

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

**ADAM J. ROPER<sup>1</sup>, PETER LEVERETT<sup>1</sup>, TIMOTHY D. MURPHY<sup>1</sup>, PETER A. WILLIAMS<sup>1,\*</sup> AND DAVID E. HIBBS<sup>2</sup>**

<sup>1</sup>School of Science and Health, University of Western Sydney, Locked Bag 1797, Penrith, New South Wales 2751, Australia

<sup>2</sup>Faculty of Pharmacy, University of Sydney, Sydney, New South Wales 2006, Australia

### **ABSTRACT**

The extent to which secondary Sb minerals control Sb dispersion in the supergene environment is yet to be fully understood. Stability studies of klebensbergite 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  $\text{HNO}_3$  was determined at 298.15 K and the data obtained used to derive  $\Delta G_f^\circ$  (klebensbergite, s, 298.15 K) =  $-2056.4 \pm 5.0$  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 klebensbergite 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,  $V = 962.81(11)$  Å<sup>3</sup>,  $Z = 4$ . 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:** Klebensbergite, antimony, antimony sulfate, solubility, stability, structure, hydrogen bonding