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Gordon J. Miller

*Iowa State University*, [gmillers@iastate.edu](mailto:gmillers@iastate.edu)

M. Smith

*Iowa State University*

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# Hexamolybdenum Octatelluride, Mo<sub>6</sub>Te<sub>8</sub>

## Abstract

The title compound, isotypic with Mo<sub>6</sub>Se<sub>8</sub>, contains Mo<sub>6</sub>Te<sub>8</sub> clusters. All eight faces of a distorted octahedral Mo cluster [Mo---Mo 2.699 (2), 2.785 (2), ~,] are capped by Te atoms, six of which also provide bridges to neighboring clusters.

## Disciplines

Materials Chemistry | Other Chemistry | Physical Chemistry

## Comments

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## Hexamolybdenum Octatelluride, $\text{Mo}_6\text{Te}_8$

GORDON J. MILLER AND MARK SMITH

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

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### Abstract

The title compound, isotypic with  $\text{Mo}_6\text{Se}_8$ , contains  $\text{Mo}_6\text{Te}_8$  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

$\text{Mo}_6\text{Te}_8$  was reported in 1970 (Grandjean *et al.*, 1970) to be isostructural with  $\text{Mo}_6\text{Se}_8$  (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  $\text{Mo}_6\text{Te}_8$  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.  $\text{Mo}_6\text{Te}_8$  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,  $\text{Mo}_6\text{Te}_8$  warrants a complete structural determination.

The title compound contains interlinked  $\text{Mo}_6\text{Te}_8$  clusters, the central metal cluster moiety of which is a distorted octahedral  $\text{Mo}_6$  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  $\text{Mo}_6$  cluster, with six Te2 atoms capping six of the faces of the  $\text{Mo}_6$  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  $\text{Mo}_6\text{Te}_8$  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

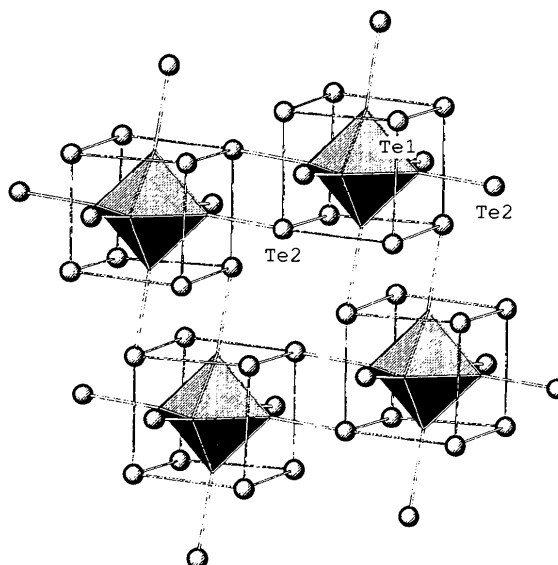


Fig. 1. Coordination and connectivity of the  $\text{Mo}_6$  clusters. For clarity, only intercluster Mo—Te2 bonds are represented. The lines drawn between Te atoms indicate only the cubic environment of the  $\text{Mo}_6$  cluster, and do not represent bonds (Te...Te ca 3.9 Å).

resides in the large eight-coordinate spaces formed by the chalcogens from eight different  $\text{Mo}_6Q_8$  units. The corresponding position in  $\text{Mo}_6\text{Te}_8$  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  $\text{Mo}_6\text{Te}_8$  formed as by-products of reactions loaded according to the stoichiometry  $\text{Mo}_3X_3\text{Te}$  ( $X$  = 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

$\text{Mo}_6\text{Te}_8$   
 $M_r = 1596.44$   
 Hexagonal  
 $R\bar{3}$   
 $a = 10.1790$  (10) Å  
 $c = 11.674$  (2) Å  
 $V = 1047.5$  (2) Å<sup>3</sup>  
 $Z = 3$   
 $D_x = 7.592$  Mg m<sup>-3</sup>  
 $D_m$  not measured

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

#### Data collection

Siemens P4 diffractometer  
 $\omega$  scans

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

Absorption correction:  $h = -1 \rightarrow 11$   
 $\psi$  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 ( $\text{\AA}^2$ )

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

	x	y	z	$U_{\text{eq}}$
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  $2.44 \text{ \AA}$  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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