

Klebelbergite, $\text{Sb}_4\text{O}_4\text{SO}_4(\text{OH})_2$: Stability relationships, formation in Nature, and refinement of its structure

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ABSTRACT

The extent to which secondary Sb minerals control Sb dispersion in the supergene environment is yet to be fully understood. Stability studies of klebelbergite have been undertaken to better understand its role in controlling Sb mobility and relationships with other secondary Sb minerals. Solubility in aqueous 0.1084 M HNO_3 was determined at 298.15 K and the data obtained used to derive ΔG_f° (klebelbergite, s, 298.15 K) = -2056.4 ± 5.0 kJ/mol. Solubility data have been used to deduce the conditions under which the mineral can form as a thermodynamically stable phase. The single-crystal X-ray structure of synthetic klebelbergite has been determined 293 K and is essentially the same as that reported earlier for atoms with $Z \geq 8$. Crystal data: orthorhombic, space group $Pca2_1$, $a = 5.7563(4)$, $b = 11.2538(7)$, $c = 14.8627(9)$ Å, $V = 962.81(11)$ Å³, $Z = 4$. Refinement converged to $R_1 = 0.0154$ for 2206 unique reflections with $I > 2\sigma(I)$. The present study has located the hydroxyl H atoms on both O5 and O9. The H-bond arrangements are somewhat different to those proposed earlier with the quasi-linear O9-H \cdots O3 interaction having $\angle(\text{DHA}) = 171(6)^\circ$. The O5 hydroxyl H atom lies in a different position to that proposed earlier and is involved in a bifurcated H-bond arrangement with O2 and with itself in a symmetry-related position, with $\angle(\text{DHA}) = 133(5)$ and $125(5)^\circ$, respectively.

Keywords: Klebelbergite, antimony, antimony sulfate, solubility, stability, structure, hydrogen bonding