

Polarized Fe XANES Pre-Edge Spectroscopy of Feldspars

M.D. Dyar (Mount Holyoke College), J.S. Delaney (Rutgers U.), G.R. Rossman (Caltech) and S.R. Sutton (U. Chicago)

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Beamline(s): X26A

Introduction: Although feldspar is the most commonly-occurring mineral in the Earth's crust, its use as a petrogenetic indicator has not been greatly exploited until recently, as evolving analytical techniques have refined characterizations of the amount, valence state, and site occupancy of Fe in the feldspar structure. Understanding of Fe valence and site occupancy in feldspar is of particular importance in igneous petrology because it can be used to quantify redox conditions, and therefore oxygen fugacity, during crystallization. Development of methodology for analysis of felsic magma constituents has the potential to greatly expand our knowledge of how such systems evolve during crystallization. In this study, we describe a calibration line to be used in evaluating $\text{Fe}^{3+}/\Sigma\text{Fe}$ in feldspars for which this is unknown and evaluate the effects of crystal orientation on the feldspar XANES spectra.

Methods and Materials: Samples used for the calibration line were analyzed by Hofmeister and Rossman (1984), while the samples used for polarization studies came from studies of the dielectric constant in feldspar (Shannon et al., 1992a, b). For a pure Fe^{2+} endmember of the calibration line, we used anorthite from the Serra de Mage meteorite (AMNH 3782-6). Samples with intermediate $\text{Fe}^{3+}/\Sigma\text{Fe}$ contents came from Lake County, Oregon (GRR #13761) and Halloran Springs, California (GRR #13759). For the Fe^{3+} endmember, we used yellow orthoclase from Itrongay, Madagascar (GRR #13762), the same locality studied by earlier workers.

For the polarization studies, two different samples with contrasting compositions were chosen: a clear, pale straw colored anorthite from Great Sitka Island, Aleutian Islands, AK (USNM 137041; GRR 56163-6) and a colorless orthoclase crystal from Madagascar (GRR 62047-48). These samples were oriented for cutting by polarized light and by back-reflection Laue photographs. Rectangular slabs were then cut perpendicular to the reciprocal axes a^* , b^* , and c^* with a low-speed diamond saw. Finally, these slabs were doubly polished (Shannon et al., 1992a,b). Because the orientation of these slabs relative to the coplanar crystallographic axes was not known, it was necessary to take XANES spectra every 30° over a 180° range by rotating the crystals between spectra. Measurements were made at NSLS beamline X26A using a roughly $20\ \mu\text{m}$ diameter beam.

Results: The three feldspars standards were run at seven consecutive beam sessions. A typical calibration line is shown in Figure 1. This calibration was then used to analyze the Fe^{3+} contents of feldspars with unknown Fe^{3+} contents in rocks from the Skaergaard Complex, East Greenland; the Atascosa Lookout Lava Flow, Arizona; Martian basalts from meteorites; lunar mare basalts; eucrites; Olduvai Gorge, Tanzania; and Adirondack anorthosites from NY.

Polarization studies of feldspars are ongoing. In Figure 2, we show a series of spectra taken from Slab A at 30° angles. Changes in peak positions and intensities are obvious, and are currently being investigated. However, all of these spectra yield similar Fe^{3+} contents, ranging from 67.9% at 0° to 88.1% at 60° . These differences probably represent the major source of the $\pm 10\%$ error observed using this method to determine Fe^{3+} .

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References: Hofmeister, A.M., and Rossman, G.R. (1984) determination of Fe^{3+} and Fe^{2+} concentrations in feldspar by optical absorption and EPR spectroscopy. *Phys. & Chem. of Minerals*, 11, 213-224. Shannon, R.D., et al. (1992a) Dielectric constants of crystalline and amorphous spodumene, anorthite and diopside and the oxide additivity rule. *Phys. & Chem. of Minerals*, 19, 148-156. Shannon, R.D., et al. (1992b) Dielectric constants of topaz, orthoclase, and scapolite and the oxide additivity rule. *Physics and Chemistry of Minerals*, 19, 166-170.

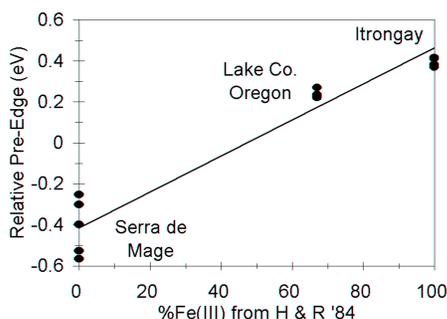


Figure 1. Feldspar calibration line for session nsls446. Error bars are ± 0.05 eV for pre-edge position and $\pm 10\%$ on $\% \text{Fe}^{3+}$.

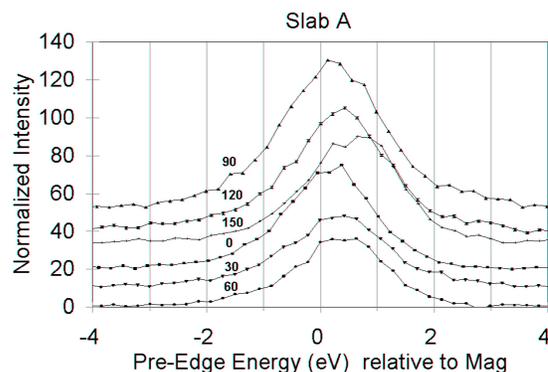


Figure 2. Polarized XANES pre-edge spectra of an orthoclase slab oriented perpendicular to the a axis. Spectra were taken at 30° increments of rotation relative to an arbitrary edge of the crystal.