

Preparation, Crystal Structure at 298 and 90 K and Phase Transition in $(\text{C}_2\text{H}_5\text{NH}_3)_2[\text{SbBr}_5]$ Studied by the Single Crystal X-Ray Diffraction Method

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The reaction of antimony(III) oxide with ethylamine, in molar ratios from 1:1 to 1:10, in concentrated hydrobromic acid leads to the formation of one product – bis(ethylammonium) pentabromoantimonate(III). The structure of $(\text{C}_2\text{H}_5\text{NH}_3)_2[\text{SbBr}_5]$ was determined at 298 and 90 K, below and above the phase transition that occurs at about 158.5 K. The orthorhombic system was found in both phases, space groups *Cmca* and *Pbca* at 298 and 90 K, respectively. At both temperatures the structure consists of $[\text{SbBr}_6]^{3-}$ octahedra connected *via cis* bromine atoms forming one-dimensional zig-zag $\{[\text{SbBr}_5]^{2-}\}_n$ chains. The ethylammonium cations fill the space between polyanionic chains. The organic and inorganic substructures are held together by a system of $\text{N}(\text{-H})\cdots\text{Br}$ interactions. Their influence on the deformation of $[\text{SbBr}_6]^{3-}$ octahedra is well reflected in differences in the corresponding Sb–Br bond lengths and Br–Sb–Br angles in both phases. The phase transition is of the first order and the order-disorder type. It is related to changes in the molecular dynamics of the ethylammonium cations. In the low-temperature phase the organic cations are ordered, while at 298 K both crystallographically independent cations are disordered. The type of disorder is realized by the presence of two positions of the methyl carbon atoms.

Key words: Bromoantimonates(III), Ethylammonium Cation, Phase Transition, Octahedral Distortion