

ing five hydrogen atoms were subsequently located on difference Fourier maps after two iterations. The maximum peak height on a complete difference Fourier map was 0.18 electron per cubic angstrom. Full-matrix least-squares refinement of 117 parameters included an isotropic extinction parameter, anisotropic thermal parameters for the nonhydrogen atoms, and isotropic thermal parameters for the hydrogen atoms. The y coordinate of the central carbon atom was held constant. The atomic scattering factors found in the *International Tables for Crystallography* (Kynoch, Birmingham, England, 1974), vol. 4, were used; those for C, N, and O are found on p. 73, and the H scattering factors are listed on p. 102. Refinement continued until the parameter shifts were less than 2×10^{-7} with a goodness-of-fit of 0.902. The final agreement factors were $R = 0.026$ and R_w (weighted R value) = 0.038.

9. R. Rudman, *J. Chem. Phys.* **66**, 3139 (1977).
10. The research at Adelphi University was supported under NSF grants CHE 75-13935 and DMR 76-20639.

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Chemistry of Oceanic Particulate Matter and Sediments: Implications for Bottom Sediment Resuspension

Abstract. Analyses of suspended particulate matter from the eastern equatorial Pacific Ocean have defined a 400-meter-thick benthic nepheloid layer enriched in aluminum, silicon, iron, and manganese relative to the overlying waters. Chemical mass-balance calculations suggest that the concentration increases in the benthic nepheloid layer are due to resuspension from the fraction of the local bottom sediments in the size range ≤ 1 micrometer.

A benthic nepheloid layer (BNL) is nearly ubiquitous in the deep sea (1), but the processes that generate and maintain this feature are poorly understood. Light scattering profiles and current meter measurements in high-energy environments along the continental margins (2) and in abyssal topographic gaps such as the Vema Channel (3) suggest that the thickness and intensity of the BNL are a function of current speed, bathymetry, or both; this is strong circumstantial evidence that bottom sediment resuspension is an important factor in maintaining the BNL. The efficiency of resuspension in the lower-energy environments of the ocean basins, however, is inherently more suspect. Compositional factors of oceanic particulate matter can be sensitive indicators of benthic processes (4), although sampling and analytic difficulties have severely limited this approach in the deep sea. We report here on changes in the particulate chemistry within the BNL relative to both the overlying clearer water and the underlying bottom sediments. Our evidence suggests that significant resuspension of the fine fraction ($\leq 1 \mu\text{m}$) of deep-sea sediments can occur under the influence of even moderate benthic currents (mean speed $< 8 \text{ cm/sec}$).

Samples of suspended particulate matter (SPM) were collected in 30-liter polyvinyl chloride sampling bottles from depths below 450 m at three stations (A at $8^\circ 27' \text{N}$, $150^\circ 47' \text{W}$; B at $11^\circ 42' \text{N}$, $138^\circ 24' \text{W}$; and C at $15^\circ 00' \text{N}$, $126^\circ 00' \text{W}$) in the eastern equatorial Pacific manganese nodule province. The particulate matter was collected on Nuclepore membrane filters (pore size, $0.4 \mu\text{m}$) by closed-system filtration and analyzed for Al, Si, Mn, and Fe by secondary emis-

sion thin-film x-ray fluorescence (5). A sample of the surficial bottom sediment was collected at each station by a box corer.

The regional thickness of the BNL, on the basis of sharply increased Al percentages in the SPM, is approximately 400 m (Fig. 1). Total SPM concentrations within this layer are 10 to 40 percent above the midwater average of $\sim 9 \mu\text{g/liter}$. Scatter diagrams of the Si, Mn, and Fe concentrations versus the Al concentration (Fig. 2) define the chemical characteristics of the BNL and describe some important interelement relationships.

Particulate Si (Fig. 2a) is mostly biogenic ($> \text{about } 75 \text{ percent}$) above the BNL, its average concentration is 12.3 percent by weight, and it is poorly correlated with Al ($r = .25$). Within the BNL, Si is highly correlated with Al ($r = .89$), although the average Si/Al ratio (4.3) is higher than the average for eastern central Pacific sediments (~ 3.0) (6), which

suggests the presence of some residual biogenic Si debris, particularly since these stations are positioned above the equatorial band of siliceous ooze (6). The pattern for Fe and Mn is more complex. The percentages of both elements increase significantly within the BNL relative to the overlying waters (Fig. 2, b and c), but their variation within the BNL is only moderately correlated with that of Al, signifying that at least one other phase besides the aluminosilicates influences the distribution of Fe and Mn.

The BNL in this region may be due to either or both of two effects: accumulation of sinking particles from water layers at the surface and middle depths, or resuspension of some fraction of the bottom sediments, either locally or up-current from the study area. The accumulation hypothesis requires an extensive change in the particulate chemistry as the sinking suspended matter reaches the BNL. An increase in the weight percentages of Al, Si, Fe, and Mn would result if other fractions low in these elements—such as CaCO_3 and particulate organic matter—were preferentially lost from the BNL, but the observed increases in absolute SPM concentration and the highly variable enrichment (0.9 to 5.1) of these elements within the BNL argue against this mechanism. Similarly, a complicated differential settling scheme might be devised for each station to produce the observed enrichment, but would require unrealistic settling velocity differences to account for the two- to fivefold increases in Al within the BNL.

Alternatively, resuspension can alter the BNL chemistry by selectively injecting a chemically distinct fraction of the bottom sediment. Samples of the surficial bottom sediment from each station were therefore size-fractionated by settling in distilled water, and portions of the fine ($\leq 1 \mu\text{m}$) fraction (44 to 76 per-

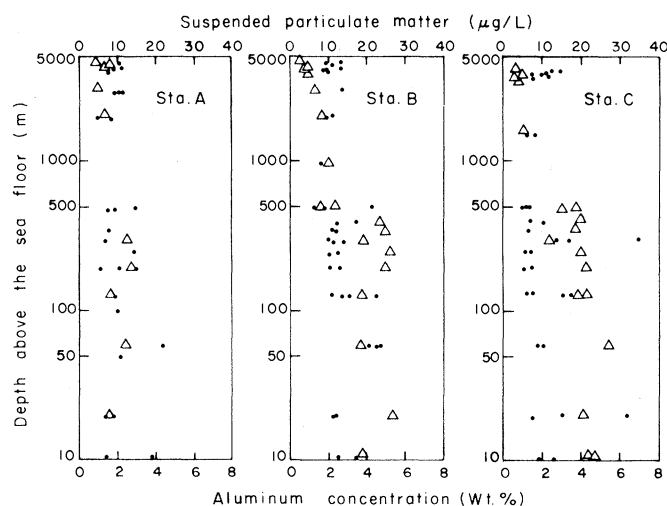


Fig. 1. Suspended particulate matter concentration (●) and percentage Al by weight (Δ) in the water column at three stations in the eastern equatorial Pacific. Note that the depth scale is logarithmic.

cent of the total) and of the total sample were analyzed by the methods used for the SPM samples. The possibility that resuspension generates the BNL was tested by calculating the theoretical chemical composition of a BNL created by addition of the fine fraction of the local bottom sediment in an amount equal to the concentration increase observed at each station (Table 1).

Agreement between the calculated values of Al, Si, Fe, and Mn and the observed BNL values is excellent in nearly

all cases: only Al and Si at station C do not agree within 1 standard deviation (S.D.) (7). The composition of the total fraction of the bottom sediment is also given in each case in Table 1 and shows that Fe is enriched and Si and Mn are depleted in the fine fraction, whereas Al is about the same in both fractions.

Long-term (6 months at A and B and 1 month at C) benthic current meter measurements for each station reveal an increase in the mean speed in going from A to C. The mean and maximum (hourly

average) current speeds were at A (at 30 m above bottom), 2.1 and 10.1 cm/sec; at B (at 30 m above bottom), 5.2 and 13.4 cm/sec (8); and at C (at 50 m above bottom), 8.1 and 18.5 cm/sec (10-minute average) (9). The measured currents are well correlated with the observed BNL parameters, station A having by far the weakest BNL and lowest Al values.

The limiting factors in any experimental attempt to measure the effect of observed currents on the deep-sea bed are (i) the difficulty of accurately duplicating in a laboratory the geotechnical (water content and shear strength) and biological properties of the surface microlayer of oceanic sediments, a limitation that could be overcome by flume measurements in situ (10); and (ii) the impossibility of observing a controlled experiment over geological time scales. Nevertheless, flume observations indicate that an artificial bed of pelagic clay roughened with manganese nodules can be rapidly eroded by a current (1 m off the bed) as weak as 12 cm/sec provided the water content of the clay is increased to 84 percent (11), about 20 percent higher than typical deep-sea "surface" measurements on sediments retrieved by coring devices (12). In fact, there is some experimental evidence which implies that this threshold of rapid erosion is merely an observational one and that for any applied shear stress there will be some erodible clay flocs (13). We suggest, therefore, that the resuspension inferred by our chemical mass-balance considerations could be generated by long-term action of the relatively weak benthic flow, although the influence of brief and infrequent occasions of maximum current speed might be considerable.

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References and Notes

1. M. Ewing and S. D. Connary, *Geol. Soc. Am. Mem.* 126 (1970), p. 41; S. Eittreim, A. L. Gordon, M. Ewing, E. M. Thorndike, P. Bruckhausen, in *Studies in Physical Oceanography*, A. L. Gordon, Ed. (Gordon & Breach, New York, 1972), p. 19; W. S. Plank, H. Pak, J. R. V. Zaneveld, *J. Geophys. Res.* 77, 1689 (1972); R. A. Feely, L. Sullivan, W. M. Sackett, in *Suspended Solids in Water*, R. J. Gibbs, Ed. (Plenum, New York, 1974), p. 281; P. E. Biscaye and S. L. Eittreim, *Mar. Geol.* 23, 155 (1977).
2. P. E. Biscaye and S. L. Eittreim, in *Suspended Solids in Water*, R. J. Gibbs, Ed. (Plenum, New York, 1974), p. 227; E. T. Baker, *Mar. Geol.* 21, 67 (1976).
3. D. A. Johnson, S. E. McDowell, L. G. Sullivan, P. E. Biscaye, *J. Geophys. Res.* 81, 5771 (1976).
4. R. A. Feely, *Mar. Chem.* 3, 121 (1975); P. R. Betzer and M. E. Q. Pilson, *Deep-Sea Res.* 18, 753 (1971).

Table 1. Chemical mass-balance calculations for the benthic nepheloid layer (BNL). The above-BNL particulate matter and the fraction of the bottom sediment $\leq 1 \mu\text{m}$ are assumed to be mixed in proportion to the mass concentration increase observed within the BNL at each station (10.6:1 at A, 2.5:1 at B, and 3:1 at C). The accuracy of the model can be assessed by comparing the calculated BNL composition with the observed composition. Calculated and observed composition values are given as mean ± 1 S.D.

Element	Observed, above BNL (%)	Bottom sediment ($\leq 1 \mu\text{m}$) (%)	Calculated, BNL (%)	Observed, BNL (%)	Bottom sediment (total) (%)
Station A					
Al	1.1	7.6	1.7 ± 0.2	2.4 ± 0.8	6.3
Si	14.2	22.8	15.0 ± 2.0	12.4 ± 4.1	26.3
Fe	2.5	8.6	3.0 ± 1.2	3.5 ± 1.3	6.0
Mn	0.049	0.359	0.076 ± 0.020	0.085 ± 0.027	0.948
Station B					
Al	1.3	8.8	3.4 ± 0.6	4.4 ± 0.6	8.6
Si	13.0	24.8	16.4 ± 3.0	20.1 ± 3.3	27.7
Fe	2.1	8.5	3.9 ± 0.5	4.1 ± 1.0	6.0
Mn	0.044	0.288	0.114 ± 0.012	0.108 ± 0.036	1.677
Station C					
Al	0.8	7.4	2.4 ± 0.2	4.1 ± 0.9	9.7
Si	8.9	19.6	11.5 ± 0.8	15.5 ± 2.8	29.1
Fe	2.0	8.4	3.6 ± 0.6	3.7 ± 0.6	6.0
Mn	0.046	0.403	0.133 ± 0.017	0.101 ± 0.021	0.702

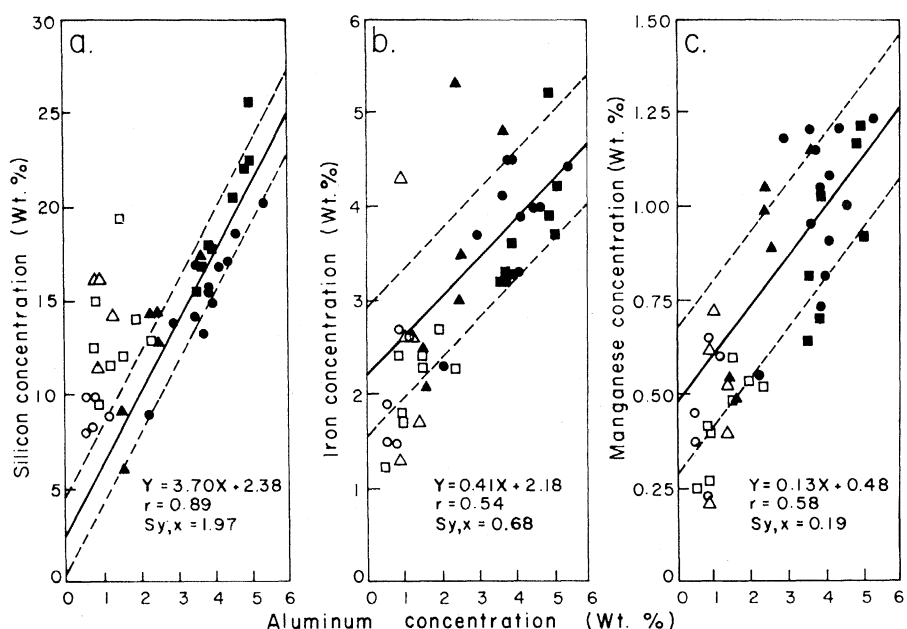


Fig. 2. Scatter diagrams of the (a) silicon, (b) iron, and (c) manganese concentrations against the aluminum concentration for the midwater (open symbols) and BNL (closed symbols) samples at stations A (Δ , $\blacktriangle$), B ($\square$, $\blacksquare$), and C ($\circ$, $\bullet$). The solid line represents the least-squares fit of the BNL data. The dashed lines indicate the standard error. Note that nearly all of the midwater silicon values exceed the standard error of the fit of the data, indicating the predominance of biogenic silica in this region of the water column.

5. E. T. Baker and D. Z. Piper, *Deep-Sea Res.* **23**, 181 (1976).
6. S. E. Calvert and N. B. Price, *Mar. Chem.* **5**, 43 (1977).
7. A *t*-test performed on the hypothesis that there is no difference between the calculated and observed means of the elemental percentages in the BNL resulted in acceptance at $P < .05$ for all samples except Al at stations B and C.
8. S. P. Hayes, *Proc. Offshore Tech. Conf. OTC-2774* (1977), p. 387.
9. A. F. Amos, O. A. Roels, A. Z. Paul, *Proc. Offshore Tech. Conf. OTC-2456* (1976), p. 341.
10. R. Young, *Mar. Geol.* **23**, M11 (1977).
11. P. Lonsdale and J. B. Southard, *ibid.* **17**, M51 (1974).
12. G. H. Keller, in *Civil Engineering in the Oceans* (American Society of Civil Engineers, Ann Arbor, Mich., 1968), p. 381.
13. E. Partheniades, *Proc. Am. Soc. Civ. Eng. J. Hydraul. Div.* **91**, 105 (1965).
14. We thank J. Nevins and G. Massoth for field and laboratory assistance. R. W. Sternberg, J. Cline, and J. Larrance critically read the manuscript. This study was supported by the NOAA Deep Ocean Mining Environmental Studies Program.

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Superconductivity of Americium

Abstract. *The metal americium becomes superconducting at temperatures as high as 0.79 K for the room temperature, double-hexagonal-close-packed phase. We also have evidence of a slightly higher transition temperature for the face-centered-cubic phase. This discovery of superconductivity in the midst of nonsuperconducting man-made elements is somewhat surprising.*

Americium, atomic number 95, is a member of the actinide series of elements in which all members above 92 are man-made. Our recent tests with single-phase, double-hexagonal-close-packed (dhcp) samples of Am all showed onsets of superconductivity between 0.79 K and 0.55 K. One sample with only the high-temperature face-centered-cubic (fcc) structure present showed an onset at 1.05 K. Samples containing mixtures of these two structures seemed to show the lowest transition temperatures, but experimental difficulties made the interpretation difficult.

Previous studies on americium metal (1) have shown that both a low-temperature ($\approx 700^\circ\text{C}$) dhcp form and a high-temperature fcc form exist. The fcc form can be retained at room temperature by quenching the metal from high temperatures. In 1971, Hill (2) noted that Am might become superconducting in view of its similarities to La, although it seemed unlikely. In 1975, Johansson and Rosengren (3) predicted a superconducting transition temperature of 2 to 4 K on the basis of their parameterization of trends in properties among the 4*f* and 5*f* electron elements. At approximately the same time, the resistivity of Am from room temperature down to low temperatures was being measured (4, 5). As a result, possibly, of the prediction by Johansson and Rosengren, the studies were continued to about 2 K to look for superconductivity. None was seen.

Recently, the availability of multi-milligram quantities of very pure ^{243}Am led to our tests for superconductivity to temperatures well below 2 K. The use of the 243 isotope, as opposed to the 241 species, reduced significantly the heat generated by the radioactivity (6.3 as compared to 115 mW/g), making the cooling problems less severe.

Our Am samples were prepared by reducing pure AmO_2 with Th metal, then distilling and condensing the more volatile Am metal on cooled quartz or tantalum receivers. We distilled most of the preparations a second time. The five different products were examined by spark-source mass spectrometry and, except for one of the products, the total impurities (excluding hydrogen, nitrogen, and oxygen) ranged from 50 to 240 ppm. Background corrections were not made, and thus these levels represent maximums.

The product exceeding these limits contained Yb at 2000 ppm as an additional impurity. The Yb was introduced through distillation equipment previously used for Yb even though the equipment had been acid-leached afterward. The presence of the Yb did not clearly affect the superconductivity of the Am.

We detected the superconductivity by measuring the a-c susceptibility of the samples in two different cryostats. The first was a ^3He system, capable of temperatures just below 0.4 K, in which we cooled the bare samples by submersion in liquid ^3He . Here we put the samples in one of two copper coils that were operated at their resonant frequencies (100 to 1000 Hz) while forming parts of two legs of an a-c bridge. We could move the samples, and their Bakelite basket, in and out of these coils during the measurements as a check against background effects.

The second cryostat was a ^3He - ^4He dilution refrigerator capable of temperatures from above 4 K to below 11 mK when operated without radioactive samples. In this system we placed the samples, and a bit of vacuum grease, in copper and beryllium-copper holders since better cooling is observed by additionally making electrical contact between the

samples and their colder surroundings than is seen by simple submersion in liquid helium. In the dilution refrigerator, loaded and empty dummy holders were placed in opposing secondary coils located within a large primary coil which applied an a-c magnetic field. We extracted the a-c susceptibility from these secondaries with a lock-in amplifier. In the dilution refrigerator we could not move the samples in and out of the coils without warming the system to room temperature. However, previous work with other superconducting samples of various sizes and shapes, as well as correlations with higher-temperature ^3He data give us confidence in the Am results. With radioactive samples, the need to confine contamination in the dilution refrigerator dictates that new holders must be used for each run. Occasionally magnetic behavior in the BeCu parts of the holders is seen and obscures the detection of superconductivity in the samples.

X-ray analysis of all samples before and after the low-temperature runs (6) showed that the sample structures were either single-phase dhcp, single-phase fcc, or mixed fcc and dhcp. The single-phase fcc structure (high-temperature form) was obtained because the thin depositions (5 to 10 μm thick) were collected on a cold receiver, which effected a quench.

It should be noted that radioactive superconductors often exhibit somewhat strange behaviors as a consequence of their self-heating coupled with the fact that materials have a drop in thermal conductivity associated with their superconducting transitions. The most obvious consequence of this is an increased temperature width of the superconducting transition. This arises because it is more and more difficult to cool the center of a sample as the interface with the superconducting outer region moves inward from the surface with decreasing temperatures. This increased width suggests, for superconducting radioactive materials, the assignment of T_c (transition temperature) to the intersection of the high-temperature base line with the extrapolation of the linear portion of the plot of susceptibility as a function of temperature. The conventional assignment of T_c as the temperature of the center of the transition would result in an unrealistically low T_c for Am. For some materials, full transitions can never even be seen.

A second, less obvious, phenomenon is oscillatory superconductivity. This was first reported by Olsen (7) but clearly occurs more generally than he dis-