

32. DATA REPORT: CHEMICAL ANALYSES OF THE LEG 140 REFERENCE SAMPLE¹

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INTRODUCTION

To facilitate comparisons among various Leg 140, Hole 504B geochemical data sets, an inter-laboratory geochemical reference sample was prepared from approximately 1 kg of moderately phyrlic, plagioclase-clinopyroxene-olivine diabase. This sample was taken from a single "junk basket" piece (Sample 140-504B-182M-3, 7–20 cm, referred to here as BAS-140) collected from the sheeted dike section of Hole 504B (1°13.611'N, 83°43.818'W), at an approximate depth of 1622 mbsf.

SAMPLE PREPARATION

Aboard the *JOIDES Resolution*, the recovered sample was sawn and then broken into smaller pieces with a water-cooled diamond saw and steel hammer. Excessively altered material, hammer and saw marks were removed by wet-grinding on a diamond abrasive wheel. The sample was then sonicated in distilled water and methanol for 10 min, and dried at 110°C for more than 2 hrs. Rock chips larger than 1 cm in diameter were placed between two disks of Delrin plastic and crushed in a hydraulic press so they could fit into the grinding vessel. The total sample was then divided into 50-g to 80-g batches and ground for about 1.5 min per batch (<30 µm grain size) in a tungsten carbide (WC) shatterbox. Accumulated batches of sample powder were homogenized in a 1-gal plastic bag for about 1 hr and then distributed in roughly 30-g aliquants to interested members of the shipboard party.

Grinding samples in a WC container introduces significant amounts of W, Co, Ta, and to a lesser extent Nb, and possibly Zn (Thompson and Bankston, 1970; Joron et al., 1980; Hickson and Juras, 1986; Frey et al., 1991). Of these elements, only Nb, Co, and Zn are reported here. The amount of Nb contamination appears to be much less than 1 ppm (Sparks, this volume), and there is no significant indication that this kind of contamination has affected sample homogeneity.

RESULTS

Tables 1 and 2 list the individual results from the eight laboratories that contributed to this report. It is not our intention to present any of these results as either "preferred" or "recommended" values. These values are for comparison purposes only. The various laboratories and associated Leg 140 scientists are: GeoForschungsZentrum Potsdam (GFZ) — E. Zuleger; Georgia State University (GSU) — D.A. Vanko; Shipboard Scientific Party (LEG140); Justus-Liebig-Universität (JLU) — E. Zuleger; University of Michigan (MICH) — J.C. Alt; Université Paris (PAR) — P. Agrinier; Stanford University (STAN) — J.W. Sparks; University of Tasmania (TAS) — A.W. McNeill. The analytical techniques employed at each laboratory are given in the

notes below each table. Estimated values of analytical precision are listed along with the individual results.

Major element analyses (SiO₂ through P₂O₅, Table 1) were corrected for loss on ignition (LOI), and reported on an anhydrous basis. The left-most column of numbers represents the unweighted mean concentration and standard deviation of multiple analyses. Where there is only one analysis for a given element, that analysis is given as the mean. The standard deviations of the mean for oxides SiO₂ through P₂O₅ are all similar to, or less than, the values for analytical error. This remarkably strong agreement demonstrates there are no significant inter-laboratory calibration biases, or major element heterogeneities. The similarity of analytical techniques and wide use of commonly reported international geostandards no doubt contributed to this agreement. Each major element analysis was determined by X-ray fluorescence (XRF) on Li-borate glass discs.

Trace element concentrations (Table 2), although in many cases very precise, exhibit a wider range of values resulting from inter-laboratory calibration biases, differing analytical methods, and extremely low concentrations of highly incompatible elements such as Nb and Rb. Overall, there is good agreement between analyses of Ni, Cu, Ga, Sr, and Y; moderate agreement between Zr analyses; and moderate to poor agreement for many of the REE as well as S, V, Cr, and Zn values. Sample homogeneity on the trace element scale is not a likely factor in many of the discrepancies. The error values reported by LEG140 represent the standard deviation of the mean (1 σ) for six separate splits of BAS-140 (Shipboard Scientific Party, 1992). For each element, this variation is about the same as the estimated analytical precision, further demonstrating the homogeneity of the sample (we are assuming that these particular elements serve as appropriate indicators to the distribution of REE; however, they may not apply to S).

It is beyond the scope of this report to judge the accuracy of these analyses. We leave it up to the reader to make further comparisons and inquiries, as needed.

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* Abbreviations for names of organizations and publications in ODP reference lists follow the style given in *Chemical Abstracts Service Source Index* (published by American Chemical Society).

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Table 1. Major and minor oxide analyses of Leg 140 reference sample BAS-140.

	Mean \pm 1 σ	Laboratories ^a					PAR
		GSU	LEG140	JLU	STAN	TAS	
SiO ₂ (wt%)	50.5 \pm 0.1	50.3 \pm 0.2	50.6 \pm 0.1	50.6 \pm 0.2	50.6 \pm 0.2	50.4 \pm 0.1	
TiO ₂	0.98 \pm 0.01	0.98 \pm 0.01	0.97 \pm 0.01	1.00 \pm 0.01	0.99 \pm 0.01	0.97 \pm 0.01	
Al ₂ O ₃	14.6 \pm 0.03	14.53 \pm 0.05	14.58 \pm 0.05	14.6 \pm 0.1	14.58 \pm 0.09	14.57 \pm 0.03	
Fe ₂ O ₃ * ^b	11.1 \pm 0.10	11.09 \pm 0.05	11.15 \pm 0.07	11.3 \pm 0.1	11.08 \pm 0.05	11.04 \pm 0.06	
MnO	0.19 \pm 0.005	0.18 \pm 0.005	0.18 \pm 0.002	0.19 \pm 0.005	0.19 \pm 0.003	0.19 \pm 0.005	
MgO	8.15 \pm 0.09	8.22 \pm 0.04	8.01 \pm 0.05	8.22 \pm 0.05	8.14 \pm 0.08	8.19 \pm 0.02	
CaO	12.4 \pm 0.10	12.28 \pm 0.02	12.51 \pm 0.03	12.5 \pm 0.0	12.41 \pm 0.04	12.32 \pm 0.01	
Na ₂ O	1.84 \pm 0.04	1.86 \pm 0.04	1.85 \pm 0.05	1.77 \pm 0.02	1.85 \pm 0.03	1.87 \pm 0.05	
K ₂ O	0.01 \pm 0.004	0.01 \pm 0.005	0.004 \pm 0.003	<LLD ^c		<LLD	
P ₂ O ₅	0.08 \pm 0.01	0.08 \pm 0.005	0.06 \pm 0.01	0.08 \pm 0.004	0.07 \pm 0.002	0.09 \pm 0.01	
Total (anhydrous)	99.9	99.6	99.9	100.3	99.9	99.7	
Fe ₂ O ₃	2.72 \pm 0.12			2.72 \pm 0.12			
FeO	7.72 \pm 0.06			7.72 \pm 0.06			
LOI ^d	0.59 \pm 0.13				0.50	0.68	
H ₂ O*	1.12 \pm 0.14		1.25 \pm 0.06	1.15			0.97 \pm 0.08
CO ₂	0.05 \pm 0.03		0.03 \pm 0.01	0.07			

Note: Blank spaces = not determined.

^a Laboratories, associated techniques and references (as given by analysts) = GSU: XRF — major elements (LaTour, 1989); JLU: XRF — major elements, Karl Fisher titration — H₂O*, infrared spectrophotometry — CO₂ (Zuleger et al., this volume); LEG140: XRF — major elements, gas chromatography (GC) — H₂O* and CO₂ (Shipboard Scientific Party, 1992); PAR: manometric determination following sample fusion — H₂O* (Coleman et al., 1982); STAN: XRF — major elements (Sparks, this volume); TAS: XRF — major elements (Norrish and Chappell, 1977).

^b Fe₂O₃* = Total iron reported as Fe₂O₃.

^c <LLD = Less than the lower limit of detection.

^d LOI = Loss on ignition.

Table 2. Trace element and isotope analyses of Leg 140 reference sample BAS-140.

	Laboratories*							
	LEG140	JLU (XRF)	JLU (ICPES)	STAN	GFZ	TAS	PAR	MICH
Li (ppm)					1.2 ± 0.1			
S		954 ± 7		887 ± 4				860 ± 50
Cl				87 ± 4				
Sc					43 ± 3	40.7		
V	331 ± 5			342 ± 2				
Cr	175 ± 1	194 ± 2		192 ± 2	186 ± 10	185		
Co					55 ± 4			
Ni	84 ± 0.5	81 ± 2		85 ± 1	85 ± 3			
Cu	83 ± 0.5	83 ± 2		84 ± 1	81 ± 2			
Zn	76 ± 0.4	78 ± 1		85 ± 1	80 ± 2			
Ga		16 ± 1		15.6 ± 0.4	15.5 ± 0.3			
Rb	<0.4			<0.5	0.06 ± 0.005			
Sr	44.5 ± 0.3	47 ± 1		45.5 ± 0.4	44 ± 1			
Y	25.9 ± 0.2	27 ± 1	24.5 ± 0.7	26.6 ± 0.2	24 ± 1			
Zr	45.4 ± 0.5	49 ± 0.5		44.8 ± 0.3				
Nb	0.5 ± 0.1	<5		<0.3	0.5 ± 0.1			
La			1.0 ± 0.1		0.92 ± 0.02	2.04		
Ce			4.8 ± 0.4		3.73 ± 0.13	3.85		
Pr					0.77 ± 0.04			
Nd			4.5 ± 0.1		4.9 ± 0.1	5.45		
Sm			2.0 ± 0.1		2.08 ± 0.05	2.07		
Eu			0.61 ± 0.01		0.78 ± 0.03	0.7		
Gd			3.0 ± 0.1		3.2 ± 1			
Tb					0.63 ± 0.01	0.56		
Dy			3.5 ± 0.1		4.5 ± 0.1			
Ho			0.87 ± 0.03		0.97 ± 0.03	0.94		
Er			2.6 ± 0.0		2.9 ± 0.1			
Tm					0.44 ± 0.02			
Yb			2.5 ± 0.0		3.0 ± 0.1	2.88		
Lu			0.40 ± 0.01		0.42 ± 0.02	0.46		
Hf					1.1 ± 0.2	1.39		
Bi					0.02 ± 0.001			
Th					0.025 ± 0.006			
U					0.008 ± 0.002			
δ ¹⁸ O (‰)						4.34 ± 0.1		
δD						-44 ± 2		

* Laboratories, associated techniques and references (as given by analysts) = GFZ: inductively coupled plasma mass spectrometry (Zuleger and Erzinger, this volume); LEG140: XRF (Shipboard Scientific Party, 1992); STAN: XRF (Sparks, this volume); JLU: XRF — Zr, Y, Sr, Zn, Cu, Ni, and Cr, Leco Analyzer — S, inductively coupled plasma emission spectrometry — rare earth elements and Y (Zuleger and Erzinger, this volume); MICH: colorimetric titration — S as sulfide (Alt et al., 1989); TAS: instrumental neutron activation analysis (Becquerel Laboratories, Sydney, Australia); PAR: mass spectrometry (Coleman et al., 1982).