ATR-FTIR spectroscopy to measure H_2O and CO_2 in silicate glass

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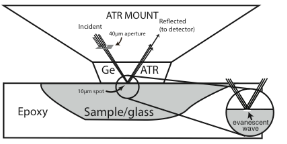
Over the past two years, we have developed protocols to measure H_2O and CO_2 in silicate glass with Attenuated Total Reflectance (ATR) FTIR spectroscopy. Our initial calibration [1] demonstrated the feasibility of the technique for quantifying total H_2O (H_2O_t), both as molecular H_2O (H_2O_m) and OH^- in glass within polished epoxy mounts prepared for EPMA and SIMS analysis.

In our setup, an ATR accessory crystal (Ge) is inserted into the FTIR microscope, the IR source beam is directed through the crystal, and a background spectrum is collected in air. By raising the stage, the sample makes contact with the Ge crystal, such that a small percentage of the incident light is absorbed within the upper few hundred nm of the sample. Our initial study [1] demonstrated that absorbance at 3450 cm^{-1} and 1630 cm^{-1} correlated with H_2O_t and H_2O_m concentration, respectively. This permits quantification of these species in silicate glass (calc-alkaline basalt through rhyolite) with a single calibration, adjusted for glass density. Other ATR crystal types and geometries will likely require separate calibration curves.

Recently, we explored calibrations for dissolved carbonate in basaltic and basaltic andesite glasses, as well as molecular CO_2 in rhyolitic glass. In both cases, calibration is clearly feasible, though with detection limits significantly higher than for transmission FTIR. By using a N_2-purge in our FTIR microscope to minimize atmospheric CO_2, we can resolve dissolved molecular CO_2 as low as 150 ppm. Carbonate peaks at 1400-1500 cm^{-1} are not affected by atmospheric CO_2, though H_2O vapor can interfere with resolution of the peaks, and as with transmission FTIR, peak heights are difficult to define due … line and the presence of the … ntly, the detection limits for … n. Our future work will be t … CO_2 in silicate glass, 2) low… 3) constrain the variation of tl… e coefficient) with glass com…



[1] Lowenstern & Pitcher (2013) *Am. Mineral.* **98**, 1660-1668.