

# Solvothermal Synthesis, Crystal Structure and Properties of $[\text{Mg}(\text{en})_3][\text{Sb}_4\text{S}_7]$ – 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 75<sup>th</sup> birthday*

The new thioantimonate  $[\text{Mg}(\text{en})_3][\text{Sb}_4\text{S}_7]$  containing for the first time a  $[\text{Mg}(\text{en})_3]^{2+}$  cation as structure-directing unit was synthesized under solvothermal conditions applying elemental Mg,  $\text{SbCl}_3$ , S and ethylenediamine. The compound crystallizes in the monoclinic space group  $P2_1/c$  with  $a = 9.9267(6)$ ,  $b = 14.254(1)$ ,  $c = 17.259(1)$  Å,  $\beta = 102.611(7)^\circ$ ,  $V = 2383.1(3)$  Å<sup>3</sup>,  $Z = 4$ . In the structure trigonal  $\text{SbS}_3$  pyramids are joined to form an  $\text{Sb}_3\text{S}_3$  ring. The rings are connected through  $\text{SbS}_3$  units yielding an undulated chain anion running along  $[001]$ . Considering so-called secondary Sb–S bonds, a layer-like thioantimonate anion is formed. The  $[\text{Mg}(\text{en})_3]^{2+}$  cations are located between the layers. Relatively short  $\text{S} \cdots \text{H} - \text{N}$  contacts suggest hydrogen bonding interactions between the cation and the  $[\text{Sb}_4\text{S}_7]^{2-}$  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  $\text{cm}^{-1}$  which can be assigned to different Sb–S vibrations.

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