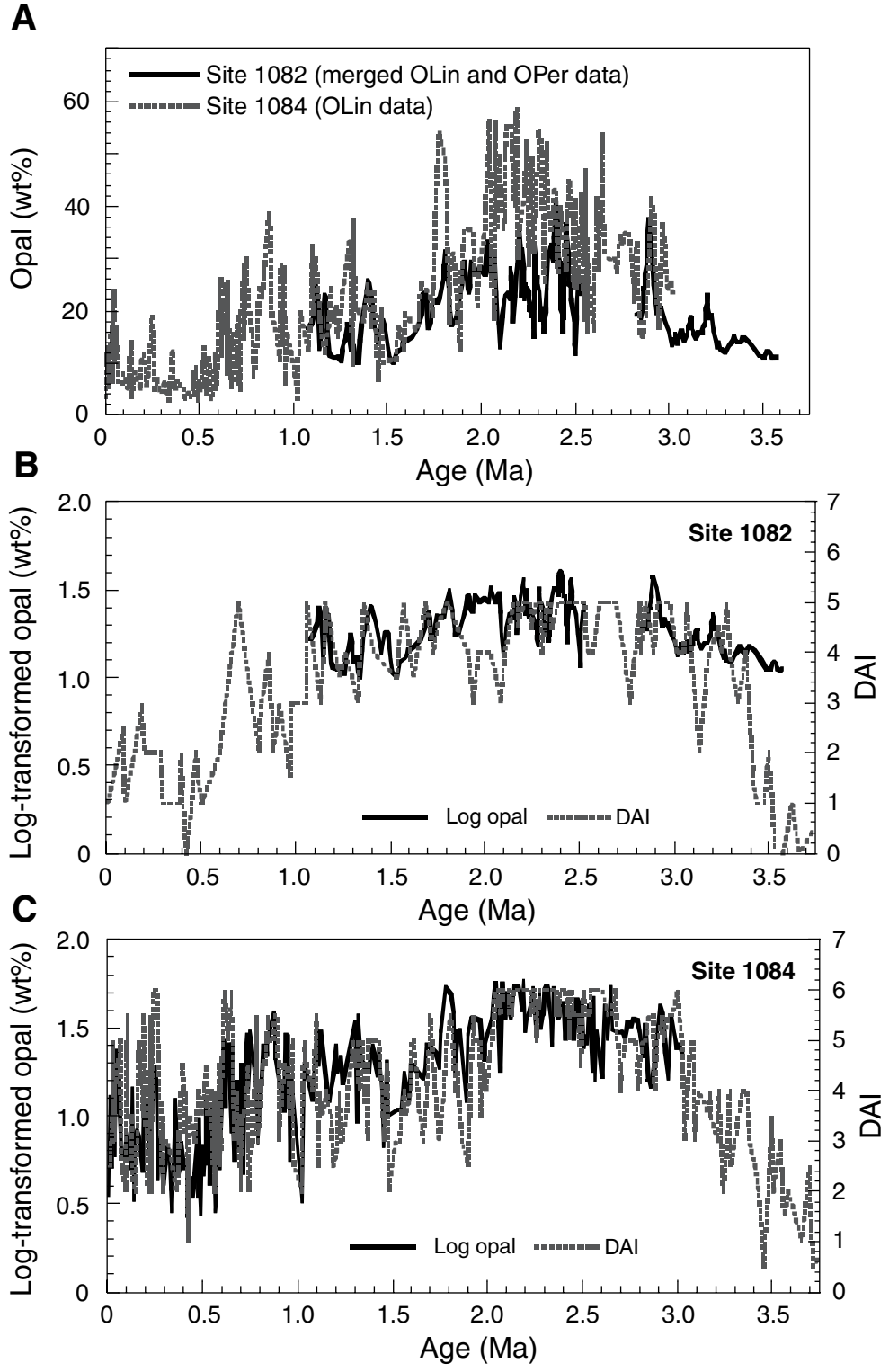

Table T1. Mean opal concentrations and distance to the mean of OLin and OPer selected samples.

OLin depth (mbsf)	OLin opal (wt%)	OPer depth (mbsf)	OPer opal (wt%)	Mean	Distance to mean (wt%)	Distance to mean (%)
121.86	9.87	121.90	3.06	6.46	3.40	52.6
130.29	9.60	130.32	3.97	6.78	2.81	41.5
138.40	13.7	138.40	5.29	9.48	4.19	44.2
228.24	17.7	228.28	8.28	13.0	4.74	36.4
132.28	18.1	132.30	11.2	14.6	3.44	23.6
140.38	19.0	140.41	13.0	16.0	3.02	18.9
226.30	22.1	226.28	12.0	17.0	5.02	29.5
167.26	20.9	167.30	15.2	18.0	2.86	15.9
189.48	23.9	189.50	20.5	22.2	1.72	7.73
242.88	28.0	242.91	25.4	26.7	1.33	4.96
187.50	32.5	187.52	28.9	30.7	1.82	5.92
205.96	35.1	206.00	31.7	33.4	1.67	4.99
238.86	41.8	238.90	34.4	38.1	3.75	9.84

Note: OLin = modified from Mortlock and Froelich (1989), OPer = Müller and Schneider (1993).