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Synchrotron-based X-ray diffraction of the lead apatites series Pb10(PO4)6Cl2-Pb10(AsO4)6Cl2

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Pyromorphite Pb10(PO4)6Cl2 and mimetite Pb10(AsO4)6Cl2, members of the apatite group minerals, have recently gained considerable attention as a metal sequestration agents in water treatment and contaminated soil remediation. The unique crystal structure and chemistry of those minerals allows for numerous substitutions of both metal cations and anionic complexes. The vast majority of studies conducted to date, however focused on the cationic substitutions in these minerals. Little is known about the response of the structure to anionic substitutions, which may also influence properties, stability and environmental behaviour of studied apatites. Natural pyromorphite and mimetite often exhibit certain degree of anionic substitutions, which suggests relative stability of their solid solutions in natural environment. From diffraction studies on mimetie-pyromorphite series synthesized at 60-80° C it has been found that the solid solution between these two end members is complete. The temperature of the synthesis however, renders the obtained data inapplicable to near-Earth surface environments. Furthermore, published X-ray data is not supported by the chemical analysis of precipitated solids.

In order to supplement existing data, a number of compounds covering a wide range of compositions between mimetite-pyromorphite end members were synthesized by drop-wise mixing of the respective chemical reagents at room temperature. Products were characterized by SEM coupled with EDS as well as synchrotron-based X-ray diffraction. Diffraction experiments were carried on the dedicated high-resolution high-throughput powder diffractometer at Sector 11-BM B of the Advanced Photon Source, Argonne National Laboratory, USA. Analyses were performed at room temperature using monochromatic radiation of the wavelength of 0.42 Å. Intensities of the diffracted X-rays were collected on a 12-element Analyzer/Detector System offering supreme resolution and greatly reduced data collecting time. Raw data from each of the 12 detectors was calibrated, merged and reduced using in-house routine. The diffraction data was analyzed by Rietveld method using the computer program GSAS and EXPGUI. The background was modeled using Chebyschev polynomials of the first kind. Apatite starting atomic parameters came from the refinement based on neutron data in P63/m of Holly Springs hydroxylapatite. The results from Rietveld refinement confirm that the pyromorphite-mimetite solid solution is continuous at low temperatures. The refinement of the occupancies of the anionic position in the mineral structure of all synthesized compounds was in good agreement with the theoretical composition of the samples based on the chemistry of the starting solutions.

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