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Characterization of fluor-chlorapatites by electron probe microanalysis with a focus on time-dependent intensity variation of halogens

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Abstract

Prior research has shown that fluorine and chlorine X-ray count rates vary with exposure to the electron beam during electron probe microanalysis (EPMA) of apatite. Stormer et al. (1993) and Stormer and Pierson (1993) demonstrate that the EPMA-operating conditions affect the halogen intensities in F-rich natural Durango and Wilberforce apatites and in a Cl-rich apatite. Following these studies, we investigated the effects of operating conditions on time-dependent X-ray intensity variations of F and Cl in a broad range of anhydrous fluor-chlorapatites. We tested 7, 10, and 15 kV accelerating voltages; 4, 10, and 15 nA beam currents; 2, 5, and 10 μm diameter fixed spot sizes; and the influence of 2 distinct crystal orientations under the electron beam. We find that the halogen X-ray intensity variations fluctuate strongly with operating conditions and the bulk F and Cl contents of apatite.

We determined the optimal EPMA operating conditions for these anhydrous fluor-chlorapatites to be: 10 kV accelerating voltage, 4 nA beam current (measured at the Faraday cup), 10 μm diameter fixed spot, and the apatite crystals oriented with their *c*-axes perpendicular to the incident electron beam. This EPMA technique was tested on a suite of 19 synthetic anhydrous apatites that covers the fluorapatite-chlorapatite solid-solution series. The results of these analyses are highly accurate; the F and Cl EPMA data agree extremely well with wet-chemical analyses and have an R^2 value >0.99.

Keywords: Fluorapatite, chlorapatite, fluor-chlorapatite, EPMA measurement, fluorine, chlorine, Durango fluorapatite