

Solvothermal Synthesis of $[\text{C}_6\text{H}_{17}\text{N}_3]\text{Sb}_{10}\text{S}_{16}$: A New Thioantimonate(III) with an *in-situ* Formed Organic Amine Cation

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Dedicated to Prof. Dr. H. Schmidbaur on the occasion of his 70th birthday

The new thioantimonate(III) $[\text{C}_6\text{H}_{17}\text{N}_3][\text{Sb}_{10}\text{S}_{16}]$ ($\text{C}_6\text{H}_{17}\text{N}_3$ = 2-piperazine-N-ethylamine cation) was obtained under solvothermal conditions showing a unique anionic framework. The compound crystallizes in the monoclinic space group $P2_1/c$ with four formula units in the unit cell. The lattice parameters are $a = 11.530(2)$, $b = 25.042(5)$, $c = 13.709(3)$ Å, $\beta = 111.25(3)^\circ$, $V = 3689(2)$ Å³. The thioantimonate(III) anion is formed by interconnection of nine trigonal pyramidal SbS_3 units and one SbS_4 moiety. These primary building units share common corners and edges yielding Sb_3S_3 and Sb_2S_2 hetero-rings. Further condensation leads to strong undulated two atoms thick layers extending in the [010] direction, with a modulation period of about 14 Å. Very large $\text{Sb}_{31}\text{S}_{31}$ rings within the layers show a ‘double-ellipsoidal’ shape with approximate dimensions of $8.9 \cdot 9.3$ Å. The cations are located at the inflexion points of the layers and act as pillars between successive layers. The layers are stacked onto each other in a way that channels parallel to [001] are formed accommodating the organic cations. A remarkable observation is that the 2-piperazine-N-ethylamine cation is formed by cyclization of tren molecules (tren = tris(2-aminoethyl)amine) under *in-situ* conditions.

Key words: Thioantimonate(III), Solvothermal Synthesis, Crystal Structure