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Analytical Protocol For Synchrotron X-Ray Fluorescence Microprobe Determination Of Halides In Low-Salinity Aqueous-Fluid Inclusions

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Aqueous halogens are particularly useful as tracers of geologic processes. Because aqueous fluid inclusions in quartz are relatively common and preserve past geologic fluids, we are developing a protocol for determining halogen ratios in these materials. The synchrotron X-ray fluorescence microprobe (SXRFM) is well suited for this purpose. Previous SXRFM investigations on fluid inclusions have been successful in determining chloride and bromide in briny inclusions having salinities of 20 wt % NaCl or more. Because we are interested in fluid inclusions that may have trapped ancient sea water and therefore preserve a record of its halogen chemistry, we are developing a protocol for determining Cl/Br in low-salinity inclusions. Characteristic X-rays for Cl are strongly absorbed by a quartz host at even moderate depths ($>5 \mu\text{m}$). Br in these inclusions is present at concentrations 200-300 times less than Cl. To confidently measure Cl/Br, we use a two-step procedure. For our experimental calibrations we prepared synthetic fluid inclusions with a salinity of 3.5 wt % and Cl/Br wt. ratios of 50, 250, and 500. Br is determined using an exciting energy of 13.7 keV, just above the critical excitation energy of 13.468 keV, and Cl is determined using a more appropriate exciting energy of 6 keV (the critical excitation energy for Cl is 2.819 keV). In order to co-register or correlate the two experiments, an element that is excited by both the 6 keV and the 13.7 keV beam and that has characteristic X-rays intermediate to those of Cl and Br is needed. We use about 15,000 ppm Iodine in synthetic inclusions for this purpose. Quantification of the X-ray spectra requires knowledge of the differential absorption of Cl and Br X-rays by the thickness of host material. This is accomplished by monitoring the differential absorption of the two intermediate L-lines (Iodine in the case of the synthetic inclusions). Ca, Mn, or Fe, which are common in natural fluid inclusions, can also be used. Initial results using synthetic fluid inclusions are promising. We hope to reduce analytical uncertainties and then apply this technique to determining Cl/Br concentrations in natural aqueous fluid inclusions.