

## Silver(I) trimercury(II) antimonate(V), $\text{AgHg}_3\text{SbO}_6$

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Single crystals of  $\text{AgHg}_3\text{SbO}_6$  were obtained from solid-state synthesis at elevated oxygen pressures. The structure exhibits a variation of the  $\text{K}_4\text{CdCl}_6$  structure type. Chains of face-sharing  $\text{SbO}_6$  and elongated  $\text{AgO}_6$  octahedra run along  $[001]$ , and these chains are connected by linearly coordinated Hg atoms. The occurrence of  $\text{AgO}_6$  octahedra instead of trigonal prisms, and of O—Hg—O dumbbells instead of irregular eight-coordinated oxygen polyhedra, distinguishes the new compound from the known analogues of this type of structure. The heavy atoms are located on special positions; Ag is at a site with 32 symmetry, Sb at a site with  $\bar{3}$  symmetry and Hg at a site with twofold symmetry.

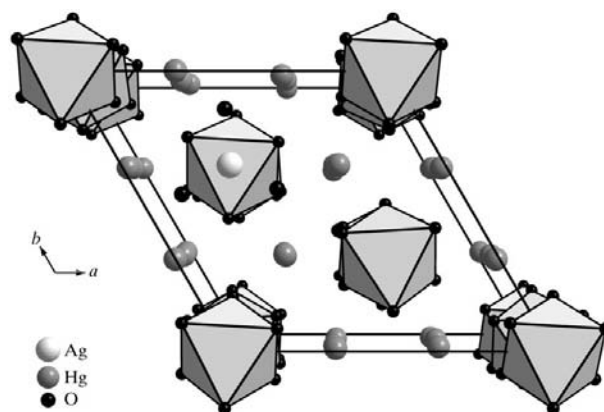
### Comment

$\text{AgHg}_3\text{SbO}_6$  crystallizes rhombohedrally in space group  $R\bar{3}c$ , with one independent atom of each element at the Wyckoff positions  $6b$ ,  $18e$ ,  $6a$  and  $36f$  of the hexagonal setting. Its crystal structure (Fig. 1) consists of isolated, almost ideal,  $\text{SbO}_6$  octahedra separated by  $\text{Ag}^+$  ions in the  $c$  direction and by  $\text{Hg}^{2+}$  ions in the  $ab$  plane. The  $\text{Ag}^+$  ions are found to be in an octahedral environment of oxygen and the  $\text{Hg}^{2+}$  ions in a linear coordination. All  $M$ —O distances are in very good agreement with other compounds containing these elements with similar coordination numbers. The  $\text{SbO}_6$  octahedra are arranged in the sense of a cubic close packing. The  $\text{SbO}_6$  and trigonally elongated  $\text{AgO}_6$  octahedra are stacked alternately to form chains along  $[001]$  by sharing faces (Fig. 2a). These chains are surrounded by six spiral rods of Hg, and the Hg rods are in turn surrounded by three polyhedral chains.

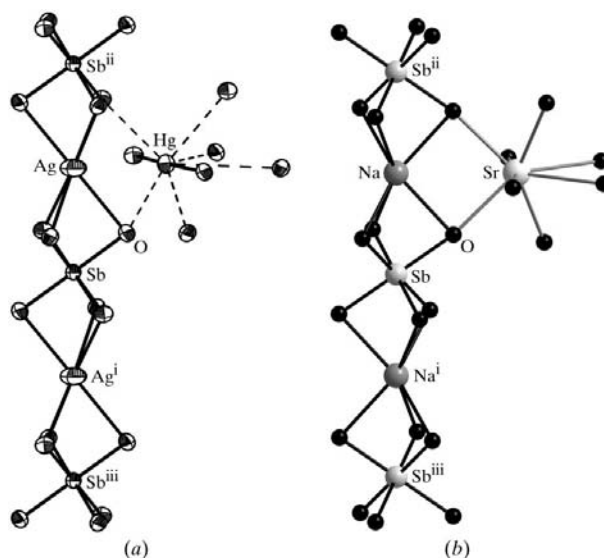
The structure closely resembles the  $\text{K}_4\text{CdCl}_6$  structure type (Bergerhoff & Schmitz-Dumont, 1956), where  $\text{K}^+$  ions at two crystallographically different sites separate isolated  $\text{CdCl}_6$  octahedra. In detail, one  $\text{K}^+$  in a trigonal-prismatic oxygen coordination, together with the  $\text{CdCl}_6$  octahedra, form chains along  $[001]$ ; the octahedra in these columns are present in two alternating orientations. The remaining three of the four  $\text{K}^+$  ions are situated between these columns in irregular coordination polyhedra of eight  $\text{Cl}^-$  ions. Derivatives of this struc-

ture type are adopted by many oxides to form compounds of the general type  $A_3A'BO_6$  [e.g.  $\text{Sr}_4\text{PtO}_6$  (Randall & Katz, 1959),  $\text{Sr}_3\text{LiIrO}_6$  (Davis *et al.*, 2003, and references therein),  $\text{Sr}_3\text{NaSbO}_6$  (Battle *et al.*, 2001) and  $\text{Sr}_3\text{CuIrO}_6$  (Neubacher & Müller-Buschbaum, 1992)].

Although  $\text{AgHg}_3\text{SbO}_6$  and  $\text{K}_4\text{CdCl}_6$  are isopointal, the structures exhibit striking differences. Firstly, the  $\text{Hg}^{2+}$  ion does not behave as a spherical alkaline or alkaline earth cation, but prefers a characteristic dumbbell-like coordination by only two O atoms in the title compound. The next four O-atom neighbours at about 2.8–2.9 Å cannot be attributed to the first coordination sphere of  $\text{Hg}^{2+}$ , and two more O atoms



**Figure 1**  
A perspective view of the crystal structure of  $\text{AgHg}_3\text{SbO}_6$ , showing the Ag and Hg atoms and  $\text{SbO}_6$  octahedra. The octahedra are tilted outwards at an angle of  $6.5^\circ$ .



**Figure 2**  
(a) A column of  $\text{SbO}_6$  and strongly elongated  $\text{AgO}_6$  octahedra in  $\text{AgHg}_3\text{SbO}_6$ . The coordination of one Hg atom is indicated; dashed lines denote distances longer than 2.78 Å. Displacement ellipsoids are drawn at the 50% probability level. (b) A related column of  $\text{SbO}_6$  octahedra and twisted trigonal  $\text{NaO}_6$  prisms in  $\text{Sr}_3\text{NaSbO}_6$  ( $\text{K}_4\text{CdCl}_6$  type; Battle *et al.*, 2001). One  $\text{Sr}^{2+}$  ion with eight coordinating O atoms is also shown. The tilt of the octahedra is about  $45.3^\circ$ . All unlabelled atoms are oxygen. [Symmetry codes: (i)  $-x, -y, -z$ ; (ii)  $x - y, -y, -z + \frac{1}{2}$ ; (iii)  $x - y, -y, -z - \frac{1}{2}$ .]

are found at an even more remote distance of 3.4 Å. This strongly directing feature of the packing causes significant deviations from the  $K_4CdCl_6$  type. In particular, the  $SbO_6$  octahedra, which share O atoms with the O—Hg—O dumbbells, are in an eclipsed orientation along the  $c$  axis, with a dihedral angle of  $6.5^\circ$ . In all related compounds of this structure type with alkali or alkaline earth elements instead of mercury, this angle is found to be in the range  $40\text{--}50^\circ$  [e.g.  $45.3^\circ$  in  $Sr_3NaSbO_6$  (Battle *et al.*, 2001) and  $42.3^\circ$  in  $K_4CdCl_6$  (Beck & Milius, 1986)]. As another consequence, the  $Ag^+$  ion in  $AgHg_3SbO_6$  is coordinated by a heavily elongated but slightly twisted octahedron of O atoms, as illustrated in Fig. 2(a), while the  $A'O_6$  polyhedra in all other compounds adopting the  $K_4CdCl_6$  structure type must always be described as twisted trigonal prisms (Fig. 2b).

## Experimental

Red crystals of  $AgHg_3SbO_6$  were obtained as a by-product from the reaction of  $Ag_2O$  (290 mg),  $HgO$  (108 mg) and  $Sb_2O_3$  (73 mg) at elevated oxygen pressures of 100 MPa at 773 K (Linke & Jansen, 1997). The reactants were finely ground and placed in a gold crucible, which was then sealed from one side and mechanically closed from the other. Single crystals were isolated and glued on to glass capillaries.

### Crystal data

$AgHg_3SbO_6$	Mo $K\alpha$ radiation
$M_r = 927.39$	Cell parameters from 3254 reflections
Trigonal, $R\bar{3}c$	$\theta = 2.6\text{--}27.2^\circ$
$a = 9.627$ (3) Å	$\mu = 74.86$ mm $^{-1}$
$c = 12.601$ (4) Å	$T = 293$ (2) K
$V = 1011.3$ (5) Å $^3$	Block, red
$Z = 6$	$0.2 \times 0.2 \times 0.2$ mm
$D_x = 9.136$ Mg m $^{-3}$	

### Data collection

Stoe IPDS-2 diffractometer	177 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{int} = 0.084$
Absorption correction: integration ( <i>X-SHAPE</i> ; Stoe & Cie, 2002)	$\theta_{max} = 27.1^\circ$
$T_{min} = 0.003$ , $T_{max} = 0.018$	$h = -12 \rightarrow 6$
901 measured reflections	$k = 0 \rightarrow 12$
254 independent reflections	$l = -16 \rightarrow 15$

### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$\Delta\rho_{max} = 2.04$ e Å $^{-3}$
$wR(F^2) = 0.060$	$\Delta\rho_{min} = -4.04$ e Å $^{-3}$
$S = 0.85$	Extinction correction: <i>SHELXL97</i> (Sheldrick, 1997)
254 reflections	Extinction coefficient: 0.00039 (4)
20 parameters	
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

**Table 1**

Selected geometric parameters (Å, °).

Sb—O ( $\times 6$ )	1.997 (9)	Hg—O <sup>ii</sup> ( $\times 2$ )	2.899 (9)
Hg—O <sup>i</sup> ( $\times 2$ )	2.057 (11)	Hg—O <sup>iii</sup> ( $\times 2$ )	3.463 (11)
Hg—O ( $\times 2$ )	2.789 (10)	Ag—O ( $\times 6$ )	2.556 (9)
O <sup>iv</sup> —Sb—O	180.0	O <sup>ix</sup> —Ag—O	111.2 (4)
O—Sb—O <sup>v</sup>	90.9 (4)	O—Ag—O <sup>x</sup>	175.9 (5)
O <sup>i</sup> —Hg—O <sup>vii</sup>	174.4 (5)	O—Ag—O <sup>vi</sup>	66.5 (3)
O <sup>viii</sup> —Ag—O	116.1 (4)		

Symmetry codes: (i)  $\frac{2}{3} - x, \frac{1}{3} - y, \frac{1}{3} - z$ ; (ii)  $\frac{2}{3} - x + y, \frac{1}{3} - x, \frac{1}{3} + z$ ; (iii)  $\frac{2}{3} + y, \frac{1}{3} - x + y, \frac{1}{3} - z$ ; (iv)  $-x, -y, -z$ ; (v)  $y, -x + y, -z$ ; (vi)  $-y, x - y, z$ ; (vii)  $\frac{1}{3} - x + y, y - \frac{1}{3}, z + \frac{2}{3} - 1$ ; (viii)  $y, x, \frac{1}{2} - z$ ; (ix)  $x - y, -y, \frac{1}{2} - z$ ; (x)  $-x, -x + y, \frac{1}{2} - z$ .

In consideration of the tendencies of Ag and Hg to adopt similar crystal chemical environments, partial Ag occupancies of the Hg-atom positions and *vice versa* were used in the initial stages of refinement, but the values refined to zero immediately in both cases.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: IZ1063). Services for accessing these data are described at the back of the journal.

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