American Mineralogist, Volume 94, pages 1377–1387, 2009

Optimizing experimental design, overcoming challenges, and gaining valuable information from the Sb *K*-edge XANES region

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Abstract

There are many challenges associated with collecting, processing, and interpreting high-energy XAS data. The most significant of these are broad spectra, minimal separation of edge positions, and high background owing to the Compton tail. Studies of the Sb system are a particular challenge owing to its complex bonding character and formation of mixed oxidation-state minerals. Furthermore, in environmental samples such as stream sediment containing mine waste, different Sb phases may coexist. Ways to overcome these challenges and achieve accurate and useful information are presented. Our investigations used Sb *K*-edge X-ray absorption near-edge spectroscopy (XANES) to elucidate Sb geochemical behavior. Several Sb mineral spectra are presented, including Sb sulfosalts, and contrasted based on the different hosting and coordination environments around the Sb atom in the crystal structure. These comparisons lead to the recognition of how the different hosting and coordination environments are manifested in the shape of the Sb mineral spectra. In fact from the shape of the spectra, the occupation of the Sb atom in a single or in multiple crystallographic sites, regardless of whether multiple phases are present in the sample, is discernible. Furthermore, we demonstrate that quantitative information can be derived from the XANES region using linear combination fitting of the derivative spectra, rather than the energy spectra. Particularly useful to the advancement of Sb research is the demonstration that a significant amount of information can be gained from the Sb *K*-edge XANES region.

Keywords: Antimony, XANES, high-energy XAS, linear combination fitting, sulfosalt