Solvothermal Synthesis, Crystal Structure and Properties of [Mg(en)3][Sb4S7] – the First Thioantimonate(III) Containing a Main Group Metal Complex Cation as Structure Director

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Dedicated to Professor Hubert Schmidbaur on the occasion of his 75th birthday

The new thioantimonate [Mg(en)3][Sb4S7] containing for the first time a [Mg(en)3]2+ cation as structure-directing unit was synthesized under solvothermal conditions applying elemental Mg, SbCl3, S and ethylenediamine. The compound crystallizes in the monoclinic space group $P2_1/c$ with $a = 9.9267(6)$ Å, $b = 14.254(1)$ Å, $c = 17.259(1)$ Å, $\beta = 102.611(7)^\circ$, $V = 2383.1(3) \text{ Å}^3$, $Z = 4$. In the structure trigonal SbS3 pyramids are joined to form an Sb3S3 ring. The rings are connected through SbS3 units yielding an undulated chain anion running along [001]. Considering so-called secondary Sb–S bonds, a layer-like thioantimonate anion is formed. The [Mg(en)3]2+ cations are located between the layers. Relatively short S···H–N contacts suggest hydrogen bonding interactions between the cation and the [Sb4S7]2− anion. The compound starts to decompose at about 220 °C. The optical band gap of 2.35 eV is in agreement with the orange color of the crystals. In the Raman spectrum prominent Sb–S resonances are seen between 250 and 400 cm$^{-1}$ which can be assigned to different Sb–S vibrations.

Key words: Thioantimonate, Solvothermal Syntheses, Crystal Structure, Spectroscopic Properties