

17. INTERLABORATORY COMPARISON OF LEG 92 STANDARD SEDIMENT SAMPLE ANALYSES¹

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INTRODUCTION

Several laboratories are involved in performing geochemical analyses of sedimentary material recovered during Leg 92. During the cruise, members of the shipboard scientific party agreed that each of these laboratories should be provided with a "standard sediment" sample to be used in an interlaboratory comparison study.

METHODS OF COMPARISON

The sample selected as an analytical standard is a clay-bearing nannofossil ooze that is typical of much of the sediment recovered during the cruise. The sample was obtained from a 6.2-m experimental core recovered at Site 602. This core was too intensely disturbed to be useful for sediment lithology or stratigraphy studies (see Site 602 Chapter, this volume) so portions of it were homogenized for use as an analytical standard.

The standard sediment sample was prepared as follows. Approximately 2.5 kg of wet sediment from Core 602 were freeze dried; the solid residue was then homogenized and split using a sample divider. Half of this material was further subdivided, and 10-g portions were sent to each laboratory that had expressed an intention to conduct sediment geochemistry analyses on Leg 92 sediments. The remaining material has been retained at the University of Michigan in Ann Arbor; portions of this are available on request.

Five laboratories returned results, which are summarized in Tables 1 and 2. The principal investigator, laboratory affiliation, and letter code used for each laboratory in the tables are as follows: V. Marchig, Federal Institute for Geosciences and Natural Resources, Hannover, Federal Republic of Germany (BGR); J. Erzinger, Mineralogy-Petrology Institute, Justus-Liebig University, Gießen, Federal Republic of Germany (JLU); M. Lyle, College of Oceanography, Oregon State University (OSU); M. Leinen, Graduate School of Oceanography, University of Rhode Island (URI); R. Owen, Department of Atmospheric and Oceanic Science, The University of Michigan (UM).

Whenever two or more laboratories reported a result for the same parameter, we calculated a simple arithmetic mean value and reported it in Tables 1 and 2. These

Table 1. Major element composition of standard sediment sample (wt. %).

	BRG	JLU	OSU	UM	URI	Mean
Si	0.80	—	0.792	—	0.452	0.68
Al	0.26	—	0.243	0.108	0.169	0.195
Ti	0.02	—	0.018	—	—	0.02
Fe	6.16	—	6.10	6.25	5.97	6.12
Ca	31.80	—	32.4	—	35.75	33.3
Mg	0.16	—	0.356	—	0.328	0.28
Mn	1.25	—	1.23	1.05	1.20	1.18
Sr	0.12	—	0.140	0.192	—	0.15
K	0.02	—	0.081	—	—	0.05
N _a	—	—	0.841	0.873	—	0.857
Cl	—	—	—	0.132	—	—
P ₂ O ₅	0.36	—	—	—	—	—
SO ₃	—	0.212	—	—	—	—
CaCO ₃	—	—	—	—	80.2	—
Total salt	—	—	—	—	2.345	—
Quartz	—	—	—	—	0.24	—
Opal	—	—	—	—	5.38	—
LOI	41.11	—	—	—	—	—

Note: — indicates not determined or below detection limit. LOI = loss on ignition.

mean values are reported only for general information. Deviations from the mean do not represent deviations from "correct" values, because the calculated mean is based on only two to four values and because different laboratories often used different methods to determine the same parameter (see following comments). An interlaboratory comparison for the major elements (Table 1) reveals a surprisingly large range of values for some elements (e.g., Si, Al, Ca, Mg, and K), although each individual laboratory did obtain good agreement for two to seven replicate analyses for each element. This suggests that there are significant differences among the various analytical and/or sample preparation methods that were employed. Despite the different analytical methods that were used, however, the agreement among different laboratories for the minor elements (Table 2) appears to be well within the variance commonly encountered in the analysis of these elements.

COMMENTS ON ANALYTICAL METHODS

BRG results were obtained by X-ray fluorescence (XRF). Values shown are the means of two to three determinations.

JLU results for B and the REEs were obtained by inductively coupled plasma (ICP) optical emission spectroscopy. Sulfur was determined using a LECO "SC 132" analyzer. Values shown are the means of two to three determinations.

¹ Leinen, M., Rea, D. K., et al., *Init. Repts. DSDP, 92*: Washington (U.S. Govt. Printing Office).

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Table 2. Minor element composition of standard sediment sample (ppm).

	BRG	JLU	OSU	UM	URI	Mean
Rb	6	—	—	—	—	—
Ba	378	—	400	306	390	368
Li	—	—	1.2	—	—	—
Y	44	—	—	—	—	—
Zr	53	—	—	—	—	—
Co	32	—	18	20.7	9.45	20
Ni	92	—	101	—	112	102
Zn	—	—	143	—	142	142
Cu	230	—	—	—	248	239
Cr	—	—	—	2.30	—	—
V	212	—	220	180	—	204
As	47	—	—	50.8	—	49
Sb	—	—	—	2.31	—	—
B	—	49	—	—	—	—
Hf	—	—	—	0.30	—	—
U	—	—	—	1.26	—	—
Br	—	—	—	44.8	—	—
Sc	—	—	—	1.89	—	—
La	—	23.6	—	27.8	—	25.7
Ce	—	9.8	—	8.84	—	9.32
Nd	—	19.3	—	15.7	—	17.5
Sm	—	5.67	—	4.86	—	5.26
Eu	—	1.36	—	1.34	—	1.35
Tb	—	0.64	—	0.71	—	0.68
Yb	—	3.04	—	2.42	—	2.73
Lu	—	0.44	—	0.48	—	0.46

Note: — indicates not determined or below detection limit.

OSU results were obtained by flame atomic absorption spectroscopy (AAS). Values shown are the means of six determinations.

UM results were obtained by instrumental neutron activation analysis (INAA). Details concerning irradiation and counting times are given in Ruhlin and Owen (this volume). Values shown are the means of four to seven determinations.

URI results for Al, Si, Ca, Ba, Mn, Fe, Cu, and Zn were obtained by flame AAS; for Co and Ni by flameless AAS. Quartz and opal were determined using the X-ray diffraction (XRD) technique described by Ellis and Moore (1973). Total salt was determined by automated Ag titration of Cl in the salt leached from 1 g of sample using 5 ml of deionized distilled water. Carbonate was determined by a pressure-bomb technique. Values shown are the means of two determinations.

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REFERENCE

Ellis, D. B., and T. C. Moore, Jr., 1973. Calcium carbonate, opal and quartz in Holocene pelagic sediments and the calcite compensation level in the South Atlantic Ocean. *J. Mar. Res.*, 31:210-227.

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