Refinement

Refinement on *F*
$R = 0.020$
$wR = 0.024$
$S = 1.48$
164 reflections
$w = 1/\sigma(F_o)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 1.713$ e Å^{-3}
$\Delta\rho_{\min} = 1.216$ e Å^{-3}

Extinction correction: *TEXSAN* (Molecular Structure Corporation, 1989)
Extinction coefficient: 0.47 (4) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
In	0	0	0	0.21 (1)
O	0.7138 (3)	0	1/4	0.30 (4)
B	0	0	1/4	0.6 (1)

Table 2. *Selected geometric parameters* (Å, °)

In—O	2.1575 (7)	O···O	2.390 (2)
B—O	1.380 (1)		
O—In—O	91.91 (2)	In—O—In	122.92 (6)
O—B—O	120	B—O—In	118.54 (3)

A weighting scheme $w = 1/\sigma(F_o)$ was employed in which $\sigma(F_o^2) = [C + \frac{1}{4}(t_c/t_b)^2(b_1 + b_2) + (pxl)^2]^{1/2}$, where C is the total number of counts per peak, t_c is the time spent counting peak intensity, t_b is the time spent counting one side of background, b_1 are the high-angle background counts, b_2 are the low-angle background counts, p is the ignorance factor, and l is $C - \frac{1}{2}(t_c/t_b)(b_1 + b_2)$.

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Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: BR1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$Sr_{11}Sb_{10}$

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Abstract

Single crystals of tetragonal (*I*4/*mmm*) $Sr_{11}Sb_{10}$ can be prepared by reaction of the elements in a welded Nb container. Although the existence of $Sr_{11}Sb_{10}$ has been reported, to date no crystal structure determination has been reported.

Comment

The Sr–Sb binary system contains a number of compounds (Scott, 1990) such as SrSb, $SrSb_3$, Sr_3Sb_2, Sr_2Sb (Martinez-Ripoll, Haase & Brauer, 1973), $SrSb_2$ (Deller & Eisenmann, 1976*b*), Sr_2Sb_3 (Eisenmann, 1979) and Sr_5Sb_3 (Martinez-Ripoll & Brauer, 1973; Hurng & Corbett, 1989; Rehr & Kauzlarich, 1994). Of these, only the latter four have been characterized by a single-crystal structure determination. Although $Sr_{11}Sb_{10}$ is not shown in the phase diagram (Scott, 1990), it has been postulated from X-ray powder patterns that it also crystallizes with the $Ho_{11}Ge_{10}$ structure type (Schmelczer, Schwarzenbach & Hulliger, 1979). $Sr_{11}Sb_{10}$ has been reported as an impurity in reactions to produce Sr_2Sb_3 (Eisenmann, 1979) but no crystal structure has been reported.

Single crystals of $Sr_{11}Sb_{10}$ have been obtained regularly during our investigations of ternary intermetallic compounds of Sr, Sb and a transition metal (Kauzlarich, 1990). In order to provide complete structural information the crystal structure was determined. The structure refinement indicated that the compound is $Sr_{11}Sb_{10}$ and that it is isotypic with $Ho_{11}Ge_{10}$ (Smith, Johnson & Tharp, 1967). Relatively few compounds crystallize with this structure type; examples include $Eu_{11}Sb_{10}$ (Schmelczer, Schwarzenbach & Hulliger, 1979), $Yb_{11}Sb_{10}$ (Clark, Simpson & Steinfink, 1970), $Ca_{11}Sb_{10}$ (Deller & Eisenmann, 1976*a*), $Ca_{11}Bi_{10}$ (Deller & Eisenmann, 1976*a*) and $Ho_{11}Ge_{8.75}Si_{1.25}$ (Schmelczer, Schwarzenbach & Hulliger, 1979). In all cases, including $Sr_{11}Sb_{10}$, the *R* value was still rather large after anisotropic refinement.

The structure type has been described in detail by Smith, Johnson & Tharp (1967) and Deller & Eisenmann (1976*a*). The structure consists of square-planar Sb rings which are connected three dimensionally by Sb_2 'dumbbells'. Such compounds can be classified as

Zintl compounds on the basis of their composition. Schmelczer, Schwarzenbach & Hulliger (1979) discussed the anomalously large values of the anisotropic displacement parameters for the $M(2)$ and $X(3)$ sites and the relatively large anisotropic displacement parameter for $X(1)$ and $M(4)$ in all of the $M_{11}X_{10}$ compounds. The extremely short $M(2)$—$X(1)$ distances and relatively short $M(4)$—$M(4)$ distances were also discussed in detail and it was noted that all these anomalies are very similar for all the $Ho_{11}Ge_{10}$ type structures. As our data are in good agreement with these observations, we will not repeat the discussion.

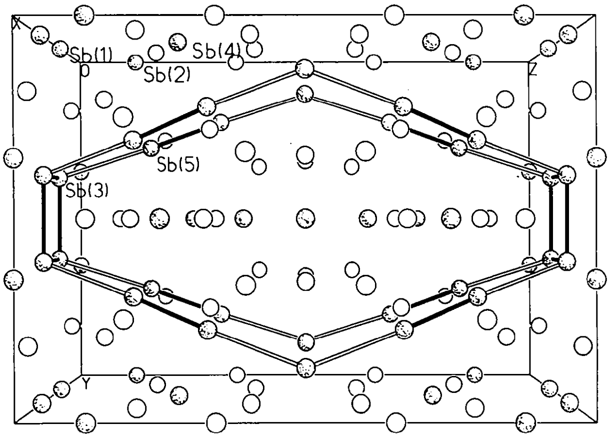

Fig. 1. View of the structure. The open circles represent Sr and the shaded circles Sb. The labels for the Sr atoms have been omitted for clarity.

Experimental

Crystals of $Sr_{11}Sb_{10}$ were obtained during reactions involving Sr, Mn and Sb. Quantitative yields of $Sr_{11}Sb_{10}$ were obtained by reacting stoichiometric amounts of the elements (Sr: Strem 99.999%; Sb: J. Matthey, 99.999%) in a niobium tube, sealed in quartz, at 727 K for one week. The product was handled in a dry box; the crystals were separated and transferred to paratone-N oil for X-ray determination.

Crystal data

$Sr_{11}Sb_{10}$
$M_r = 2181.3$
Tetragonal
*I*4/*mmm*
$a = 12.454$ (10) Å
$c = 18.066$ (11) Å
$V = 2801$ (4) Å^3
$Z = 4$
$D_x = 5.171$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6.5$–20°
$\mu = 30.178$ mm^{-1}
$T = 130$ K
Cuboid
0.2 × 0.175 × 0.15 mm
Metallic

Data collection

Syntex $P2_1$ diffractometer
ω scans

$R_{int} = 0.0942$
$\theta_{max} = 27.5°$

Absorption correction: *XABS* (Moezzi, 1987)
$T_{min} = 0.231$, $T_{max} = 0.266$
1403 measured reflections
897 independent reflections
471 observed reflections [$F > 6.0\sigma(F)$]

$h = 0 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 23$
2 standard reflections monitored every 198 reflections intensity variations: random ±2.0%

Refinement

Refinement on F
$R = 0.0799$
$wR = 0.0886$
$S = 1.31$
471 reflections
39 parameters
$w = 1/[\sigma^2(F) + 0.0044F^2]$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 8.46$ e Å^{-3}
$\Delta\rho_{min} = -7.34$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sr(1)	0	0.2517 (4)	0.1884 (30)	0.009 (1)
Sr(2)	0.1706 (7)	x	0	0.036 (2)
Sr(3)	0	0	0.3456 (9)	0.028 (4)
Sr(4)	0	0.3353 (3)	0.3961 (3)	0.010 (1)
Sb(1)	0.3504 (5)	0	0	0.026 (2)
Sb(2)	0	0	0.1232 (4)	0.007 (2)
Sb(3)	0.3808 (9)	x	0	0.086 (4)
Sb(4)	0	1/2	1/4	0.003 (1)
Sb(5)	0.2090 (3)	x	0.3208 (2)	0.020 (1)

Table 2. *Bond lengths* (Å)

Sr(1)—Sr(1)	4.434 (7)	×2	Sr(4)—Sr(4)	4.103 (9)	
Sr(1)—Sr(2)	4.138 (6)	×2	Sr(4)—Sb(1)	3.348 (6)	×2
Sr(1)—Sr(3)	4.230 (12)		Sr(4)—Sb(3)	3.602 (6)	×2
Sr(1)—Sr(4)	3.892 (8)		Sr(4)—Sb(4)	3.342 (5)	
Sr(1)—Sr(4)	4.012 (6)	×2	Sr(4)—Sb(5)	3.331 (4)	×2
Sr(1)—Sb(1)	3.619 (6)				
Sr(1)—Sb(2)	3.349 (6)		Sb(1)—Sr(1)	3.619 (6)	×2
Sr(1)—Sb(4)	3.286 (5)		Sb(1)—Sr(2)	3.087 (5)	×2
Sr(1)—Sb(5)	3.574 (6)	×2	Sb(1)—Sr(4)	3.348 (6)	×4
Sr(1)—Sb(5)	3.661 (5)	×2	Sb(1)—Sb(1)	3.727 (13)	
Sr(1)—Sr(1)	4.906 (9)	×2			
			Sb(2)—Sr(1)	3.349 (6)	×4
Sr(2)—Sr(1)	4.138 (6)	×4	Sb(2)—Sr(2)	3.739 (11)	×4
Sr(2)—Sr(2)	4.248 (17)	×2	Sb(2)—Sr(3)	4.018 (18)	
Sr(2)—Sr(4)	4.513 (9)	×4			
Sr(2)—Sb(1)	3.087 (5)	×2	Sb(3)—Sr(2)	3.703 (20)	
Sr(2)—Sb(2)	3.739 (11)	×2	Sb(3)—Sr(3)	3.491 (16)	×2
Sr(2)—Sb(3)	3.703 (20)		Sb(3)—Sr(4)	3.602 (6)	×4
Sr(2)—Sb(5)	3.870 (8)	×2	Sb(3)—Sb(3)	2.968 (22)	×2
			Sb(3)—Sb(5)	3.603 (8)	×2
Sr(3)—Sr(1)	4.230 (12)	×4			
Sr(3)—Sr(4)	4.274 (7)	×4	Sb(4)—Sr(1)	3.286 (5)	×4
Sr(3)—Sb(2)	4.018 (18)		Sb(4)—Sr(4)	3.342 (5)	×4
Sr(3)—Sb(3)	3.491 (16)	×4			
Sr(3)—Sb(5)	3.708 (6)	×4	Sb(5)—Sr(1)	3.574 (6)	×2
			Sb(5)—Sr(1)	3.661 (5)	×2
Sr(4)—Sr(1)	3.892 (8)		Sb(5)—Sr(2)	3.870 (8)	
Sr(4)—Sr(1)	4.012 (6)	×2	Sb(5)—Sr(3)	3.708 (6)	
Sr(4)—Sr(2)	4.513 (9)	×2	Sb(5)—Sr(4)	3.331 (4)	×2
Sr(4)—Sr(3)	4.274 (7)		Sb(5)—Sb(3)	3.603 (8)	×2
Sr(4)—Sr(4)	3.756 (10)		Sb(5)—Sb(5)	2.939 (9)	

The data were collected using a diffractometer equipped with a locally modified LT-1 low-temperature apparatus. The cold stream temperature is approximately 130 K. The system operates from a DEC VAX Station 3200 microcomputer. The absorption correction calculated by *XABS* is based on F_o and F_c differences.

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Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: BR1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$K_5InTe_4.KCl$

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Abstract

The structure of the title compound, pentapotassium tetratelluroindate(III)–potassium chloride (1/1), $K_5InTe_4.KCl$, contains isolated $InTe_4^{5-}$ regular tetrahedra which are separated by K^+ and Cl^- ions. This compound may be regarded as a solvated (by KCl) derivative of simple M_5InTe_4 (M = Na, K) compounds, of which the Na (orthorhombic phase) compound only has been structurally characterized to date. The Cl^- anion lying on the z axis of the hexagonal cell is octahedrally surrounded by six K^+ cations.

Comment

Single crystals of $K_5InTe_4.KCl$ have been obtained in the course of preparation of the magnetic material $Fe_5(InTe_4)_2$, discovered by Zhang, van Duyneveldt, Mydosh & O'Connor (1989). So far, single crystals of either K_5InTe_4 or $Fe_5(InTe_4)_2$ have not been obtained (Zhang, Wu & O'Connor, 1993).

The structure shown in Fig. 1 contains discrete tetrahedral $InTe_4^{5-}$ and Cl^- anions, and is closely related to that of orthorhombic *Pbcn* Na_5InTe_4 (Eisenmann, Hofmann & Zagler, 1990), of which it could be considered to be a hexagonal subcell.

Cl^- lies on the z axis and is octahedrally surrounded by six K^+ cations at distances in the range 3.110 (3)–3.194 (3) Å, in agreement with the sum of the ionic radii (1.33 for K^+ and 1.81 Å for Cl^-).

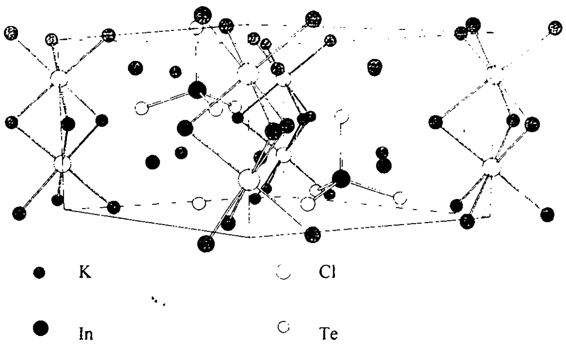

Fig. 1. Packing of $K_5InTe_4.KCl$ in the hexagonal cell.

Experimental

The synthesis was performed in several steps: preparation of K_2Te in liquid ammonia, preparation of amorphous K_5InTe_4 by the melting of appropriate quantities of K_2Te, In and Te in a tantalum crucible at 1223 K and finally reaction of K_5InTe_4 with anhydrous $FeCl_2$ in KBi molten salt.

Crystal data

$K_5InTe_4.KCl$
$M_r = 895.26$
Hexagonal
$P6_3mc$
$a = 11.288\ (7)$ Å
$c = 8.013\ (2)$ Å
$V = 884.2\ (8)$ Å^3

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 21 reflections
$\theta = 6.8$–$20.8°$
$\mu = 9.504$ mm^{-1}
$T = 298$ K