Crystal structure of caesium tetrathioantimonate(V), Cs_3SbS_4

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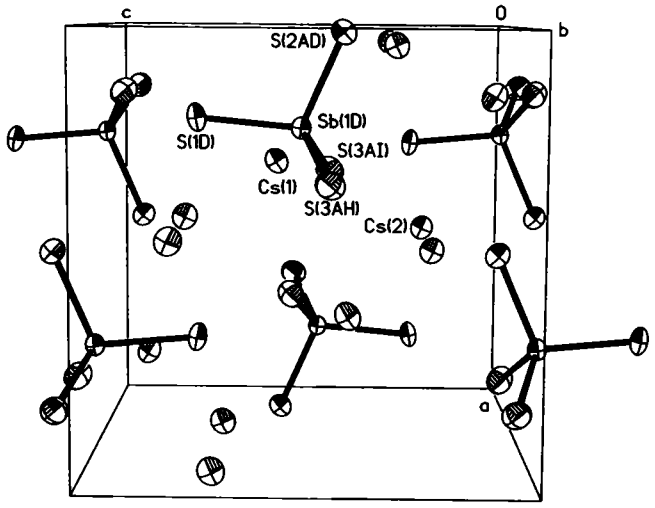

Abstract

Cs_3S_4Sb, orthorhombic, *Pnma* (No. 62), $a = 9.940(2)$ Å, $b = 11.667(2)$ Å, $c = 9.991(2)$ Å, $V = 1158.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.046$, $wR_{ref}(F^2) = 0.082$, $T = 298$ K.

Source of material

Crystals of Cs_3SbS_4 were serendipitously formed from a molten chalcogenide flux reaction of Pr, Sb, Ag, and S in Cs_2S_2. 35.3 mg Pr, 169.9 mg Sb_2S_3, 62.0 mg Ag_2S, 53.9 mg Ag, and 164.9 mg Cs_2S_2 were combined in a fused silica ampoule in an inert atmosphere glovebox, sealed under vacuum, and heated to 1123 K at a rate of 35 K / hour. After 72 hours of heating, the ampoule was cooled at 4 K / hour to room temperature. Dimethylformamide was added to dissolve remaining flux, resulting in red crystals of Cs_3SbS_4. Electron microprobe analysis confirmed the presence of Cs, Sb, and S in the crystals. Pr and Ag were found in the reaction mixture in the form of binary sulfides.

Discussion

This compound is isostructural to Rb_3SbS_4 [1]. The structure consists of isolated anionic SbS_4 tetrahedra surrounded by Cs cations.

Table 1. Data collection and handling.

Crystal:	red block, size 0.16 × 0.12 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	123.20 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, 600 frames, $\Delta\omega = 0.3°$, $\varphi = 0$, 120, 240
$2\theta_{max}$:	46.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6872, 870
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 574
$N(param)_{refined}$:	43
Program:	SHELXTL [2]

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References

1. Bensch, W.; Duerichen, P.: Crystal structure of rubidium tetrathioantimonate(V), Rb_3SbS_4. Z. Kristallogr. **149** (1996) 636.
2. SHELXTL. Structure Determination Programs. Version 5.1, Bruker AXS, Inc., Madison, Wisconsin, USA1998.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs(1)	4*c*	0.3470(2)	1/4	0.5876(2)	0.040(1)	0.068(1)	0.034(1)	0	–0.0064(9)	0
Cs(2)	8*d*	0.5452(1)	0.04146(9)	0.2046(1)	0.0395(7)	0.0241(6)	0.0353(7)	0.0000(6)	0.0024(6)	–0.0009(5)
Sb(1)	4*c*	0.2750(2)	1/4	0.0188(2)	0.0246(9)	0.0200(9)	0.019(1)	0	–0.0008(8)	0.0
S(1)	4*c*	0.2967(6)	1/4	0.2512(5)	0.042(4)	0.024(3)	0.017(4)	0	0.004(3)	0.0
S(2)	4*c*	0.4894(6)	1/4	0.9252(6)	0.032(4)	0.032(4)	0.031(4)	0	0.005(3)	0
S(3)	8*d*	0.3366(5)	–0.0844(4)	0.4541(5)	0.040(3)	0.040(3)	0.040(3)	–0.012(2)	–0.005(2)	0.013(2)

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