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minated by four O atoms in a  $\Psi$ -trigonal bipyramidal geometry. The Sb—O distances of 1.95 (1) (2 $\times$ ) and 2.17 (1) Å (2 $\times$ ) (Fig. 1) are in the range known for other Sb—O compounds [1.90 Å in Na<sub>3</sub>SbO<sub>3</sub> (Stöver & Hoppe, 1980); 1.98 Å in cubic Sb<sub>2</sub>O<sub>3</sub> (Svensson, 1974); 1.98 Å in orthorhombic Sb<sub>2</sub>O<sub>3</sub> (Svensson, 1975)]. The two short Sb—O bonds in the equatorial position of the  $\Psi$ -trigonal bipyramid include an angle of 103.4 (3)°, whereas the O(equatorial)—Sb1—O(axial) angles deviate only slightly from 90°. The O—Sb—O angle involving the two axial ligands [161.0 (3)°] is appreciably smaller than 180°, in accordance with the Gillespie–Nyholm concept.

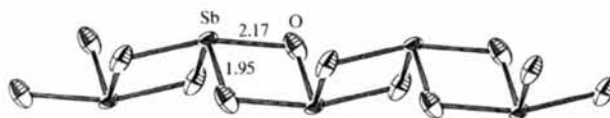


Fig. 1. View of the  $\infty$ [SbO<sub>2/2</sub>O'<sub>2/2</sub>] chains (50% probability ellipsoids). Sb—O distances are given in Å.

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## Caesium Dioxoantimonate(III), CsSbO<sub>2</sub>

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### Abstract

CsSbO<sub>2</sub> crystallizes in the monoclinic space group *C2/c*, is isotypic with NaBiO<sub>2</sub> and CsBiO<sub>2</sub>, and contains infinite  $\infty$ [SbO<sub>2/2</sub>O'<sub>2/2</sub>] chains running parallel to [001].

### Comment

In the Cs—Sb—O system, the oxoantimonates(V) Cs<sub>3</sub>SbO<sub>4</sub> (Duquenoy, 1974) and CsSbO<sub>3</sub> (Josien & Duquenoy, 1980) are known from X-ray powder investigations, but no structural parameters based on single-crystal data have been published until now. Whereas the complete series of alkali metal oxobismuthates(III) is known, the caesium oxoantimonate(III) CsSbO<sub>2</sub>, which can be synthesized by reaction of Sb<sub>2</sub>O<sub>3</sub> with elemental caesium, is the first Sb<sup>III</sup> compound in the corresponding ternary Sb system to be characterized by single-crystal methods. It crystallizes in the monoclinic system with space group *C2/c* and is isotypic with the bismuthates(III) NaBiO<sub>2</sub> (Schwedes & Hoppe, 1972) and CsBiO<sub>2</sub> (Zoche & Jansen, 1997). In accordance with the lone pair of Sb<sup>III</sup>, the Sb atoms are coor-

The SbO<sub>4</sub>  $\Psi$ -trigonal bipyramids are connected *via* common edges to form infinite  $\infty$ [SbO<sub>2/2</sub>O'<sub>2/2</sub>] chains running parallel to the [001] direction. The chains form a hexagonal closed-rod packing.

Each Cs atom is coordinated by six O1 atoms in a distorted trigonal prismatic arrangement, with Cs—O distances ranging from 2.98 (1) to 3.38 (1) Å (Fig. 2). Their coordination polyhedra are connected *via* common edges to form chains running parallel to [101]; the

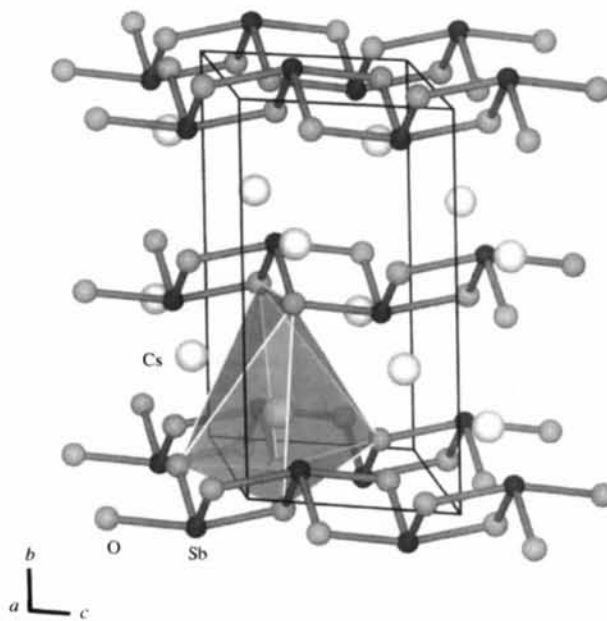


Fig. 2. The crystal structure of CsSbO<sub>2</sub> showing the caesium coordination polyhedron.

chains are linked by common corners. Each [CsO<sub>6/3</sub>] polyhedron connects three Sb—O chains.

As expected, the Sb—O bonds in CsSbO<sub>2</sub> are somewhat shorter than the Bi—O bonds in CsBiO<sub>2</sub> [2.06 (2×) and 2.34 Å (2×)]. The O—Sb—O angles are comparable to the corresponding O—Bi—O angles in CsBiO<sub>2</sub>. The most remarkable difference between the antimonate and the bismuthate is the distinctly longer *b* axis of CsSbO<sub>2</sub> (*b/c* = 1.75 for CsSbO<sub>2</sub> versus 1.54 for CsBiO<sub>2</sub>). The orientation of the lone pair parallel to the *b* axis hints at a more stereochemically active lone pair for Sb than for Bi.

## Experimental

Liquid caesium (443 mg, 3.33 mmol, ABCR, 99.9%) was reacted with powdered Sb<sub>2</sub>O<sub>5</sub> (548 mg, 1.70 mmol, ABCR, 99%) in a corundum crucible under an argon atmosphere. The mixture was heated to 773 K over a period of 5 h and was then cooled to room temperature at a rate of 4 K h<sup>-1</sup>. The colourless crystals of CsSbO<sub>2</sub> obtained were prismatic. The X-ray powder patterns of the samples can be indexed with the single-crystal data of CsSbO<sub>2</sub> and show weak reflections of CsO<sub>2</sub> and of additional unknown compounds.

### Crystal data

CsSbO<sub>2</sub>  
*M<sub>r</sub>* = 286.66  
 Monoclinic  
*C2/c*  
*a* = 8.1023 (8) Å  
*b* = 9.7724 (10) Å  
*c* = 5.5763 (8) Å  
 $\beta$  = 121.90 (8)°  
*V* = 374.84 (8) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 5.080 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.7107 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 10.3–25.6°  
 $\mu$  = 16.716 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism  
 0.48 × 0.25 × 0.18 mm  
 Colourless, transparent

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: analytical, numerical (Alcock & Marks, 1993)  
 $T_{\min}$  = 0.037,  $T_{\max}$  = 0.121  
 470 measured reflections  
 429 independent reflections

416 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}}$  = 0.035  
 $\theta_{\max}$  = 27.48°  
 $h$  = -10 → 8  
 $k$  = 0 → 12  
 $l$  = 0 → 7  
 3 standard reflections  
 frequency: 60 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)]$  = 0.038  
 $wR(F^2)$  = 0.107  
 $S$  = 1.14  
 429 reflections

$\Delta\rho_{\max}$  = 1.94 e Å<sup>-3</sup>  
 $\Delta\rho_{\min}$  = -2.50 e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97* (Sheldrick, 1997)

21 parameters  
 $w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 9.3684P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.043$

Extinction coefficient: 0.0099 (11)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Cs1—O1 <sup>i</sup>	2.984 (6)	Sb1—O1 <sup>iii</sup>	1.954 (5)
Cs1—O1 <sup>ii</sup>	3.159 (6)	Sb1—O1 <sup>i</sup>	2.168 (5)
Cs1—O1 <sup>iii</sup>	3.382 (6)		
O1 <sup>iii</sup> —Sb1—O1 <sup>ii</sup>	103.4 (3)	O1 <sup>iii</sup> —Sb1—O1 <sup>i</sup>	78.3 (2)
O1 <sup>iii</sup> —Sb1—O1 <sup>i</sup>	89.9 (2)	O1 <sup>i</sup> —Sb1—O1 <sup>iii</sup>	161.0 (3)

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

The atomic coordinates for CsBiO<sub>2</sub> were taken as a starting model for the refinement. A numerical absorption correction was applied using the program *ABSPSI* (Alcock & Marks, 1993). Face indices and distances to the centre were respectively: 100 and  $\bar{1}00$ , 0.24 mm; 010 and  $\bar{0}10$ , 0.12 mm; 001 and  $\bar{0}0\bar{1}$ , 0.09 mm.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *HELENA* (Spek, 1993). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP* (Johnson, 1968) and *DRAWxtl* (Finger & Kroeker, 1997). 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: JZ1279). Services for accessing these data are described at the back of the journal.

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