American Mineralogist, Volume 98, pages 1660–1668, 2013

Amorphous Materials: Properties, structure, and durability

Analysis of H_2O in silicate glass using attenuated total reflectance (ATR) micro-FTIR spectroscopy‡

Jacob B. Lowenstern* and Bradley W. Pitcher†

U.S. Geological Survey, Volcano Science Center, MS 910, 345 Middlefield Road, Menlo Park, California 94025, U.S.A.

Abstract

We present a calibration for attenuated total reflectance (ATR) micro-FTIR for analysis of H_2O in hydrous glass. A Ge ATR accessory was used to measure evanescent wave absorption by H_2O within hydrous rhyolite and other standards. Absorbance at 3450 cm^{-1} (representing total H_2O or H_2Ot) and 1630 cm^{-1} (molecular H_2O or H_2O_m) showed high correlation with measured H_2O in the glasses as determined by transmission FTIR spectroscopy and manometry. For rhyolite,

$$wt\%H_2O = 245(\pm9)\cdot A_{3450} - 0.22(\pm0.03)$$

and

$$wt\%H_2O_m = 235(\pm11)\cdot A_{1630} - 0.20(\pm0.03)$$

where A_{3450} and A_{1630} represent the ATR absorption at the relevant infrared wavelengths. The calibration permits determination of volatiles in singly polished glass samples with spot size down to ~5 μm (for H_2O-rich samples) and detection limits of ~0.1 wt% H_2O. Basaltic, basaltic andesite and dacitic glasses of known H_2O concentrations fall along a density-adjusted calibration, indicating that ATR is relatively insensitive to glass composition, at least for calc-alkaline glasses. The following equation allows quantification of H_2O in silicate glasses that range in composition from basalt to rhyolite:

$$wt\%\ H_2O = (\omega\cdot A_{3450}/\rho) + b$$

where $\omega = 550 \pm 21$, $b = -0.19 \pm 0.03$, ρ = density, in g/cm^3, and A_{3450} is the ATR absorbance at 3450 cm^{-1}.

The ATR micro-FTIR technique is less sensitive than transmission FTIR, but requires only a singly polished sample for quantitative results, thus minimizing time for sample preparation. Compared with specular reflectance, it is more sensitive and better suited for imaging of H_2O variations in heterogeneous samples such as melt inclusions. One drawback is that the technique can damage fragile samples and we therefore recommend mounting of unknowns in epoxy prior to polishing. Our calibration should hold for any Ge ATR crystals with the same incident angle (31°). Use of a different crystal type or geometry would require measurement of several H_2O-bearing standards to provide a crystal-specific calibration.

Keywords: IR spectroscopy, glass properties, FTIR, water, new technique, igneous petrology, ATR, glass