Profile data from ω/θ scans
Absorption correction: none
4171 measured reflections
2471 independent reflections
1922 reflections with $I > 2\sigma(I)$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 13$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity decay: 4.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.090$
$S = 1.018$
2471 reflections
101 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.988$ e Å^{-3}
$\Delta\rho_{min} = -1.928$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0288 (16)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sr1—O3^{i}	2.533 (2)	Fe1—O4	2.113 (2)
Sr1—O3	2.559 (2)	Fe1—O7	2.199 (2)
Sr1—O7	2.566 (2)	P1—O3^{vi}	1.513 (2)
Sr1—O2^{ii}	2.637 (2)	P1—O1^{vii}	1.515 (2)
Sr1—O6^{iii}	2.648 (2)	P1—O7	1.529 (2)
Sr1—O1^{iv}	2.657 (3)	P1—O5^{iii}	1.605 (2)
Sr1—O4	2.722 (2)	P2—O4	1.523 (2)
Sr1—O7^{i}	2.776 (2)	P2—O6	1.524 (2)
Fe1—O1	1.988 (2)	P2—O2^{viii}	1.536 (2)
Fe1—O2^{v}	2.049 (2)	P2—O5	1.604 (2)
Fe1—O6^{vi}	2.113 (2)		
O3^{vi}—P1—O1^{vii}	113.78 (13)	O4—P2—O2^{viii}	111.98 (12)
O3^{vi}—P1—O7	112.94 (13)	O6—P2—O2^{viii}	111.02 (12)
O1^{vii}—P1—O7	108.35 (13)	O4—P2—O5	103.91 (12)
O3^{vi}—P1—O5^{iii}	107.80 (13)	O6—P2—O5	108.02 (12)
O1^{vii}—P1—O5^{iii}	105.71 (12)	O2^{viii}—P2—O5	106.41 (12)
O7—P1—O5^{iii}	107.86 (12)	P2—O5—P1^{ix}	127.39 (14)
O4—P2—O6	114.82 (12)		

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (iv) $x - 1, y, z$; (v) $x, 1 + y, z$; (vi) $1 + x, y, z$; (vii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (viii) $1 - x, 1 - y, -z$; (ix) $x - \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$.

Absorption corrections were attempted (Gaussian method), but they did not improve the results of the refinement. The rather complex shape of the crystal makes a good numerical correction difficult to obtain. Thus, the results are based on observed structure factors with no absorption correction. Only about half the reflections in the *hkl* range indicated could be measured, but all independent reflections were measured at least once.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *DIAMOND* (Bergerhoff, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1225). Services for accessing these data are described at the back of the journal.

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Ta_5Sb_4

Sung Kwon Kang† and Gordon J. Miller

Department of Chemistry, Iowa State University, Ames, IA 50011, USA. E-mail: gmiller@iastate.edu

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Abstract

The title compound, pentatantalum tetraantimonide, was obtained as a by-product of the reaction between Ba and Sb mixed in a 1:2 molar ratio using tantalum as a reaction container. This compound adopts the Ti_5Te_4 structure type, which consists of chains of vertex-sharing Ta_6Sb_8 face-capped octahedral clusters. The resulting formulation is $Ta_4Ta_{2/2}Sb_{8/2}$. Distance ranges are: Ta—Ta 2.930 (1)–3.301 (1) and Ta—Sb 2.775 (2)–3.074 (1) Å; there are no bonding Sb···Sb distances. Closer inspection reveals that the Ta substructure also resembles chains cut out from *bcc* packing.

Comment

During investigations on the binary barium–antimony system using sealed tantalum containers for their preparation, the compound Ta_5Sb_4 was obtained as a side product. Previously, this compound was prepared by direct reaction between the elements and characterized by X-ray powder diffraction (Boller & Nowotny, 1964; Furuseth *et al.*, 1965). Ta_5Sb_4 is isostructural with tetragonal Ti_5Te_4 (Grønvold *et al.*, 1961), which adopts the space group *I*4/*m*. Unit-cell parameters for Ta_5Sb_4 have been determined to be $a = 10.2357 (14)$ and $c = 3.5425 (7)$ Å. The shortest interatomic distances

† Permanent address: Department of Chemistry, Chungnam National University, Taejon 305-764, Korea.

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are Ta—Ta 2.930 (1) and Ta—Sb 2.775 (2) Å. These distances are close to the shortest distance found in elemental tantalum (*bcc*, 2.86 Å; Mueller, 1977) and the sum of the metallic radii of Ta and Sb (Barrett *et al.*, 1963), which is 2.88 Å. The closest Sb···Sb distance in this compound is 3.525 Å, which indicates that there is no bonding interaction between Sb atoms, as this is much longer than twice the metallic radius of Sb.

The Ti_5Te_4 structure has been discussed with respect to the condensation of Ti_6Te_8 face-capped octahedral clusters *via* opposite vertices, which leads to a quasi-infinite chain of composition $Ti_{2/2}Ti_4Te_{8/2} = Ti_5Te_4$ (Simon, 1992). In Ta_5Sb_4, as in the other examples, the octahedra are compressed by approximately 22%; the eight octahedral edges involving the apex atoms are 2.930 (1) Å long, whereas the four edges in the waist are 3.297 (1) Å long. This distance, within standard error, equals the shortest Ta—Ta distance found between adjacent chains of 3.301 (1) Å. The apex Ta atoms are bonded to four Sb atoms arranged in a square-planar structure at a distance of 3.074 (1) Å, the other Ta atoms being coordinated by a distorted square pyramid of Sb atoms; in the equatorial plane, the Ta—Sb distances are 2.790 (1) and 2.827 (1) Å, while the remaining distance to an adjacent column is 2.775 (2) Å. This strong donor–acceptor interaction between Ta atoms in the waist with Sb atoms from neighboring chains in the axial position of the local square pyramid may explain the distortion of the octahedra (Simon, 1992), but the alternative explanation uses the assumption that the shared apex Ta atoms prefer a *bcc*-type environment with eight nearest-neighbor Ta atoms (Chen & Franzen, 1972).

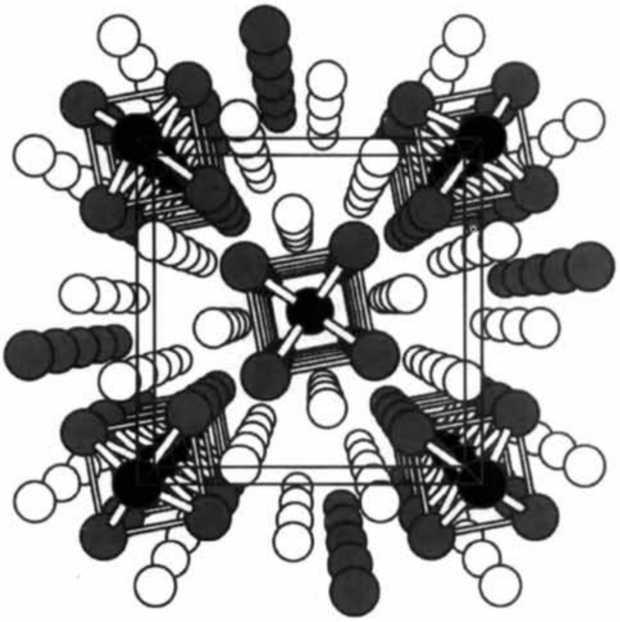

Fig. 1. An (001) perspective projection of Ta_5Sb_4. Light circles are Sb atoms and dark circles are Ta atoms.

As indicated by Simon (1992), this structure type occurs for a wide range of electron counts from 44 (Ti_5Te_4) to 50 valence electrons (Mo_5As_4). Ta_5Sb_4 exhibits 45 valence electrons. According to band-structure calculations of M_5X_4 chains (Kumar & Heine, 1984*a*,*b*), minima in their electronic energy densities of states occur for 44–45 and 49–50 valence electrons. Ta_5Sb_4 is in good agreement with the lower values.

Experimental

The title compound was obtained as a by-product when elemental Ba [rod, Aesar (99.99%)] and Sb [powder, 100 mesh, Aesar (99.999%)] in a 1:2 molar ratio were loaded into a tantalum tube (Noble-Met. Ltd, > 99.85%, 0.375 OD) in an argon-filled glove-box, sealed in an arc melter under argon, and then heated to 1173 K for 3 d in a fused-silica jacket. The reaction container was slowly cooled to 673 K at 10 K h^{-1} and then quenched to room temperature. When the tantalum tube was opened in the argon-filled glove-box, thin grey needle-shaped crystals of the title compound were found in the product. Suitable single crystals were mounted in 0.2 mm thin-walled capillaries for subsequent diffraction experiments.

Crystal data

Ta_5Sb_4	Mo $K\alpha$ radiation
$M_r = 1391.75$	$\lambda = 0.71073$ Å
Tetragonal	Cell parameters from 39 reflections
$I4/m$	$\theta = 7.28$–$14.66°$
$a = 10.2357 (14)$ Å	$\mu = 87.556$ mm^{-1}
$c = 3.5425 (7)$ Å	$T = 293 (2)$ K
$V = 371.15 (10)$ $Å^3$	Needle
$Z = 2$	$0.10 \times 0.03 \times 0.03$ mm
$D_x = 12.454$ Mg m^{-3}	Grey
D_m not measured	

Data collection

Rigaku AFC-6*R* diffractometer	218 reflections with $I > 2\sigma(I)$
$2\theta-\omega$ scans	$R_{int} = 0.096$
Absorption correction: ψ scan (North *et al.*, 1968)	$\theta_{max} = 30.05°$
$T_{min} = 0.138$, $T_{max} = 0.199$	$h = -14 \rightarrow 14$
655 measured reflections	$k = 0 \rightarrow 14$
314 independent reflections	$l = 0 \rightarrow 4$
	3 standard reflections every 150 reflections intensity decay: 0.4%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 4.49$ e $Å^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$\Delta\rho_{min} = -4.60$ e $Å^{-3}$
$wR(F^2) = 0.065$	Extinction correction: *SHELXL97*
$S = 1.062$	Extinction coefficient: 0.00108 (8)
314 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
16 parameters	
$w = 1/[\sigma^2(F_o^2) + 18.3209P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max} < 0.001$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Ta1	0	0	0	0.0050 (4)
Ta2	0.37078 (8)	0.31212 (8)	0	0.0066 (3)
Sb3	0.29588 (13)	0.05161 (13)	0	0.0066 (4)

The crystal structure was solved by direct methods (*SHELXS*97; Sheldrick, 1990). The space groups *I*4, *I*$\bar{4}$ and *I*4/*m* were allowed based upon the observed systematic absences. The space group *I*4/*m* was selected for initial refinements, and this group was confirmed by comparing the refinement results using the other two groups, which were identical with *I*4/*m* within three s.u.'s. The Ta and Sb atoms were readily located from the *E* map and refined with anisotropic displacement parameters. The reflection (110) with $2\theta = 5.62°$ was omitted from the refinement due to the close proximity and possible interference from the beam stop of the X-ray diffractometer. The largest residuals in the final difference map were 4.49 e Å^{-3} at a distance of 1.82 Å from Ta1 and −4.60 e Å^{-3} on Ta1.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1990). Program(s) used to solve structure: *SHELXS*97. Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1226). Services for accessing these data are described at the back of the journal.

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Strontium tetramolybdate dihydrate, $SrMo_4O_{13}\cdot 2H_2O$

William T. A. Harrison†

Department of Chemistry, University of Western Australia, Nedlands WA 6907, Australia. E-mail: wtah@chem.uwa.edu.au

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Abstract

$SrMo_4O_{13}\cdot 2H_2O$ contains infinite sheets of distorted MoO_6 and MoO_5 moieties sharing edges and vertices. Inter-layer nine-coordinate Sr^{2+} cations [d_{av}(Sr—O) = 2.678 (3) Å] and water molecules complete the structure, which is isostructural with that of $BaMo_4O_{13}\cdot 2H_2O$.

Comment

$SrMo_4O_{13}\cdot 2H_2O$ (Fig. 1) is confirmed to be isostructural with $BaMo_4O_{13}\cdot 2H_2O$ (Harrison *et al.*, 1995). The polyhedral connectivity of the Mo/O groups results in buckled anionic sheets of stoichiometry $[Mo_4O_{13}]^{2-}$, which propagate normal to [100]. Both vertex-sharing and edge-sharing of the MoO_6 and MoO_5 groups occurs in this phase, which has essentially the same sheet structure as $BaMo_4O_{13}\cdot 2H_2O$.

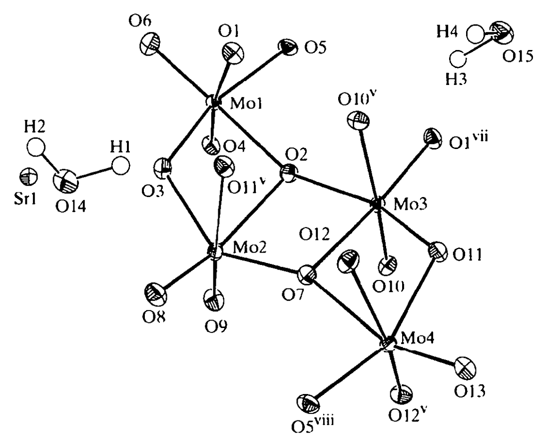

Fig. 1. Fragment of the $SrMo_4O_{13}\cdot 2H_2O$ structure, drawn with 50% displacement ellipsoids. The very long Mo2—O11^v bond is indicated by a thin line (see text). Symmetry codes are as in Table 1.

The Sr1 atom is nine-coordinate to O atoms [d_{av}(Sr1—O) = 2.678 (3) Å] in irregular coordination (Fig. 2). Four of these neighbours are the O atoms of

† Present address: Department of Chemistry, University of Aberdeen, Aberdeen AB24 3UE, Scotland.