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Mineralogical composition of Sb-rich mine waste in Pezinok, Slovakia

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Antimony is classified as a priority pollutant; it is known to be toxic and probably also carcinogenic. Nevertheless, little attention is paid to this pollutant in comparison with its geochemical cousin, the element arsenic, because they commonly occur together. At past or active mining sites, the primary Sb minerals (mostly stibnite, Sb2S3) weather to form Sb oxides, Sb-rich hydrous ferric oxide, or to release Sb into the environment.
The Pezinok deposit was mined until 1992 and left ~80000 m3 of waste behind. Beside the rock-forming silicates, the waste contains mostly Fe oxides enriched in Sb and As. A minor fraction of the grains represents Sb oxides where Sb prevails over Fe and As. The back-scattered electron images suggest that the Fe-rich grains grew by precipitation from the aqueous solutions. On the other hand, the Sb-rich grains formed most likely by in-situ oxidation and transformation from stibnite (Sb2S3).
X-ray microdiffraction was carried out at the SUL-X beamline at the ANKA synchrotron source to identify the mineral phases present in the waste. The most common phase is goethite, very often with elevated Sb content. Interestingly, Fe oxides with higher As content appear to be X-ray amorphous. The grains where Sb dominates over Fe are poorly crystalline; Rietveld refinement of our patterns hints that this material is structurally close to the mineral tripuhyite (FeSbO4). The presence of poorly crystalline iron oxides (ferrihydrite) is likely but was obscured by intense background from the supporting glass.
X-ray fluorescence confirmed the close association of Fe and As in the studied samples. X-ray absorption near-edge structure (XANES) spectra measured on multiple weathering grains showed that arsenic is always present in its highest oxidation state, i.e., as As5+. The collected extended X-ray absorption fine-structure (EXAFS) spectra can be interpreted in terms of tetrahedral coordination of As by oxygen (as (AsO4)3-); the weaker features in the second coordination shell correspond probably to the bidentate-binuclear complex that has been postulated on the iron oxides in previously published studies. Currently, we are analysing the As EXAFS spectra from grains where Sb predominates and is assumed to be pentavalent.