$V = 890.97$ Å^3
$Z = 2$
$D_x = 2.093$ Mg m^{-3}

0.35 × 0.2 × 0.1 mm
Colorless

Data collection

Siemens *R*3*m*/*E* diffractometer
Wyckoff scans
Absorption correction:
  ψ scans
  $T_{\min} = 0.362$, $T_{\max} = 1.000$
1353 measured reflections
1169 independent reflections
996 observed reflections
  $[F_o^2 > 3\sigma(F_o^2)]$

$R_{\rm int} = 0.0522$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = -11 \rightarrow 11$
3 standard reflections
  monitored every 97 reflections
  intensity decay: none

Refinement

Refinement on *F*
$R = 0.0331$
$wR = 0.0442$
$S = 1.13$
996 reflections
114 parameters
H atoms riding with C—H = 0.96 Å
$w = 1/[\sigma^2(F_o) + 0.0012F_o^2]$

$(\Delta/\sigma)_{\max} = -0.016$
$\Delta\rho_{\max} = 1.38$ e Å^{-3}
  (near Pt)
$\Delta\rho_{\min} = -2.20$ e Å^{-3}
Atomic scattering factors
  from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Pt	0	1/2	1/2	0.019 (1)
P(1)	0.1400 (2)	0.3266 (1)	0.5502 (2)	0.019 (1)
N(1)	0.3018 (10)	0.5526 (6)	0.3205 (7)	0.048 (3)
N(2)	0.3994 (8)	0.1641 (5)	0.5111 (5)	0.029 (2)
N(3)	0.3610 (8)	0.2065 (4)	0.7329 (5)	0.024 (2)
N(4)	0.1180 (8)	0.0986 (4)	0.6002 (5)	0.027 (2)
C(1)	0.190 (1)	0.5351 (7)	0.3866 (8)	0.027 (2)
C(2)	0.319 (1)	0.2707 (6)	0.4562 (7)	0.029 (2)
C(3)	−0.0027 (9)	0.1968 (6)	0.5573 (8)	0.027 (2)
C(4)	0.2747 (9)	0.3194 (6)	0.7094 (6)	0.025 (2)
C(5)	0.4897 (9)	0.1810 (6)	0.6409 (7)	0.031 (2)
C(6)	0.254 (1)	0.0750 (6)	0.5117 (7)	0.031 (2)
C(7)	0.218 (1)	0.1170 (6)	0.7262 (7)	0.029 (2)

Table 2. *Selected geometric parameters* (Å, °)

Pt—P(1)	2.305 (2)	C(1)—N(1$^{\rm i}$)	1.15 (1)
Pt—C(1)	1.975 (9)		
P(1)—Pt—P(1$^{\rm i}$)	180.0	P(1)—Pt—C(1)	90.6 (2)
C(1)—Pt—C(1$^{\rm i}$)	180.0	P(1$^{\rm i}$)—Pt—C(1)	89.4 (2)

Symmetry code: (i) $-x, 1-y, 1-z$.

The *SHELXTL-Plus* program package (Sheldrick, 1991) was used for crystallographic calculations.

Support from the National Science Foundation (CHE 9300107), the Robert A. Welch Foundation and the Texas Advanced Research Program is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1042). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexamolybdenum Cluster with One Telluride and Seven Chloride Capping Ligands

Masahiro Ebihara, Toshihiro Imai and Takashi Kawamura

Department of Chemistry, Faculty of Engineering, Gifu University, Yanagido, Gifu 501-11, Japan

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Abstract

Tetraethylammonium hepta-μ_3-chloro-μ_3-telluro-hexachlorohexamolybdate, $(Et_4N)_3[(Mo_6Cl_7Te)Cl_6]$, has an octahedral hexamolybdenum cluster core whose faces are capped by seven Cl atoms and one Te atom. The capping telluride is disordered in the crystal. The average Mo—Mo distance is not much different from those in $[(Mo_6Cl_7Z)Cl_6]^{3-}$ (Z = S, Se).

Comment

The hexamolybdenum cluster unit has been observed in solid-state Chevrel-phase compounds, $M_xMo_6Z_8$ (M = Cu, Pb, *etc.*; Z = S, Se, Te) (Yvon, 1978). Similar Mo$_6$-cluster structures have been found in discrete halide-cluster ions, $[(Mo_6X_8)X'_6]^{2-}$ ($X = X'$ = Cl, Br). The Chevrel-phase compounds have formal oxidation states of Mo_6^{12+} to Mo_6^{16+}, whereas the reported halide clusters are known to have only Mo_6^{12+}. The halide–chalcogenide

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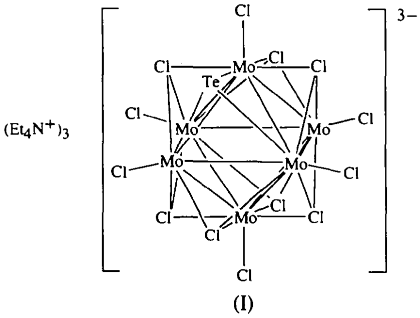
mixed capping-ligand clusters $Mo_6X_{8-x}Z_x^{n+}$ are closely related to both structures. The discrete mixed-ligand clusters $[(Mo_6Cl_7Z)Cl_6]^{3-}$ were first synthesized by Michel & McCarley (1982) as pyridinium salts. We have reported the structures of the Mo_6^{12+} complexes $Cs_3[(Mo_6Cl_7Z)Cl_6].H_2O$ (Z = S, Se) (Ebihara, Toriumi & Saito, 1988) and $(Et_4N)_3[(Mo_6Br_7S)Cl_6]$ (Ebihara, Isobe, Sasaki & Saito, 1992) and the redox potentials, electronic absorption spectra, and ^{95}Mo NMR spectra were measured for their $^nBu_4N^+$ salts. The structures of the Mo_6^{13+} species $(^nBu_4N)_2[(Mo_6X_7Z)Cl_6]$ (X, Z = Cl, S; Cl, Se; Br, S) were also reported (Ebihara, Isobe, Sasaki & Saito, 1992). However, in all of these crystal structures the chalcogenide atom was disordered. Thus, we decided to synthesize the cluster with a larger capping chalcogenide, $[(Mo_6Cl_7Te)Cl_6]^{3-}$, as its Et_4N^+ salt, (I), in the hope of obtaining crystals without disorder of the chalcogenide ligand; the results are reported here.



(I)

Unfortunately, the telluride ion is disordered in the crystal of $(Et_4N)_3[(Mo_6Cl_7Te)Cl_6]$. There are two independent capping-atom sites in the unit cell, Te1 (x, y, $\frac{1}{2}$) and Te2 (x, x, z). Te1 was refined as 0.058 Te + 0.942 Cl and Te2 as 0.192 Te + 0.808 Cl. In isostructural $(Et_4N)_3[(Mo_6Br_7S)Cl_6]$ (Ebihara, Isobe, Sasaki & Saito, 1992), the occupancy of Br is greater at the corresponding site to Te2 of the present complex than the other site. These results indicate that a larger atom tends to occupy the (x, x, z) site.

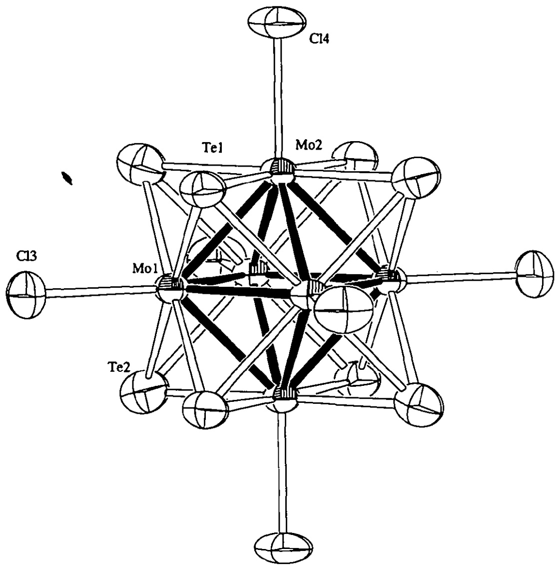


Fig. 1. Structure of the hexamolybdenum cluster showing 50% probability displacement ellipsoids.

The average Mo—Mo distance in the title cluster [2.613 (2) Å] is similar to those in the $[(Mo_6Cl_7Z)Cl_6]^{3-}$ (Z = Cl, Se) clusters [2.604 (2)–2.612 (3) Å (Michel & McCarley, 1982; Ebihara, Toriumi & Saito, 1988)]. The $[(Mo_6Cl_7Te)Cl_6]^{3-}$ ion shows one irreversible oxidation wave in acetonitrile, whereas a reversible wave has been observed for $[(Mo_6Cl_7S)Cl_6]^{3-}$ and $[(Mo_6Cl_7Se)Cl_6]^{3-}$ (Ebihara, Toriumi & Saito, 1988). Thus, although the title cluster has an average Mo—Mo distance similar to those of the other chalcogenide substituted species, the redox behaviour of each is different.

Experimental

The cluster ion was obtained by a similar method to that used for $[(Mo_6Cl_7Se)Cl_6]^{3-}$, using Te powder instead of Se (Ebihara, Toriumi & Saito, 1988). The crystal of the Et_4N^+ salt was grown by slow diffusion of Et_4NCl in aqueous 6 M HCl solution into an aqueous 6 M HCl solution of the cluster.

Crystal data

$(C_8H_{20}N)_3[Mo_6Cl_{13}Te]$
M_r = 1554.89
Tetragonal
$P4_2/mnm$
a = 11.995 (1) Å
c = 16.692 (2) Å
V = 2401.7 (3) Å^3
Z = 2
D_x = 2.150 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 12.1–15.0°
μ = 2.857 mm^{-1}
T = 296 K
Prismatic
0.15 × 0.05 × 0.05 mm
Orange brown

Data collection

AFC-7R diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
T_{min} = 0.844, T_{max} = 0.998
3124 measured reflections
1663 independent reflections
988 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.046
θ_{max} = 27.5°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 21$
3 standard reflections monitored every 150 reflections
intensity decay: 0.29%

Refinement

Refinement on F
R = 0.038
wR = 0.043
S = 1.52
988 reflections
76 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + (0.017F)^2/4]$

$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

		Occupancy	x	y	z	$U_{\rm eq}$
Mo1	8(*j*)	1.0	0.57709 (5)	$1-x$	0.57842 (5)	0.0333 (3)
Mo2	4(*g*)	1.0	0.60861 (7)	x	1/2	0.0326 (4)
Te1	8(*i*)	0.058	0.7547 (2)	0.4573 (2)	1/2	0.050 (1)
Cl1	8(*i*)	0.942	x (Te1)	y (Te1)	1/2	0.050
Te2	8(*j*)	0.192	0.3909 (1)	x	0.6561 (1)	0.0579 (8)
Cl2	8(*j*)	0.808	x (Te2)	x (Te2)	z (Te2)	0.0579
Cl3	8(*j*)	1.0	0.6831 (2)	$1-x$	0.6798 (2)	0.067 (1)
Cl4	4(*g*)	1.0	0.7530 (2)	x	1/2	0.067 (4)
N1	2(*b*)	1.0	1/2	1/2	0	0.056 (8)
N2	4(*d*)	1.0	1/2	0	1/4	0.053 (5)
C1	8(*j*)	0.5†	0.556 (2)	$1-x$	−0.080 (2)	0.11 (1)
C2	4(*e*)	1.0	1/2	1/2	−0.156 (1)	0.19 (1)
C3	8(*i*)	0.5†	0.376 (2)	0.480 (2)	0	0.08 (1)
C4	4(*f*)	1.0	0.348 (1)	x	0	0.14 (1)
C5	16(*k*)	1.0	0.5836 (7)	0.0667 (8)	0.1981 (5)	0.074 (6)
C6	16(*k*)	1.0	0.5269 (9)	0.1431 (9)	0.1372 (6)	0.113 (9)

† Occupancy fixed by symmetry.

Table 2. *Selected geometric parameters* (Å, °)

Mo1—Mo1^{i}	2.618 (2)	Mo2—Te1	2.523 (2)
Mo1—Mo1^{ii}	2.616 (2)	Mo2—Te1^{iii}	2.523 (2)
Mo1—Mo2	2.611 (1)	Mo2—Te2^{ii}	2.606 (2)
Mo1—Te1	2.534 (2)	Mo2—Te2^{iv}	2.606 (2)
Mo1—Te2	2.611 (2)	Mo2—Cl4	2.449 (4)
Mo1—Cl3	2.470 (3)		
Mo1^{i}—Mo1—Mo1^{ii}	90.00	Mo1—Mo2—Te1^{iii}	119.23 (6)
Mo1^{i}—Mo1—Mo2	59.91 (2)	Mo1—Mo2—Te2^{ii}	60.06 (4)
Mo1^{i}—Mo1—Te1	58.90 (3)	Mo1—Mo2—Te2^{iv}	120.23 (5)
Mo1^{i}—Mo1—Te2	119.78 (4)	Mo1—Mo2—Cl4	134.88 (2)
Mo1^{i}—Mo1—Cl3	133.26 (8)	Mo1^{i}—Mo2—Mo1^{ii}	90.24 (4)
Mo1^{ii}—Mo1—Mo2	59.94 (2)	Mo1^{i}—Mo2—Mo1^{iv}	60.11 (4)
Mo1^{ii}—Mo1—Te1	118.64 (5)	Mo1^{i}—Mo2—Te1	59.13 (5)
Mo1^{ii}—Mo1—Te2	59.93 (3)	Mo1^{i}—Mo2—Te1^{iii}	119.23 (6)
Mo1^{ii}—Mo1—Cl3	136.73 (8)	Mo1^{i}—Mo2—Te2^{ii}	120.23 (5)
Mo2—Mo1—Mo2^{ii}	89.76 (4)	Mo1^{i}—Mo2—Te2^{iv}	60.06 (4)
Mo2—Mo1—Te1	58.69 (5)	Mo1^{i}—Mo2—Cl4	134.88 (2)
Mo2—Mo1—Te1^{v}	118.80 (4)	Mo1^{ii}—Mo2—Mo1^{iv}	60.17 (4)
Mo2—Mo1—Te2	119.89 (4)	Te1—Mo2—Te1^{iii}	178.0 (1)
Mo2—Mo1—Te2^{ii}	59.87 (4)	Te1—Mo2—Te2^{ii}	90.00 (2)
Mo2—Mo1—Cl3	135.09 (2)	Te1—Mo2—Te2^{iv}	90.00 (2)
Te1—Mo1—Te1^{v}	90.40 (9)	Te1—Mo2—Cl4	91.01 (6)
Te1—Mo1—Te2	178.32 (6)	Te1^{iii}—Mo2—Te2^{ii}	90.00 (2)
Te1—Mo1—Te2^{ii}	89.64 (6)	Te1^{iii}—Mo2—Te2^{iv}	90.00 (2)
Te1—Mo1—Cl3	90.29 (7)	Te1^{iii}—Mo2—Cl4	91.01 (6)
Te1^{v}—Mo1—Te2	89.64 (6)	Te2^{ii}—Mo2—Te2^{iv}	179.7 (1)
Te1^{v}—Mo1—Te2^{ii}	178.32 (6)	Te2^{ii}—Mo2—Cl4	89.83 (5)
Te1^{v}—Mo1—Cl3	90.29 (7)	Te2^{iv}—Mo2—Cl4	89.83 (5)
Te2—Mo1—Te2^{ii}	90.27 (8)	Mo1—Te1—Mo1^{i}	62.20 (6)
Te2—Mo1—Cl3	91.39 (7)	Mo1—Te1—Mo2	62.18 (6)
Mo1—Mo2—Mo1^{i}	60.17 (4)	Mo1^{i}—Te1—Mo2	62.18 (6)
Mo1—Mo2—Mo1^{ii}	60.11 (4)	Mo1—Te2—Mo1^{ii}	60.12 (5)
Mo1—Mo2—Mo1^{iv}	90.24 (4)	Mo1—Te2—Mo2^{ii}	60.08 (5)
Mo1—Mo2—Te1	59.13 (5)	Mo1^{ii}—Te2—Mo2^{ii}	60.08 (5)

Symmetry codes: (i) $x, y, 1-z$; (ii) $1-x, 1-y, z$; (iii) y, x, z; (iv) $1-x, 1-y, 1-z$; (v) $1-y, 1-x, z$.

The structure was solved on the basis of the structure of isostructural $(\mathrm{Et_4N})_3[(\mathrm{Mo_6Br_7S})\mathrm{Cl_6}]$ (Ebihara, Isobe, Sasaki & Saito, 1992). Each independent capping site was refined as Te + Cl and the sum of the occupancies of Te was restricted to 0.25. Since one $\mathrm{Et_4N^+}$ ion was disordered at the *mmm* site, the occupancies of C1 and C3 were set equal to 0.5.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software.* Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to refine structure: *TEXSAN LS.* Software used to prepare material for publication: *TEXSAN TABLES.*

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: NA1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[Bis(3-aminopropyl)amine-*N*,*N'*,*N''*]-[glycylglycinato(2–)-*N*,*N'*,*O*]-chromium(III) Perchlorate Monohydrate, $[\mathrm{Cr(C_4H_6N_2O_3)(C_6H_{17}N_3)]ClO_4.H_2O}$

JONG-HA CHOI*

Department of Chemistry, Andong National University, Andong 760-749, Korea

IL-HWAN SUH AND SANG-HEE KWAK

Department of Physics, Chungnam National University, Daejeon 305-764, Korea

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Abstract

The mixed-ligand complex $[\mathrm{Cr(dpt)(Gly\text{-}Gly)]ClO_4.H_2O}$ [where dpt = bis(3-aminopropyl)amine and $\mathrm{H_2Gly\text{-}Gly}$ = glycylglycine] adopts a structure in which the ligands are tridentate and coordinate meridionally. The four amine N atoms, the carboxyl O atom and the peptide N atom

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