

## DATA REPORT: GEOCHEMICAL ANALYSIS OF INDIAN OCEAN STANDARD SEDIMENT SAMPLE<sup>1</sup>

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### INTRODUCTION

Several laboratories which are involved in the geochemical analysis of sedimentary material recovered during Ocean Drilling Program Leg 121 have analyzed a "standard sediment sample" for interlaboratory comparison purposes. The results of these analyses are summarized in this report, along with a brief description of the analytical methods that were used.

### INDIAN OCEAN STANDARD SEDIMENT SAMPLE

The sample selected as an analytical standard is a composite of material from Sections 121-756C-1H-4 and 121-756C-1H-5. (This core was too intensely disturbed to be useful for stratigraphy studies.) The lithology of this sample consists of a nannofossil ooze with foraminifers and containing trace amounts of volcanic glass, and is typical of much of the material recovered during Ocean Drilling Program (ODP) Leg 121.

The material analyzed by each laboratory was initially prepared by freeze-drying approximately 2.5 kg of wet sediment from Core 121-756C-1H. The solid residue was then homogenized and split using a sample divider. Half of this material was further subdivided, and 10-g portions were distributed to each laboratory that participated in this study. The remaining material has been retained at The University of Michigan and portions are available upon request.

### PARTICIPATING LABORATORIES

Five different laboratories at four different institutions participated in the elemental analysis, carbonate analysis, and/or stable isotope analysis of the standard sediment sample. The principal investigators, their laboratory and institutional affiliations, and the letter code used for each laboratory in the tables of this report are as follows:

#### *Elemental Analyses*

Jan Smit, Institute of Earth Sciences, Free University, P.O. Box 7161, 1007MC Amsterdam, The Netherlands (Code: FUN).

Alan R. Huffman (Center for Tectonophysics) and Ted D. Oldham (Department of Geology), Texas A&M University, College Station, TX 77843 (Code: TAMU).

Robert M. Owen and Andrew R. B. Zimmerman, Marine Geochemistry Laboratory, Department of Geological Sciences, The University of Michigan, Ann Arbor, MI 48109 (Code: UM/MG).

#### *Carbonate Analyses*

John Farrell, Department of Geological Sciences, Brown University, Providence, RI 02912 (Code: BU).

Robert M. Owen and Andrew R. B. Zimmerman (see above).

#### *Stable Isotope Analyses*

John Farrell (see above).

Kyger C Lohmann, Stable Isotope Laboratory, Department of Geological Sciences, The University of Michigan, Ann Arbor, MI 48109 (Code: UM/SI).

### ANALYTICAL METHODS

Each of the three laboratories that performed an elemental analysis of the standard sample used an instrumental neutron activation analysis (INAA) method. At TAMU this procedure involved irradiating 70-mg samples (sealed in polyethylene vials) for 30 s at a thermal neutron flux of  $10^{13} \text{ cm}^{-2}\text{s}^{-1}$ , and counting for 30 s after a 15-min delay. Multi-element standards used in this analysis were USGS G-2 granite and NBS 1633A Coal Flyash. Samples and standards were irradiated a second time for 14 hr, and counted after a 7-day delay (1000-s count) and a 21-day delay (4000-s count) for intermediate- and long-lived isotopes, respectively. Estimated analytical uncertainties relative to known standards are less than 10% for Na, Ca, Al, La, Lu, Sm, Yb, Ce, Co, Cr, Cs, Eu, Fe, Nd, Sc, Tb, and Zn; about 10% for Dy, Sb, Ba, and Sr; and 10%–20% for Se. Other elements measured were below the detection limit for the method employed (see Huffman et al., 1990, for details). Information concerning the procedural details, detection limits, analytical standards, and estimated analytical error for the INAA methods used at FUN and UM/MG are given in Smit and ten Kate (1982) and Ruhlin and Owen (1986a, 1986b), respectively. The relative standard deviations for replicate analyses at UM/MG were between 1%–15% for all elements except V, Dy, Ce, Eu, Ni, Tb, and Th, which were between 15%–30%, and for Al, Nd, Cr, and Hg, which were between 30%–45%. Calcium carbonate analyses at both BU and UM/MG are done using a vacuum gasometric technique (e.g., Jones and Kaiteris, 1983). Details of the laboratory and analytical procedures used for the determination of stable isotopes ( $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$ ) at BU and UM/SI are reported in Farrell and Janecek (this volume) and Carpenter and Lohmann (1989), respectively.

### RESULTS AND DISCUSSION

The results of the elemental, carbonate, and stable isotope analyses of their standard sediment sample are presented in Tables 1–3, respectively. Each laboratory conducted these analyses according to its own procedures, and we note that subtle procedural differences commonly exist even when the same general technique is used to measure the same parameter. The values shown for each parameter in the tables are generally the mean values of several replicate analyses, especially for the elemental analyses (see following discussion). For these reasons, as well as the fact that there is no presumed "recommended value" for any of the measured parameters, we have deliberately avoided calculating a grand mean value in those cases where more than one laboratory has reported a result for the same parameter.

The elemental analysis results from FUN represent one subsample (Table 1, column A) of the standard sample, which was rinsed for 3 days in distilled water to remove sea salt, and a second subsample (Table 1, column B), which was untreated. The differ-

<sup>1</sup>Peirce, J., Weissel, J., Taylor, E., Alt, J., et al., 1991. *Proc. ODP, Sci. Results*, 121: College Station, TX (Ocean Drilling Program).

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**Table 1. Elemental analysis of Indian Ocean standard sediment sample.**

	FUN		TAMU		UM/MG
	A	B	A	B	
Al	N	N	923	1050	1240
As	0.54	0.63	D	D	D
Ba	212	212	205	222	238
Br	0.83	29.4	D	D	26.6
Ca (%)	38.5	36.3	38.44	38.63	N
Ce	3.1	3.5	3.58	3.63	4.02
Co	2.96	2.70	3.20	3.15	2.50
Cr	1.50	1.24	1.84	1.89	3.91
Cs	D	D	0.12	D	N
Dy	N	N	1.50	1.03	1.40
Eu	0.208	0.203	0.248	0.262	0.217
Fe	910	870	950	1012	945
Hf	0.07	0.031	N	N	0.165
Hg	0.54	1.33	N	N	1.77
La	4.74	4.87	4.76	5.03	5.18
Lu	0.116	0.113	0.098	0.106	0.115
Mn	N	N	199	199	195
Mg	N	N	N	N	2880
Na	783	5000	5558	5471	4900
Nd	4.7	4.8	2.76	3.24	9.54
Ni	D	D	D	D	15.2
Rb	1.7	D	D	D	D
Sb	0.072	0.069	0.107	D	0.300
Sc	0.536	0.506	0.61	0.64	0.514
Se	N	N	0.43	0.58	D
Sm	0.883	0.887	0.81	0.82	0.785
Sr	1150	1140	1480	1470	1680
Tb	0.169	0.149	0.182	0.186	0.226
Th	0.232	0.240	0.25	0.27	0.346
U	0.23	0.26	D	D	D
V	N	N	3.0	3.5	6.07
Yb	0.743	0.731	0.721	0.731	0.845
Zn	65	45	51.0	56.2	40.7

Notes: All results except Ca are in units of  $\mu\text{g/g}$ . D = below detection limit; N = not analyzed.

**Table 2.  $\text{CaCO}_3$  analysis of standard sediment sample.**

	BU	UM/MG	TAMU <sup>a</sup>
% $\text{CaCO}_3$ (mean)	93.34	96.59	96.23
Standard deviation	1.47	1.83	—
Number of analyses	9	8	20

<sup>a</sup> TAMU results estimated from INAA for Ca, assuming sample = 100%  $\text{CaCO}_3$ .

ences in the FUN results for Na, Br, and Hg are attributed to the effect of the rinsing procedure, whereas the differences for Hf content are probably due to relatively high analytical error (J. Smit, pers. comm., 1990).

The TAMU elemental analysis (except for Sr; see below) was done in two separate batches (Table 1, columns A and B), each of which contained 10 subsamples of the standard sample. In some cases the subsamples in either batch showed values that were below the detection limit. Consequently, the reported TAMU results are the mean values of two replicate analyses for Cs, Se, and V; three to nine replicates for Cr, Dy, and Sb; and 10 replicates for the remaining elements. The Sr data from TAMU were reported separately, and represent the mean values of two different batches of four samples each, using single standardization with NBS flyash 1633A (A. Huffman, pers. comm., 1990).

The UM/MG elemental analysis was done in 11 different batches, each of which contained between one and five subsamples of the standard sample. As was the case at TAMU, some subsamples in different batches showed values below the detection limits for certain elements. The reported UM/MG results are the mean values of two replicates for Ni; three to eight replicates for Al, Cr, Hf, Sb, and V; and 12–24 replicates for the remaining elements.

Except for Cr and Sb, the range of values for those elements measured by all three laboratories appears to be within the variance commonly encountered in the analysis of these elements. The concentrations of Cr and Sb in the standard sample apparently are very close to the detection limits for INAA, as indicated by the difficulty of obtaining measurable values at those laboratories (TAMU and UM/MG) which did replicate analyses.

The results of the  $\text{CaCO}_3$  analyses are summarized in Table 2. Our experience with replicate analyses using the gasometric technique (used at BU and UM/MG) is that an analytical precision of 1%–2% is usually obtained at a single laboratory, and interlaboratory comparisons usually agree to within 3%–5%. The results from BU and UM/MG fall within these typical ranges. For comparison purposes, we have also reported a  $\text{CaCO}_3$  result from TAMU, which was calculated from their INAA data for Ca and assumes the sample was 100%  $\text{CaCO}_3$ . This estimate is in good agreement with the results of the gasometric analyses. Farrell and Janecek (this volume) and Howard (in press) analyzed aliquots of shipboard samples for wt%  $\text{CaCO}_3$  at BU and determined that their results were similar ( $\pm 1.0\%$ ) to those obtained in the shipboard laboratory.

The results of the stable isotope analyses for  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  from two different laboratories are summarized in Table 3. Among the 18 samples analyzed at UM/SI, six were both roasted (at  $\sim 370^\circ\text{C}$ ) and ground to a fine-grained powder prior to the analysis, while the remainder were analyzed "as is." No significant difference was observed between the results obtained for the samples that underwent roasting/powdering pretreatment and those that did not. The methods of stable isotope analysis at BU

**Table 3. Stable isotope analyses of the ODP standard sediment sample.**

Standard	BU		UM/SI <sup>a</sup>	
	$\delta^{13}\text{C}$ (‰ to PDB)	$\delta^{18}\text{O}$ (‰ to PDB)	$\delta^{13}\text{C}$ (‰ to PDB)	$\delta^{18}\text{O}$ (‰ to PDB)
NBS-18	-4.93	-22.95	-4.86	-23.00
NBS-19	1.91	-2.10	1.86	-2.22
NBS-20	-1.00	-4.18	-1.07	-4.11
ODP mean	0.86	1.91	0.74	1.87
Standard deviation (n =)	0.12 (27)	0.17 (27)	0.17 (13)	0.20 (13)

<sup>a</sup>  $\delta^{13}\text{C}_{\text{norm}} = \delta^{13}\text{C}_{\text{meas}} (1.000) + 0.030$ ;  $\delta^{18}\text{O}_{\text{norm}} = \delta^{18}\text{O}_{\text{meas}} (1.054) + 0.312$ .

are described in Farrell and Janecek (this volume). Both laboratories routinely intermix laboratory standards (BP-4 at BU; NBS-20 at UM/SI) with sample analyses to monitor daily variations in instrumental parameters. The relatively high standard deviations reported by each laboratory for each isotope are likely due to small heterogeneities within the standard sediment sample (K. C. Lohmann, pers. comm., 1990). To facilitate direct comparison of isotopic data included in this volume, normalization equations have been developed following an interlaboratory cross-calibration based on NBS powered standards (Table 3).

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