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Redetermination of the Structures of $CuTaS_3$ and Nb_2Se_9

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Abstract. Copper tantalum trisulfide, $CuTaS_3$: $M_r = 340{\cdot}69$, orthorhombic, $Pnma$, $a = 9{\cdot}488\,(5)$, $b = 3{\cdot}486\,(2)$, $c = 11{\cdot}754\,(5)$ Å, $V = 388{\cdot}7$ Å^3, $Z = 4$, $D_x = 5{\cdot}83$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}7093$ Å, $\mu = 346{\cdot}4$ cm^{-1}, $F(000) = 598$ (with anomalous dispersion), $T = 120$ K, $R(\text{on } F_o^2) = 0{\cdot}091$ for 984 averaged reflections (including those having $F_o^2 < 0$). The general features of the structure are as reported by Crevecoeur & Romers [*Proc. K. Ned. Akad. Wet. Ser. B* (1964), **67**, 289–291]. The structure resembles a honeycomb with large empty channels along [010]. The honeycomb network is established *via* the interconnection of slabs with the four-atom repeat Cu—Ta—Ta—Cu. The Cu—Ta distance is short [2·781 (2) Å], suggesting a Cu—Ta interaction. Diniobium nonaselenide, Nb_2Se_9: $M_r = 896{\cdot}45$, triclinic, $P\bar{1}$, $a = 8{\cdot}160\,(2)$, $b = 8{\cdot}284\,(2)$, $c = 13{\cdot}060\,(3)$ Å, $\alpha = 121{\cdot}22\,(2)$, $\beta = 124{\cdot}00\,(2)$, $\gamma = 91{\cdot}74\,(2)^\circ$, $V = 558{\cdot}3$ Å^3, $Z = 2$, $D_x = 5{\cdot}33$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}7093$ Å, $\mu = 309{\cdot}5$ cm^{-1}, $F(000) = 765$ (with anomalous dispersion), $T = 120$ K, $R(\text{on } F_o^2) = 0{\cdot}093$ for 3250 reflections (including those having $F_o^2 < 0$). The structure is comprised of one-dimensional chains that run along the *c* axis, as reported by Meerschaut, Guemas, Berger & Rouxel [*Acta Cryst.* (1979), B**35**, 1747–1750]. The chains contain Nb atoms in bicapped trigonal prisms of Se atoms. The chains contain Nb—Nb bonds [$d = 2{\cdot}895\,(2)$ Å] as well as Se—Se bonds. In addition to Se pairs, the structure also contains an Se_5 unit.

Introduction. During the search for new ternary chalcogenides we have synthesized many known binary and ternary compounds. The structures and physical properties of most of these compounds were well known. But among these compounds were $CuTaS_3$ and Nb_2Se_9 whose structures were known with limited accuracy owing either to poor crystal quality or to the method of analysis. For $CuTaS_3$ the data were obtained from visually estimated intensities from Weissenberg photographs (Crevecoeur & Romers, 1964). The intensity data for Nb_2Se_9 were obtained from a twinned crystal with Weissenberg multiple-film techniques

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(Meerschaut, Guemas, Berger & Rouxel, 1979). These compounds possess structural features that may aid in the understanding of other ternary chalcogenides. In particular, the structure of Nb_2Se_9 contains a five-membered polyselenide ion and $CuTaS_3$ possesses a short Cu—Ta distance [2·781 (2) Å] similar to the Ni—Ta distance of 2·747 (7) Å in Ta_2NiS_5 (Sunshine & Ibers, 1985). Because of the significance of these two compounds the redetermination of their structures was undertaken.

Experimental. The compound $CuTaS_3$ resulted from grinding a sample of composition Ta_2NiS_8 through a brass sieve and reheating the sample with I_2 (~1% by weight) at 950K for 7d. Apparently the brass sieve serves as the source of the Cu metal, as it did in the synthesis of $Ta_2Cu_{0.8}S_6$ (Squattrito, Swepston & Ibers, 1987). The Nb_2Se_9 crystals were prepared by heating Ni, Nb and Se powders in a 3:2:8 ratio at 900 K for 17 d. In both reactions only a few crystals were formed and the remaining products were not characterized. Qualitative analysis of the crystals with an EDAX-equipped Cambridge S-4 scanning electron microscope indicated the elements present in the crystals. As systematic syntheses of these materials have been previously reported no attempt was made to improve the yields of the reactions (Crevecoeur & Romers, 1964; Fedorov, Mischenko & Ustimenko, 1984).

For $CuTaS_3$ the crystal used for data collection was a pentagonal needle capped by {010} and bound by $(10\bar{1})$, $(\bar{1}01)$, $(00\bar{1})$, $(\bar{1}0\bar{1})$, and (201); crystal volume $6{\cdot}7\times10^{-5}$ mm³. Unit-cell parameters were derived from a least-squares analysis of 25 reflections, in the range $15<\theta<20°$, automatically centered on an Enraf–Nonius CAD-4 X-ray diffractometer. Intensity data were collected with the $\omega/2\theta$ scan technique; intensities of six standard reflections monitored throughout data collection were constant within intensity statistics. A total of 2511 reflections, measured to $(\sin\theta_{\max})/\lambda = 0{\cdot}7884$ Å^{-1} in the index range $-14\le h\le 14$, $-5\le k\le 5$, $-18\le l\le 18$, were corrected for absorption with the use of the analytical method (de Meulenaer & Tompa, 1965); min. and max. transmission factors were 0·62 and 0·74. When those reflections having $F_o^2>3\sigma(F_o^2)$ were averaged in space group *Pnma* the *R* index for averaging $(\sum|\langle F^2\rangle - F^2|/\sum F^2)$ was 0·092 (590 unique data). The corresponding index for averaging in $Pn2_1a$ was 0·082 (927 unique data). As there is no strong support for the non-centrosymmetric space group, all the F_o^2 values were averaged in space group *Pnma* to yield 984 unique reflections.

For Nb_2Se_9 the crystal used for data collection was a hexagonal needle capped by $\{10\bar{1}\}$ and bound by $\{\bar{1}00\}$, $\{0\bar{1}0\}$, and $\{\bar{1}10\}$; crystal volume $7{\cdot}5\times10^{-4}$ mm³. Unit-cell parameters were derived as above on 25 reflections in the range $15<\theta<18°$. Intensity data were collected as for $CuTaS_3$. A total of 6498 reflections, measured to $(\sin\theta_{\max})/\lambda = 0{\cdot}7049$ Å^{-1} in the index range $-11\le h\le 11$, $-11\le k\le 11$, $-17\le l\le 17$, were corrected for absorption (de Meulenaer & Tompa, 1965); min. and max. transmission factors were 0·30 and 0·49. The F_o^2 values were averaged to yield 3249 unique data $[R_{\rm int}(F^2)=0{\cdot}045]$.

The structures were refined from the previous models (Crevecoeur & Romers, 1964; Meerschaut, Guemas, Berger & Rouxel, 1979). Scattering factors and anomalous-dispersion terms were taken from the usual sources (Cromer & Waber, 1974; Cromer, 1974). All calculations were performed on a Harris 800 computer with methods and programs standard for this laboratory (Waters & Ibers, 1977). For $CuTaS_3$ the final refinement on F_o^2, based on 31 variables and 984 observations, resulted in an *R* index on F^2 of 0·091 with Δ/σ not exceeding 0·05. Other residuals are $wR(F^2)=0{\cdot}109$, where the weights are derived from counting statistics and a value of *p* of 0·04, and $R(F)$ $[F_o^2>3\sigma(F_o^2)]=0{\cdot}042$. Error in an observation of unit weight is 0·98. For Nb_2Se_9 the final refinement on F_o^2, based on 101 variables and 3249 observations, resulted in an *R* index on F^2 of 0·093 and an isotropic extinction parameter (Zachariasen, 1968) of $6{\cdot}0\,(3)\times10^{-6}$, with Δ/σ not exceeding 0·30. Other residuals are $wR(F^2)=0{\cdot}141$, where the weights were determined as above, and $R(F)$ $[F_o^2>3\sigma(F_o^2)]=0{\cdot}054$. Error in an observation of unit weight is 1·38. Table 1 lists fractional coordinates and equivalent isotropic thermal parameters for the two structures.*

Discussion. The general features of the structures of $CuTaS_3$ and Nb_2Se_9 are the same as previously reported (Crevecoeur & Romers, 1964; Meerschaut, Guemas, Berger & Rouxel, 1979). Selected bond distances and angles for the two compounds are given in Table 2 ($CuTaS_3$) and Table 3 (Nb_2Se_9).

A drawing of the structure of $CuTaS_3$ is presented in Fig. 1. This three-dimensional structure resembles a honeycomb, as large empty channels run parallel to the *b* axis. The open nature of the structure resembles that in $Nb_2Pd_3Se_8$ (Keszler & Ibers, 1984). The honeycomb network in $CuTaS_3$ is established through the interconnection of slabs with the four-atom repeat Cu—Ta—Ta—Cu. The Cu—Ta distance in this repeat is short [2·781 (2) Å], suggestive of some Cu—Ta interaction. This distance is slightly longer than the 2·74 (1) Å originally reported (Crevecoeur & Romers, 1964) and closer to the value of 2·804 (7) Å found in the related compound $Ta_2Cu_{0.8}S_6$ (Squattrito, Swepston & Ibers,

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* Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43654 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1987). A similar M—Ta interaction is seen in Ta_2NiQ_5 (Q = S, Se) (Sunshine & Ibers, 1985).

Four-probe single-crystal conductivity measurements along the needle axis, b, attempted with the use of previously described procedures (Phillips, Anderson, Schramm & Hoffman, 1979), indicate a conductivity less than the sensitivity of the apparatus ($<10^{-3}\ \Omega^{-1}\ cm^{-1}$). The lack of close S—S interactions [$S—S_{min} = 3{\cdot}254\ (7)$ Å] and the insulating behavior are consistent with the valence description $Cu^{1+}Ta^{5+}3S^{2-}$. This description however, would not predict the observed Cu—Ta interaction as Cu^{1+} (d^{10}) and Ta^{5+} (d^0) do not possess lone electrons for bonding. The description $Cu^{2+}Ta^{4+}3S^{2-}$ with strong Cu—Ta interactions would also explain the observed insulating behavior. This is similar to the model proposed for the Ta_2NiQ_5 (Q = S, Se) system (Sunshine & Ibers, 1985).

Table 1. *Positional parameters and equivalent isotropic thermal parameters for* $CuTaS_3$ *and* Nb_2Se_9

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

	x	y	z	B_{eq}(Å^2)
$CuTaS_3$				
Ta	0·107835 (68)	$\frac{1}{4}$†	0·130232 (54)	0·40 (1)
Cu	0·27219 (20)	$\frac{1}{4}$	0·78709 (17)	0·61 (4)
S(1)	0·22080 (41)	$\frac{1}{4}$	0·59643 (34)	0·69 (8)
S(2)	0·08083 (39)	$\frac{1}{4}$	0·90295 (33)	0·56 (8)
S(3)	0·07673 (39)	$\frac{1}{4}$	0·32272 (33)	0·58 (8)
Nb_2Se_9				
Nb(1)	0·04875 (18)	0·03806 (18)	0·13768 (13)	0·54 (6)
Nb(2)	0·98173 (18)	0·03435 (18)	0·39680 (13)	0·52 (6)
Se(1)	0·44883 (21)	0·22346 (21)	0·41794 (16)	0·83 (7)
Se(2)	0·36024 (21)	0·21163 (21)	0·68955 (15)	0·78 (7)
Se(3)	0·30902 (20)	0·12676 (21)	0·91555 (15)	0·68 (7)
Se(4)	0·18642 (21)	0·34186 (20)	0·14806 (15)	0·78 (7)
Se(5)	0·14762 (22)	0·33849 (20)	0·41327 (16)	0·82 (7)
Se(6)	0·10474 (22)	0·33676 (20)	0·67616 (16)	0·84 (7)
Se(7)	0·80922 (21)	0·18178 (21)	−0·00231 (16)	0·78 (7)
Se(8)	0·78935 (21)	0·10193 (21)	0·69431 (15)	0·69 (7)
Se(9)	0·72876 (21)	0·18866 (21)	0·27957 (16)	0·79 (7)

† The five independent atoms of $CuTaS_3$ are in Wyckoff position 4(c) with site symmetry m.

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *for* $CuTaS_3$

Ta—S(3)	2·282 (4)	S(3)—Ta—S(1)	104·5 (1)
Ta—2S(1)	2·417 (3)	S(3)—Ta—S(2)	93·5 (1)
Ta—2S(2)	2·529 (3)	S(3)—Ta—S(2)	167·1 (1)
Ta—S(2)	2·683 (4)	S(1)—Ta—S(1)	92·3 (1)
Ta—Cu	2·781 (2)	S(1)—Ta—S(2)	87·3 (1)
Ta—Ta	3·486 (2)	S(2)—Ta—S(2)	77·2 (1)
Cu—S(2)	2·270 (4)	S(2)—Cu—S(1)	114·6 (2)
Cu—S(1)	2·294 (5)	S(2)—Cu—S(3)	113·0 (1)
Cu—2S(3)	2·295 (3)	S(1)—Cu—S(3)	108·1 (1)
Cu—Cu	3·486 (2)		

Table 3. *Selected bond distances* (Å) *and bond angles* (°) *for* Nb_2Se_9

Nb(1)—Nb(2)	3·732 (2)	Se(4)—Se(7)	2·319 (2)
Nb(1)—Nb(1)	2·895 (2)	Se(2)—Se(6)	2·316 (2)
Nb(2)—Nb(2)	2·895 (2)	Se(8)—Se(1)	2·365 (2)
Nb(1)—Se(4)	2·630 (2)	Se(1)—Se(5)	2·643 (2)
Nb(1)—Se(5)	2·673 (2)	Se(5)—Se(9)	2·663 (2)
Nb(1)—Se(7)	2·642 (2)	Se(9)—Se(3)	2·364 (2)
Nb(1)—Se(4)	2·639 (2)		
Nb(1)—Se(7)	2·621 (2)	Se(1)—Nb(1)—Se(4)	87·54 (6)
Nb(1)—Se(8)	2·719 (2)	Se(1)—Nb(1)—Se(4)	132·33 (7)
Nb(1)—Se(1)	2·630 (2)	Se(1)—Nb(1)—Se(5)	59·80 (5)
Nb(1)—Se(3)	2·715 (2)	Se(4)—Nb(1)—Se(4)	113·34 (6)
Nb(2)—Se(2)	2·639 (2)	Se(4)—Nb(1)—Se(5)	87·39 (6)
Nb(2)—Se(5)	2·671 (2)	Se(6)—Nb(2)—Se(8)	77·84 (5)
Nb(2)—Se(6)	2·621 (2)	Se(6)—Nb(2)—Se(3)	139·71 (6)
Nb(2)—Se(2)	2·647 (2)	Se(2)—Nb(2)—Se(8)	83·71 (5)
Nb(2)—Se(6)	2·634 (2)	Se(6)—Nb(2)—Se(5)	87·14 (6)
Nb(2)—Se(3)	2·718 (2)	Se(3)—Nb(2)—Se(5)	76·99 (6)
Nb(2)—Se(8)	2·721 (2)	Se(3)—Se(9)—Se(5)	83·50 (6)
Nb(2)—Se(9)	2·626 (2)	Se(5)—Se(1)—Se(8)	83·49 (6)

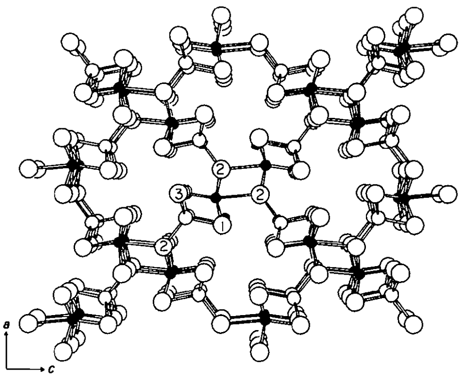
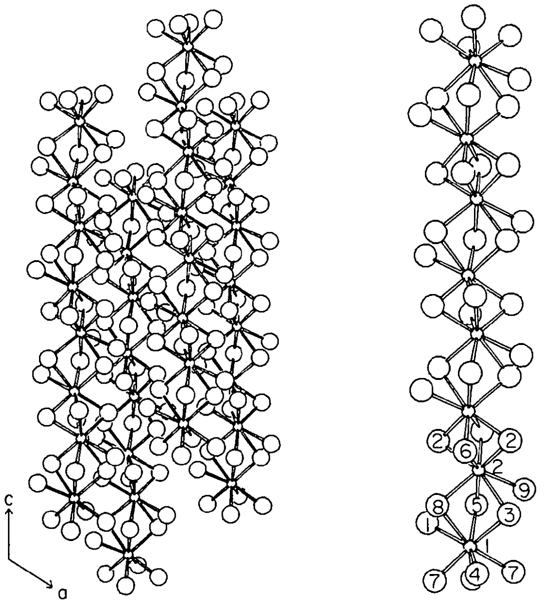


Fig. 1. A view of the structure of $CuTaS_3$ down the [010] direction. The labeling scheme is shown. Small filled circles are Ta atoms, small open circles are Cu atoms, and large open circles are S atoms.



(a) (b)

Fig. 2. Two drawings of the Nb_2Se_9 structure. In both views the small circles are Nb atoms and the large circles are Se atoms. In (a) adjacent chains of the Nb_2Se_9 structure are presented while (b) shows one such chain and the labeling scheme.

A view of the structure of Nb_2Se_9 is given in Fig. 2. The structure is comprised of one-dimensional chains that run along the *c* axis. The chains contain Nb atoms in bicapped trigonal prisms of Se atoms. The trigonal prisms are linked through a quadrilateral face to one prism and through an edge and a capping atom to a second prism. The sharing of a quadrilateral face allows for the formation of a short Nb—Nb distance [2·895 (2) Å]. This distance is similar to the value 2·871 (4) Å, found in the structurally related compound NbS_2Cl_2 (Rijnsdorp, de Lange & Wieger, 1979). In addition to this short Nb—Nb distance a variety of Se—Se bonds are also present in the structure. The quadrilateral face is formed by two pairs of Se atoms [Se—Se = 2·319 (2) and 2·316 (2) Å] while the other end of the trigonal prism contains an Se_5 unit. This Se_5 unit contains two short Se—Se bonds [2·365 (2) and 2·364 (2) Å] and two longer Se—Se distances [2·643 (2) and 2·663 (2) Å]. The bond distances and bond angles reported here, while perhaps ten times more precise, do not differ significantly from those reported earlier [*e.g.* Se(1)—Se(8) = 2·365 (2) Å *versus* 2·34 (3) Å]. The description of the structure in valence terms as $2Nb^{4+}2(Se_2)^{2-}(Se_5)^{4-}$ with an Nb—Nb bond is consistent with the observed semiconducting behavior (Rouxel, 1982).

The metal–metal bonding in both $CuTaS_3$ and Nb_2Se_9 and the Se—Se bonding in the latter compound are typical of ternary chalcogenides. As the structural phenomena in ternary chalcogenides are often linked to the physical properties a detailed knowledge of the metrical details of these compounds is of particular importance. The improved accuracy of the bond distances and angles in Nb_2Se_9 and $CuTaS_3$ will aid in characterizing metal–metal and chalcogen–chalcogen bonds in other compounds.

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Absolute Structure of LiH_2AsO_4

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Abstract. $M_r = 147·87$, orthorhombic, $Pna2_1$, $a = 6·416$ (2), $b = 7·727$ (3), $c = 7·298$ (2) Å, $V = 361·8$ Å^3, $Z = 4$, $D_x = 2·72$ g cm^{-3}, λ(Ag $K\alpha$) = 0·5608 Å, $\mu = 5·14$ cm^{-1}, $F(000) = 280$, $T = 293$ K. Final $R = 0·018$ for 1700 unique observed reflections. The absolute crystal structure has been solved. LiH_2AsO_4 is isostructural with LiH_2PO_4, but one of the two independent hydrogen bonds (these bonds involve the

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