with a long one as in $K_2V_3P_4O_{17}$ (Leclaire *et al.*, 1988). Moreover it must also be emphasized that the apex corresponding to the shortest V—O bond is not free, contrary to the Mo^V phosphates, which are always described as molybdenyl compounds.

The d^1 configuration of V^{IV} suggests the possibility of particular electron transport properties due to the one-dimensional character of $[VO_3]_\infty$ chains. In the same way, the existence of intersecting tunnels indicates possible mobility of the K^+ cations, leading to ion exchange properties and ionic conductivity.

References

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.

Borel, M. M., Goreaud, M., Grandin, A., Labbe, Ph., Leclaire, A. & Raveau, B. (1991). *Eur. J. Solid State Inorg. Chem.* **28**, 93–129.

Leclaire, A., Chahboun, A., Groult, D. & Raveau, B. (1988). *J. Solid State Chem.* **77**, 170–179.

Lii, K. H. & Wang, Y. P. (1989). *J. Solid State Chem.* **80**, 127–132.

Magneli, A. (1949). *Arkiv. Chem.* **1**, 213–269.

Magneli, A. (1953). *Acta Chem. Scand.* **7**, 315–319.

Masse, R. (1974). Thesis, Univ. of Grenoble, France.

Acta Cryst. (1991). C**47**, 1141–1143

Structure of Sb_2Te

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(*Received* 1 *August* 1990; *accepted* 26 *November* 1990)

Abstract. Diantimony–tellurium, Sb_2Te, $M_r = 371·1$, trigonal, $P\bar{3}m1$, $a = 4·272$ (1), $c = 17·633$ (3) Å, $V = 278·6$ (2) Å^3, $Z = 3$, $D_x = 6·63$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·7107 Å, $\mu = 22·11$ mm^{-1}, $F(000) = 462$, $T = 294$ K, final $R = 0·035$ for 245 independent observed reflections. The structure of Sb_2Te consists of nine layers stacked along the c axis and presents the combination of five-layer stacks of Sb_2Te_3 and two-layer stacks of Sb_2.

Introduction. The layered compounds of the Sb—Te system, such as Sb_2Te_3, SbTe and Sb_2Te, are important for industrial applications owing to the strong anisotropy in their electronic properties. These compounds, as well as the phases of the Bi–Te and Bi–Se systems, have trigonal or rhombohedral symmetries with a similar a parameter while the c parameter depends on the number of layers, which is itself connected to the chemical composition (Imamov & Semiletov, 1971). The crystal structures of Sb_2Te_3 (Semiletov, 1956; Anderson & Krause, 1974) and SbTe (Stasova & Karpinskii, 1967) are already known. In this paper, the results of the Sb_2Te crystal structure determination are reported.

Experimental. Single crystals of Sb_2Te were obtained by slow cooling a molten stoichiometric (2:1) mixture of Sb and Te. A platelet-like crystal (0·38 × 0·07 × 0·02 mm) with well defined faces was chosen. Enraf–Nonius CAD-4 diffractometer; lattice parameters determined from 25 reflections having $8·87° \le \theta \le 19·87°$, θ–2θ scan technique; $0·049 \le (\sin\theta)/\lambda \le$

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Table 1. *Fractional coordinates and equivalent B's for atoms, with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (ab\cos\gamma)\beta_{12} + (ac\cos\beta)\beta_{13} + (bc\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}(Å^2)
Te(1)	$\frac{1}{3}$	$\frac{2}{3}$	0·20971 (8)	0·43 (2)
Te(2)	0	0	0	0·32 (4)
Sb(1)	$\frac{1}{3}$	$\frac{2}{3}$	0·8865 (1)	1·47 (4)
Sb(2)	$\frac{1}{3}$	$\frac{2}{3}$	0·56637 (9)	0·41 (3)
Sb(3)	0	0	0·34690 (9)	0·49 (3)

Table 2. *Selected bond distances* (Å) (*for* Sb_2Te, SbTe, Sb_2Te_3 *and* Sb) *and angles* (°) (*for* Sb_2Te *only*), *with e.s.d.'s in parentheses*

Sb_2Te		SbTe		Sb	
Te(1)—Sb(1$^{\rm i}$)	2·993 (2)	Sb(1)—Te(1)	2·98	Sb—Sb	2·90
Te(1)—Sb(3)	3·455 (2)	Sb(3)—Te(3)	3·48	Sb—Sb	3·37
Te(2)—Sb(1$^{\rm i}$)	3·176 (2)	Sb(1)—Te(2)	3·18		
Sb(2)—Sb(3$^{\rm i}$)	2·902 (1)	Sb(3)—Sb(3)	2·92		
Sb(2)—Sb(2$^{\rm i}$)	3·400 (2)				

Sb_2Te_3	
Sb—Te(1)	2·979
Sb—Te(2)	3·168

Sb(1$^{\rm i}$)—Te(1)—Sb(1$^{\rm ii}$)	91·05 (5)	Sb(2$^{\rm i}$)—Sb(2)—Sb(3$^{\rm i}$)	93·13 (3)
Sb(1$^{\rm i}$)—Te(1)—Sb(3)	95·90 (3)	Sb(3$^{\rm vi}$)—Sb(2)—Sb(3$^{\rm i}$)	94·79 (4)
Sb(3)—Te(1)—Sb(3$^{\rm iii}$)	76·38 (3)	Sb(2$^{\rm i}$)—Sb(2)—Sb(2$^{\rm ii}$)	77·83 (4)
Sb(1$^{\rm vi}$)—Te(2)—Sb(1$^{\rm i}$)	84·51 (3)	Te(1$^{\rm vii}$)—Sb(3)—Te(1)	76·38 (4)
Te(2$^{\rm iv}$)—Sb(1)—Te(2$^{\rm v}$)	84·51 (5)	Sb(2$^{\rm i}$)—Sb(3)—Sb(2$^{\rm ii}$)	94·79 (5)
Te(1$^{\rm i}$)—Sb(1)—Te(1$^{\rm ii}$)	91·05 (6)	Te(1$^{\rm vii}$)—Sb(3)—Sb(2$^{\rm i}$)	93·76 (3)
Te(1$^{\rm i}$)—Sb(1)—Te(2$^{\rm iv}$)	92·13 (2)		

Symmetry code: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, 1+y, z$; (iv) $x, y, 1+z$; (v) $x, 1+y, 1+z$; (vi) $-x, -y, 1-z$; (vii) $-1+x, -1+y, z$.

0·680 Å^{-1}, $0 \le h \le 5$, $0 \le k \le 5$, $0 \le l \le 23$. No significant deterioration in intensity, three standard reflections, $\overline{1}\overline{1}0$, $01\overline{8}$ and $2\overline{4}0$, with average $\sigma(I)/I$ = 0·0023; empirical correction for absorption: *DIFABS* (Walker & Stuart, 1983), max. and min. values of the coefficient of correction 1·05 and 0·94. 344 independent reflections, 99 unobserved reflections [$I < 3\sigma(I)$]. Direct methods, program *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), full-matrix refinements (on F), refined parameters x, y, z and β_{ij}, $R = 0·035$, wR = 0·034, $w = 1$ for all reflections, $S = 3·35$, max. shift to e.s.d. ratio $(\Delta/\sigma)_{\max} = 0·02$, $|\Delta\rho|_{\max} = 1·6$ (4) e Å^{-3}. Atomic diffusion factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99 and 149). Computer programs of *SDP* system (B. A. Frenz & Associates, Inc., 1982), DEC PDP 11/44 computer. Atomic parameters are given in Table 1, bond lengths and angles in Table 2.* Fig. 1 shows a stereoview of the Sb_2Te crystal structure realized by *MOLDRAW* (Cense, 1989).

Results. Since Sb and Te X-ray scattering factors are very similar, it is quite difficult to deduce directly their respective atomic positions from the diffraction data. However, some arguments may be used to provide one with a guideline to the more likely occupancy for each site.

First, the unit cell contains nine layers stacked along the c axis. There is one type of atom in each layer and a unique packing mode whatever the layer, with respect to the Wyckoff positions of the space group and to the chemical composition, which was controlled by neutron activation analysis.

Thus, four types of stacking are possible.

(1) Te- Sb- Te- Sb- Sb- Sb- Sb- Te- Sb- Te;
(2) Te- Sb- Sb- Te- Sb- Sb- Te- Sb- Sb- Te;
(3) Te- Sb- Sb- Sb- Te- Te- Sb- Sb- Sb- Te;
(4) Te- Te- Sb- Sb- Sb- Sb- Sb- Sb- Te- Te.

Secondly, all analogous compounds with known crystal structure, such as BiSe (Stasova & Karpinskii, 1967), BiTe and Bi_4Te_3 (Yamana, Kihara & Matsumoto, 1979) are found to be constituted by alternate layers of Bi_2Te_3 (or Bi_2Se_3) and Bi_2, *i.e.* five-layered stacks of (TeBiTeBiTe) and two-layered stacks of (BiBi). For instance, $BiTe = 2Bi_2Te_3 + Bi_2$ and $Bi_4Te_3 = Bi_2Te_3 + Bi_2$. Similarly, the structure of Sb_2Te may be described as the sequence of one five-layered stack of Sb_2Te_3 and two two-layered stacks of Sb_2. Moreover, this arrangement, which is the first possible one suggested above, leads to a slightly better R factor (0·035) than that of the three other hypotheses ($R = 0·037$ for any one).

The coordination polyhedra are distorted octahedra. Te(1) is surrounded by three Sb(1) neighbours at

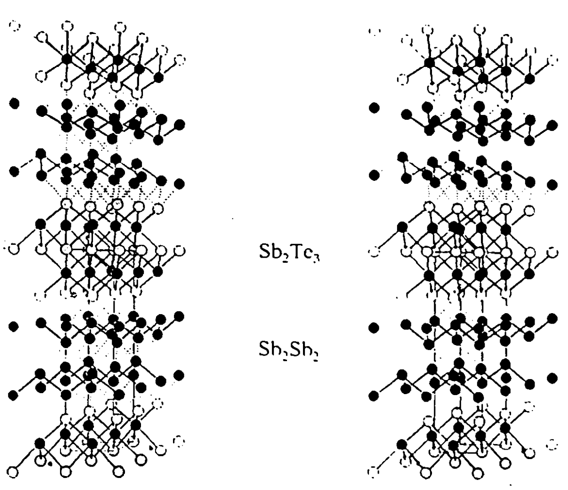

Fig. 1. Stereoview of the crystal structure of Sb_2Te.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53785 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

2·993 (2) Å and by three Sb(3) at 3·445 (2) Å, as in SbTe. Te(2) displays six equidistant Sb(1) neighbours at 3·176 (2) Å, as in Sb_2Te_3. Sb(1) is surrounded by six Te atoms, 3 Te(1) at 2·993 (2) Å and 3 Te(2) at 3·176 (2) Å, as in SbTe and Sb_2Te_3. The Sb(2) surrounding, which consists of six Sb atoms [3 Sb(3) at 2·902 (1) and 3 Sb(2) at 3·400 (2) Å], is like that in α-antimony. Sb(3) differs slightly from Sb(2) because it is bonded to 3 Te(1) at 3·455 (2) Å and 3 Sb(2) at 2·902 (1) Å. Bond length values in SbTe, Sb_2Te_3 and α-Sb are given for comparison in Table 2.

The layered structure obtained for Sb_2Te exhibits some similarities with the known structures of SbTe and Sb_2Te_3. Sb_2Te is a stacking of Sb_2Te_3 and Sb_2 in the ratio 1:2, and SbTe is known to consist of Sb_2Te_3–Sb_2–Te_3Sb_2. Thus, it may be now concluded that all known Sb–Te compounds are made from Sb_2Te_3 (five layers) as the basic sublattice component.

The authors wish to thank Mr M. Fedoroff, CECM, Vitry, for the neutron activation analysis.

References

ANDERSON, T. A. & KRAUSE, H. B. (1974). *Acta Cryst.* **B30**, 1307–1310.

B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.

CENSE, J.-M. (1989). *Tetrahedron Comput. Methodol.* **2**, 65–71.

IMAMOV, P. M. & SEMILETOV, S. A. (1971). *Sov. Phys.-Crystallogr.* **15**, 845–850.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

SEMILETOV, S. A. (1956). *Kristallografiya*, **1**, 403–406.

STASOVA, M. M. & KARPINSKII, O. G. (1967). *Zh. Strukt. Khim.* **8**, 654–661.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 159–166.

YAMANA, K., KIHARA, K. & MATSUMOTO, T. (1979). *Acta Cryst.* **B35**, 147–149.

Acta Cryst. (1991). **C47**, 1143–1145

Redetermination of the Structure of the 80 K Superconductor $YBa_2Cu_4O_8$ by Time-of-Flight Neutron Powder Diffraction

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(*Received* 6 *July* 1990; *accepted* 9 *October* 1990)

Abstract. $M_r = 745·77$, orthorhombic, *Ammm*, $a = 3·8402\,(1)$, $b = 3·8708\,(1)$, $c = 27·2309\,(3)$ Å, $V = 404·8$ Å^3, $D_x = 6·119$ g cm^{-3}, $Z = 2$, $T = 295$ K, 912 reflections included, $R_{wp} = 5·62\%$, $R_{\rm exp} = 3·34\%$. Rietveld analysis of high-resolution time-of-flight powder neutron diffraction data has been used to redetermine the structure of the 80 K superconductor $YBa_2Cu_4O_8$ (the '124 phase'). The essential features of the structure previously reported have been confirmed. This study improves upon the precision of the previously reported structural parameters, obtained by powder neutron diffraction. The compound is stoichiometric, with no occupancy of the O(5) inter-chain site.

0108-2701/91/061143-03$03.00