

Analytical Methods for Whole-Rock Geochemistry

Analytical methodology for USGS analysis of augen gneiss whole-rock samples

Large samples of augen gneiss were analyzed to minimize bias resulting from the coarse-grained or porphyritic texture of the rocks. Most rock powders analyzed were obtained by crushing ~45 kg of material; 5 samples (4016A, 4016F, 4020A, 4027, and 4030A samples were powders from 11 kg samples.

All elemental abundances in weight percent were determined by wavelength-dispersive X-ray fluorescence spectrometry (A. Bartel, J. Carr, V. McDaniel, K. Stewart, and J. Taggart, analysts; USGS, Denver, Colorado) (Taggart and others (1987). All elemental abundances in parts per million (ppm) were determined by instrumental neutron activation analysis (J. Mee, and I. Schwartz, analysts, USGS Reston, Virginia) (Baedecker and McKown, 1987), except for niobium that was determined by spectrophotometric analysis (E. Campbell and M. Doughton, analysts; USGS, Denver Colorado) (Greenland and Campbell, 1974).

All Fe reported as Fe_2O_3 for rocks analyzed by X-ray fluorescence. SiO_2 content calculated volatile-free with analysis normalized to total 100.00%. Values for REE not analyzed (Pr and Er) were determined by interpolation from REE plot.

Analytical methodology for USGS and Chemex Labs analysis of amphibolite whole-rock samples

Fresh samples of ~0.5 kg were selected for major and trace element analysis. Different aliquots from the same sample were sent to the different labs used. Analyses were performed at the U.S. Geological Survey (USGS) except for those of samples 96ADb52, 96ADb33A, and 96ADb35A that were analyzed at Chemex Labs, Vancouver, Canada. USGS-analyzed samples were ground in an alumina shatterbox. Major elements were determined by wavelength-dispersive X-ray fluorescence spectrometry (XRF) on fused glass disks in Lakewood, Colorado (J. Baker, A.J. Bartel, V. McDaniel, K. Stewart, J. Taggart, and J.S. Wahlberg analysts) (Taggart and others (1987). Ba, Rb, Sr, Y, and Zr were determined by energy-dispersive X-ray fluorescence spectrometry XRF in Menlo Park, California (P.E. Bruggman analyst) (Johnson and King, 1987). Emission spectroscopy was used for Cu, Ni, V (Menlo Park; T. Fries, P. Lamothe, and R.W. Lerner, analysts) (Golightly and others, 1987s). For all samples except 91ADb15, 91ADb 12, and 91ADb 14A, Nb was determined by spectrophotometric in Reston, Virginia, (E. Campbell, M. Doughten, and B. Libby analysts) (Greenland and Campbell, 1974). Niobium for samples 91ADb15, 91ADb12, and 91ADb14A was analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) following quantitative chemical separation (Menlo Park, S.J. Pribble analyst) (Lichte and others, 1987). The remaining elements (Co, Cr, Cs, Hf, Ta, Th, U, Zn, Sc, La, Ce, Nd, Sm, Eu, Tb, Yb, and Lu) were analyzed by instrumental neutron-activation analysis (INAA) in Reston, Virginia (J. Grossman, J.S. Mee, L.J. Schwarz, and G.A. Wandless analysts) (Baedecker and McKown, 1987). Analytical precision and accuracy of the spectrophotometric analysis of Nb have been estimated from replicate analyses of eight USGS standard rocks to be 2.9-6.4 % of the amount present (Greenland and Campbell, 1974); precision and accuracy for ICP-AES Nb analysis is comparable. Analytical uncertainties for the other methods, based on replicate analyses of standard rocks (Bacon and Driutt 1988), are reported in Table 1 of Dusel-Bacon and Cooper, 1999. Details about analytical procedures used at USGS are given in Taggart and others (1987).

Major elements (and Cr_2O_3) for the three samples (96ADb52, 96ADb33A, and 96ADb35A) analyzed by Chemex Labs, Vancouver, Canada were determined by wavelength-dispersive X-ray fluorescence spectrometry (XRF) analysis of pellets made by meta-borate fusion of powder ground in a tungsten carbide ring mill; lower detection limits are 0.01 %. Trace elements for these samples were determined by inductively coupled plasma mass spectrometry (ICP-MS) analysis of powders ground in a chrome steel ring mill that were subjected to meta-borate fusion followed by dissolution. Ni was determined by atomic absorption following a Nitric aqua regia leach.

Analytical methodology for University of Alaska, Fairbanks, XRF trace-element analysis of augen gneiss whole-rock samples

Approximately 10 gram aliquots of previously pulverized and homogenized sample material was made into 40 cm pressed pellets, using 1 drop of 5% polyvinyl alcohol solution per 2 grams of sample, and compressed to 10 tons for 2 minutes. [These same procedures were employed to create pressed pellets from 25 certified international rock standards.]

Once dried, samples were analyzed with a PanAlytical Axios X-ray Fluorescence Spectrometer using the conditions described below.

Elem	Line	X-tal	Collimator	Detector	kV	mA	Angle	Offset Bg1	Offset Bg2	PHD1	PHD1	count time	
							(°2 θ)	(°2 θ)	(°2 θ)	LL	UL	peak	bkg (2)
Rb	KA	LiF 220	150 μ m	Scint.	60	66	37.9	-1.345	3.1944	35	66	120	60 ea
Sr	KA	LiF 220	150 μ m	Scint.	60	66	35.8	-0.6952	0.7206	36	63	120	60 ea
Zr	KA	LiF 220	150 μ m	Scint.	60	66	32	-0.4492	0.768	38	61	120	60 ea
Y	KA	LiF 220	150 μ m	Scint.	60	66	33.8	-1.036	1.2756	37	63	120	60 ea
Nb	KA	LiF 220	150 μ m	Scint.	60	66	30.4	-0.5646	1.185	38	61	120	60 ea
Rh	KA-C	LiF 220	150 μ m	Scint.	60	66	26.2	-4.8694	3.6526	35	66	120	60 ea

Matrix corrections were made on standards and unknowns using the Rh Compton peak intensity. Kb interference on Ka intensity (Rb on Y, Sr on Zr, Y on Nb) were corrected by measuring Kb intensity at the respective Ka line (e.g. Rb at Y Ka) for standards and averaging the ratio of Kb:Ka intensity.

Twenty-five certified international standards (BE-N, BHVO-1, BIR-1, BR, DNC-1, DR-N, GS-N, GSP-1, JB-2, JF-1, JG-2, JP-1, MA-N, MRG-1, NBS 688, NIM-L, NIM-N, NIM-S, PCC-1, QLO-1, RGM-1, STM-1, SY-2, SY-3, and W-2) were employed in creating calibration curves.

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