

Discussion. Salts of the A_2PtF_6 type have been made only with Group I elements; all have the K_2GeF_6 structure (Wyckoff, 1965). We have found that $(NH_4)_2PtF_6$ has the K_2PtCl_6 (antifluorite) structure, in which the K^+ and $PtCl_6^{2-}$ ions occupy the F^- and Ca^{2+} sites of the fluorite structure respectively.

There is no distortion from full $m3m$ symmetry allowed for the PtF_6^{2-} ion in space group $Fm3m$, and refinement in lower-symmetry space groups did not lead to any significant departure from this geometry. Other MF_6^{n-} anions commonly show a departure from $m3m$ symmetry by a compression along a threefold axis (Clark & Russell, 1978). The Pt–F distance is 1.942 (8) Å [1.936 (4) Å in $R\bar{3}$]. The H atoms were not located.

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Structure of Bismuth Indium Sulphide $Bi_3In_5S_{12}$ *

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Abstract. $Bi_3In_5S_{12}$, monoclinic, $C2/m$, $a = 33.13$ (1), $b = 3.873$ (1), $c = 14.413$ (2) Å, $\beta = 91.21$ (2)°, $V = 1849.0$ Å³, $Z = 4$, $D_c = 5.73$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 345.8$ mm⁻¹, $F(000) = 2744$; reflection condition $hkl: h + k = 2n$; final $R = 0.067$. The structure is built up by irregular In–S octahedra and distorted mono- and bicapped trigonal prisms of Bi–S, forming chains along y .

Introduction. In the system In_2S_3 – Bi_2S_3 , three intermediate compounds were found (Krämer, 1976). Two of these have the compositions $In_2Bi_4S_9$ and $Bi_2In_4S_9$ (Krämer, 1971; Chapuis, Gnehm & Krämer, 1972); the composition of the third compound could not be determined chemically with sufficient accuracy. Therefore a structure analysis was performed which shows the correct formula to be $Bi_3In_5S_{12}$ ($3Bi_2S_3 \cdot 5In_2S_3$).

Crystals could be prepared with chlorine as transport agent in a temperature gradient of 953–873 K. By annealing stoichiometric amounts of Bi_2S_3 and In_2S_3 for several weeks at 873 K only $Bi_2In_4S_9$ and $In_2Bi_4S_9$ were formed in the corresponding ratio. Therefore chlorine is thought to stabilize the $Bi_3In_5S_{12}$ structure and small proportions thereof may be incorporated which cannot be detected by X-ray diffraction.

As-grown crystals are black and elongated along y , showing the pinacoids $\{100\}$, $\{010\}$ and $\{001\}$. Intensities from a single crystal ($25 \times 900 \times 63$ µm) were collected on an automatic four-circle diffractometer (Enraf–Nonius CAD-4) with graphite-monochromatized Mo $K\alpha$ radiation and an ω – 2θ scan mode. Calculations were performed with the XRAY system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). 7370 reflections were measured which reduced to 3092 independent reflections, 2788 of which had $I > 3\sigma(I)$. Intensities were corrected for Lorentz–polarization and absorption effects. The structure was solved from a Patterson map and successive Fourier syntheses. Refinement of positional and anisotropic

Table 1. Fractional atomic coordinates ($\times 10^4$) and their e.s.d.'s (< 1 in last digit for In and Bi)

	x	y = 0	z		x	y = 0	z
Bi(1)	1353		–124	S(1)	–60 (2)		1789 (4)
Bi(2)	3203		2623	S(2)	623 (2)		4009 (4)
Bi(3)	5516		2083	S(3)	1160 (2)		1817 (4)
				S(4)	2224 (2)		2404 (4)
In(1)	0	0	0	S(5)	3939 (2)		1853 (4)
In(2)	0		5000	S(6)	4665 (2)		3936 (4)
In(3)	2150		4167	S(7)	5538 (2)		266 (4)
In(4)	4032		5044	S(8)	6616 (2)		3988 (4)
In(5)	6729		2184	S(9)	8029 (2)		1459 (4)
In(6)	9277		2794	S(10)	8731 (2)		3972 (4)
				S(11)	7718 (2)		4111 (4)
				S(12)	6839 (2)		404 (4)

* Dedicated to Professor Dr Werner Borchert on the occasion of his 70th birthday.

Table 2. *Interatomic distances (Å) and their e.s.d.'s*

Bi(1)—S(5)	3.285 (5) 2×	In(3)—S(8)	2.632 (4) 2×
S(7)	3.379 (6) 2×	S(11)	2.702 (5) 2×
S(12)	2.622 (4) 2×	S(4)	2.557 (6)
S(3)	2.883 (6)	S(11)	2.512 (6)
S(9)	2.840 (6)	In(4)—S(2)	2.619 (4) 2×
Bi(2)—S(9)	2.619 (4) 2×	S(10)	2.658 (4) 2×
S(10)	3.232 (5) 2×	S(6)	2.664 (6)
S(11)	3.329 (5) 2×	S(8)	2.584 (6)
S(4)	3.253 (6)	In(5)—S(3)	2.748 (5) 2×
S(5)	2.700 (7)	S(4)	2.552 (4) 2×
Bi(3)—S(1)	2.745 (5) 2×	S(8)	2.635 (6)
S(2)	3.397 (5) 2×	S(12)	2.599 (6)
S(3)	2.912 (5) 2×	In(6)—S(5)	2.604 (4) 2×
S(7)	2.621 (6)	S(6)	2.833 (5) 2×
In(1)—S(1)	2.590 (6) 2×	S(1)	2.657 (7)
S(7)	2.654 (5) 4×	S(10)	2.508 (6)
In(2)—S(2)	2.533 (6) 2×		
S(6)	2.695 (4) 4×		

thermal parameters resulted in a final $R = 0.067^*$ and an average shift/error of 1.5×10^{-5} . Scattering factors of neutral atoms (Cromer & Mann, 1968) were used and corrected for anomalous dispersion. The atomic coordinates are listed in Table 1, bond lengths in Table 2.

Discussion. A view of the complete structure is displayed in Fig. 1. All atoms are located on the mirror planes at $y = 0$ and $\frac{1}{2}$. In(1,2) occupy special positions at the centres of inversion at 000 and $00\frac{1}{2}$, all others being in general positions. In(1–6) are surrounded by six S (distorted octahedra), whereas Bi(1,2) are eightfold (distorted bicapped trigonal prisms), and

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35249 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Antimony(III) Arsenic(V) Oxide

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Abstract. AsSbO_4 , $M_r = 260.67$, monoclinic, $P2_1/m$, $a = 4.794$ (2), $b = 6.925$ (2), $c = 5.307$ (2) Å, $\beta = 93.55$ (2)°, $U = 175.9$ Å³, $Z = 2$, $D_x = 4.923$ Mg m⁻³, $\mu = 17.1$ mm⁻¹ (Mo K α). $R = 5.6\%$ for 500 unique

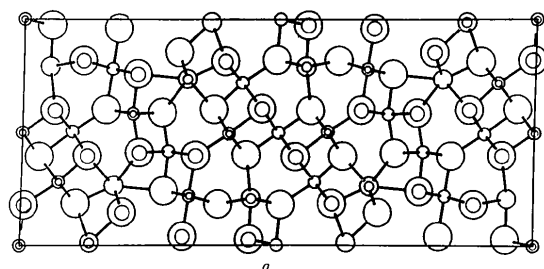


Fig. 1. Structure of $\text{Bi}_3\text{In}_5\text{S}_{12}$ viewed along b ; rings are at $y = \frac{1}{2}$, double rings at $y = 0$ (small: In, medium: Bi, large: S).

Bi(3) is sevenfold (distorted monocapped trigonal prism) coordinated. The In—S distances range from 2.51 to 2.83 Å, the Bi—S from 2.62 to 3.40 Å. The In—S octahedra are edge-shared, and the Bi—S prisms are stacked along y , all forming chains parallel to b . There is only a faint resemblance to $\text{Bi}_2\text{In}_4\text{S}_9$ (Chapuis, Gnehm & Krämer, 1972) where partially sixfold coordinated Bi and fivefold coordinated In were found which do not show up in the structure presented here.

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