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**The crystal and molecular structure of 3-nitroperchlorylbenzene: Corrigendum.** By GUS J. PALENIK, *Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada*, JERRY DONOHUE, *Department of Chemistry, University of Pennsylvania, Philadelphia, Pennsylvania*, and K. N. TRUEBLOOD, *Department of Chemistry, University of California, Los Angeles, California, U.S.A.*

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In a paper on the title compound (Palenik, Donohue & Trueblood, 1968) acknowledgement of support of the National Science Foundation was inadvertently omitted.

#### Reference

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**The crystal structure of samsonite,  $2\text{Ag}_2\text{S} \cdot \text{MnS} \cdot \text{Sb}_2\text{S}_3$ .** By J. HRUŠKOVÁ, *Geological Survey, Prague 1, Hradební 9, Czechoslovakia*, and V. SYNEČEK, *Institute of Solid State Physics, Czechoslovak Academy of Sciences, Prague 6, Cukrovarnická 10, Czechoslovakia*

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The crystal structure of samsonite,  $2\text{Ag}_2\text{S} \cdot \text{MnS} \cdot \text{Sb}_2\text{S}_3$ , has been determined from three-dimensional X-ray data. The structure is characterized by the presence of  $\text{SbS}_3$  squat pyramids and slightly deformed  $\text{MnS}_6$  octahedra. It can be uniquely described as being built up of  $\text{S-MnSbAg}_2$  tetrahedra which share corner atoms with neighbouring tetrahedra.

According to Bragg's classification (Bragg & Claringbull, 1965) of sulphides, the mineral samsonite belongs to the group of compounds in which the metal atoms are incorporated in structures resembling the compounds of S, As, Sb, and Bi. We have undertaken the crystal-structure investigation of samsonite in an attempt to gain further data on the crystal chemistry of this complex group of minerals.

The first crystallographic data of samsonite were published by Werner & Fraatz (1910) who found the monoclinic symmetry and axial ratio of this mineral. The single-crystal X-ray diffraction investigation of Frondel (1941) led to the following lattice parameters:  $a = 10.31$ ,  $b = 8.07$ ,  $c = 6.62$  Å and the space group  $P2_1/n$ . From density measurements he found two formula units ( $Z = 2$ ) in the unit cell.

Our investigation was carried out on a single crystal of samsonite from a collection of minerals in the National Museum in Prague (Coll. No 591). Small single-crystal fragments of an appropriate shape were used for taking the rotation and zero-layer Weissenberg photographs about three crystallographic axes as well as for taking equi-inclination photographs about the [001] axis. All diffraction photographs were recorded with unfiltered Mo K radiation. Visually estimated intensities of reflexions were corrected for Lorentz and polarization factors and used for calculations of projections of the Patterson function along three crystallographic axes and of the Patterson-Harker sections  $(x, \frac{1}{2}, z)$  and  $(\frac{1}{2}, y, \frac{1}{2})$ . The interpretation of these syntheses led directly to the determination of the positions of the heavy atoms in the unit cell of samsonite. These positions served for the construction of projections of the minimum function along the [001] and [010] axes. The peaks in the latter syntheses were checked by consideration

of the reasonable packing of atoms in the unit cell of samsonite. This procedure enabled us to determine the sulphur atom positions. The first projections of electron density along the [001] and [010] axes were calculated with structure factors whose signs were already reliably determined from the heavy-atom positions. The agreement between the data of minimum and electron density projections was satisfactory. All atoms except Mn lie in general fourfold positions of the space group  $P2_1/n$ . Manganese atoms lie in one of the four twofold positions of this group.

Approximate coordinates of atoms are listed in Table 1. Although these coordinates require further refinement they enable us to describe the general features of the crystal structure of samsonite. Fig. 1 shows the electron-density projection and its interpretation in terms of the atomic positions in one unit-cell of samsonite. The presence of two atomic polyhedra – the squat pyramids,  $\text{SbS}_3$ , and slightly deformed octahedra,  $\text{MnS}_6$ , – is the most characteristic feature of the crystal structure of samsonite. Although the small difference in the scattering powers of Sb and Ag does not permit the reliable distinction between these two kinds of atoms from diffraction data only, the appearance of the pyramidal  $\text{SbS}_3$  group indirectly indicates the correct fourfold positions occupied by the Sb atoms. The  $\text{SbS}_3$  group is a squat pyramid, with an Sb atom at the vertex and three sulphur atoms at an average distance of 2.4 Å at the basal corners; this can be compared with the corresponding Sb-S distance in proustite, equal to 2.44 Å (Harker, 1936). The sulphur atoms forming the triangular base of one  $\text{SbS}_3$  pyramid lie in three different fourfold positions; their average separation is 3.6 Å.

The manganese atoms are situated in one of four twofold positions of the centres of symmetry within the octa-