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Hexamolybdenum Octatelluride, Mo_6Te_8

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Abstract

The title compound, isotypic with Mo_6Se_8 , contains Mo_6Te_8 clusters. All eight faces of a distorted octahedral Mo cluster [Mo—Mo 2.699 (2), 2.785 (2) Å] are capped by Te atoms, six of which also provide bridges to neighboring clusters.

Comment

Mo_6Te_8 was reported in 1970 (Grandjean *et al.*, 1970) to be isostructural with Mo_6Se_8 (Bars *et al.*, 1973), based on photographically recorded diffraction patterns. Lattice constants were given. During the course of our studies on ternary molybdenum chalcogenide systems, crystals of Mo_6Te_8 were synthesized by chemical-transport reactions using a number of transport agents. The best crystals were grown using Sb or As at 1273 K. The material can be prepared in single-phase yield by direct combination of the elements at this temperature. Mo_6Te_8 obviously bears a close structural relationship to the important Chevrel phases $M_x\text{Mo}_6Q_8$ (M = alkali metal, alkaline earth, 3d transition metal, lanthanide, actinide or other cation; $x < 4$; Q = S, Se, Te), the superconducting and critical magnetic field properties of which have generated a great deal of synthetic and theoretical interest (see Etourneau, 1992). As such, Mo_6Te_8 warrants a complete structural determination.

The title compound contains interlinked Mo_6Te_8 clusters, the central metal cluster moiety of which is a distorted octahedral Mo_6 cluster. The distortion takes the form of an elongation of the cluster in the c direction, generating short [2.699 (2) Å] Mo—Mo distances in the crystallographic ab plane and long [2.785 (2) Å] Mo—Mo distances along the c direction. A distorted cube of Te atoms surrounds the Mo_6 cluster, with six Te2 atoms capping six of the faces of the Mo_6 cluster [Mo—Te2 2.717 (1), 2.777 (2) and 2.781 (2) Å] and Te1 atoms capping the other two faces, forming three bonds with Mo—Te1 2.732 (2) Å. The six Te2 atoms provide bridges to neighboring Mo_6Te_8 groups (Fig. 1) at the six cluster vertex sites. The distances from the vertex Mo to each bridging Te2 are equivalent [2.818 (1) Å].

Since the reactions that produced the crystals used for the present determination employed As or Sb, the possibility of incorporation of a ternary element must be addressed. In the Chevrel phases, the ternary M atom

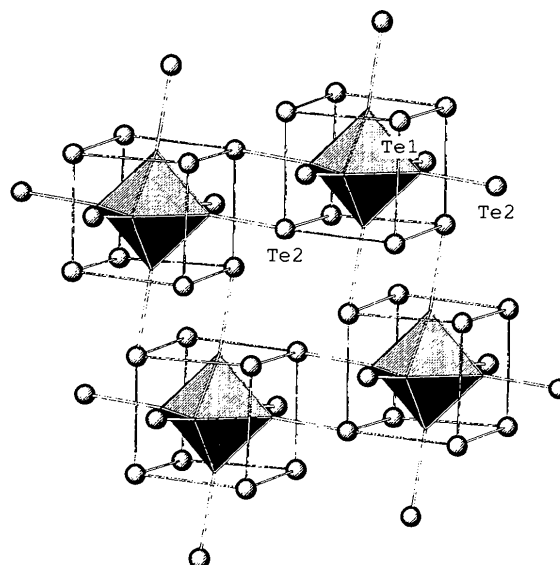


Fig. 1. Coordination and connectivity of the Mo_6 clusters. For clarity, only intercluster Mo—Te2 bonds are represented. The lines drawn between Te atoms indicate only the cubic environment of the Mo_6 cluster, and do not represent bonds (Te···Te *ca.* 3.9 Å).

resides in the large eight-coordinate spaces formed by the chalcogens from eight different Mo_6Q_8 units. The corresponding position in Mo_6Te_8 is (0, 0, 1/2), but the Fourier map shows no significant features at this position ($1.9 e \text{ \AA}^{-3}$). Furthermore, qualitative energy-dispersive spectroscopy compositional analysis using a JEOL 6100 scanning electron microscope confirmed the presence of only Mo and Te in multiple crystal samples.

Experimental

Black reflective cubic crystals of Mo_6Te_8 formed as by-products of reactions loaded according to the stoichiometry Mo_3X_3Te (X = As, Sb), and heated at 1273 K for 7 d in sealed evacuated fused-silica tubes. The compound can be synthesized in single-phase yield by stoichiometric reaction of the elements at 1273 K.

Crystal data

Mo_6Te_8
 $M_r = 1596.44$
 Hexagonal
 $R\bar{3}$
 $a = 10.1790$ (10) Å
 $c = 11.674$ (2) Å
 $V = 1047.5$ (2) Å³
 $Z = 3$
 $D_x = 7.592$ Mg m⁻³
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 50 reflections
 $\theta = 5.8$ – 25.0°
 $\mu = 21.560$ mm⁻¹
 $T = 293$ (2) K
 Block
 $0.10 \times 0.07 \times 0.06$ mm
 Black

Data collection

Siemens P4 diffractometer
 ω scans

$R_{\text{int}} = 0.067$
 $\theta_{\text{max}} = 25^\circ$

Absorption correction: $h = -1 \rightarrow 11$
 ψ scans (SHELXL93; $k = -12 \rightarrow 1$
 Sheldrick, 1993) $l = -13 \rightarrow 1$
 $T_{\min} = 0.234$, $T_{\max} = 0.274$ 3 standard reflections
 604 measured reflections every 97 reflections
 413 independent reflections intensity decay: none
 398 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2 $\Delta\rho_{\max} = 1.95 \text{ e } \text{\AA}^{-3}$
 $R[F > 4\sigma(F)] = 0.039$ $\Delta\rho_{\min} = -2.06 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.109$ Extinction correction:
 $S = 1.187$ SHELXL93
 413 reflections Extinction coefficient:
 23 parameters 0.00112 (13)
 $w = 1/[\sigma^2(F_o^2) + (0.0230P)^2$ Scattering factors from
 $+ 172.7655P]$ International Tables for
 where $P = (F_o^2 + 2F_c^2)/3$ Crystallography (Vol. C)
 $(\Delta/\sigma)_{\max} < 0.001$

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Mo	-0.14367 (12)	0.01736 (13)	0.09888 (9)	0.0157 (5)
Te1	0	0	0.29112 (13)	0.0189 (5)
Te2	-0.36957 (10)	0.00200 (10)	0.24699 (7)	0.0181 (5)

A peak of $1.9 \text{ e } \text{\AA}^{-3}$ remained in the final difference Fourier map. It is located 2.44 \AA from Te1 at (0,0,1/2), but is small considering the heavy atoms in the structure.

Data collection: P4 software (Siemens, 1993, 1994). Cell refinement: P4. Data reduction: P4. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ATOMS (Dowty, 1994). Software used to prepare material for publication: SHELXL93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1198). Services for accessing these data are described at the back of the journal.

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