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The Chevrel phase $HgMo_6S_8$

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Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Mo–S) = 0.001 Å; disorder in main residue; R factor = 0.025; wR factor = 0.026; data-to-parameter ratio = 36.2.

The crystal structure of $HgMo_6S_8$, mercury(II) hexamolybdenum octasulfide, is based on $(Mo_6S_8)S_6$ cluster units ($\overline{3}$ symmetry) interconnected through interunit Mo—S bonds. The Hg^{2+} cations occupy large voids between the different cluster units and are covalently bonded to two S atoms. The Hg atoms and one S atom lie on sites with crystallographic $\overline{3}$ and 3 symmetry, respectively. Refinement of the occupancy factor of the Hg atom led to the composition $Hg_{0.973\ (3)}Mo_6S_8$.

Related literature

For isotypic structures, see: Chevrel & Sergent (1982). For a previous report on the title compound as a polycrystalline material, see: Tarascon *et al.* (1983). For crystallographic background, see: Becker & Coppens (1974); Johnson & Levy (1974).

Experimental

Crystal data

$Hg_{0.973}Mo_6S_8$
M_r = 1027.3
Trigonal, $R\overline{3}$
a = 9.4319 (3) Å
c = 10.7028 (3) Å
V = 824.57 (4) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 21.62 mm^{-1}
T = 293 K
0.08 × 0.07 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
T_{min} = 0.298, T_{max} = 0.384
5784 measured reflections
1121 independent reflections
1069 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.026
S = 1.74
1121 reflections
31 parameters
$\Delta\rho_{max}$ = 2.64 e Å^{-3}
$\Delta\rho_{min}$ = −1.57 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hg1—S1	2.3914 (8)	Mo1—S2	2.4236 (6)
Mo1—Mo1^{i}	2.7184 (3)	Mo1—S2^{iii}	2.4896 (8)
Mo1—Mo1^{ii}	2.7515 (3)	Mo1—S2^{ii}	2.4933 (6)
Mo1—S1	2.4108 (7)	Mo1—S2^{iv}	2.4340 (8)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *JANA2000* (Petříček & Dušek, 2000); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *JANA2000*.

Intensity data were collected on the Nonius KappaCCD X-ray diffactometer system of the Centre de diffractométrie de l'Université de Rennes I (www.cdifx.univ-rennes1.fr).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2226).

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supplementary materials

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Comment

The superconducting compound $HgMo_6S_8$ was first synthesized as a powder sample by Tarascon *et al.* (1983), but no details were given on its crystal structure. In the present study, we present the crystal structure refinement of $HgMo_6S_8$ that has been determined from single-crystal X-ray diffraction data. The title compound is isostructural with the hexagonal Chevrel phases MMo_6X_8 where M is a large cation (M = alkali metal, alkaline earth, lanthanide, actinide *etc*.; X = S, Se, Te) [see, for instance, Chevrel & Sergent (1982)]. As a consequence its crystal structure consists of octahedral Mo_6 clusters surrounded by fourteen sulfur atoms with eight of them forming a distorted cube (*i*-type ligands) and the remaining six capping the faces of the S_8 cube (*a*-type ligands). In the structure of $HgMo_6S_8$, a part of the chalcogen atoms of the $Mo_6S^i{}_8S^a{}_6$ unit are shared according to the formula $Mo_6S^i{}_2S^{i\text{-}a}{}_{6/2}S^{a\text{-}i}{}_{6/2}$ to form the three-dimensional Mo—S network. The Mo_6S_8 cluster unit is centered at Wyckoff position 6*b* ($\bar{3}$ symmetry). The Mo—Mo distances within the Mo_6 clusters are 2.7184 (3) Å for the intra-triangle distances (distances within the Mo_3 triangles formed by the Mo atoms related through the threefold axis) and 2.7515 (3) Å for the inter-triangle distances. Each Mo atom is surrounded by five S atoms (4 S1 and 1 S2) forming a distorted square-based pyramid. The apex of the pyramid is shared with an adjacent unit and thus ensures the three-dimensional cohesion. Consequently, each Mo_6S_8 unit is interconnected to 6 Mo_6S_8 units to form the Mo—S framework. It results from this arrangement that the shortest intercluster Mo1—Mo1 distances between the Mo_6 clusters is 3.2934 (3) Å, indicating only weak metal-metal interaction. The Hg^{2+} cations reside in the large eight-coordinate voids formed by the chalcogen atoms from eight different Mo_6S_8 units. They are covalently bonded to two S2 atoms at a distance of 2.3914 (8) Å.

$HgMo_6S_8$ was found to be superconducting at 8 K from DC-susceptibility measurements on a batch of single crystals.

Experimental

$HgMo_6S_8$ was obtained in three steps involving, first, the syntheses of single-crystal of $InMo_6S_8$ by solid state reaction, then the preparation of the binary compound Mo_6S_8 by 'chimie douce' methods and, finally, the synthesis of the title compound by inserting mercury into the Mo_6S_8 host structure at low temperatures. Single crystals of $InMo_6S_8$ were obtained from a stoichiometric mixture of In_2S_3, MoS_2 and Mo. All handlings of materials were done in an argon-filled glove box. The initial mixture (*ca* 5 g) was cold pressed and loaded into a molybdenum crucible, which was sealed under a low argon pressure using an arc-welding system. The charge was heated at the rate of 300 K/h up to 1773 K, the temperature which was held for six hours, then cooled at 100 K/h down to 1273 K and finally furnace cooled. Mo_6S_8 was obtained by oxidation of single-crystals of $InMo_6S_8$ by iodine in a glass tube sealed under vacuum. The end of the tube containing the crystals of the In compound and an excess of iodine was placed in a furnace with about 3 cm of the other end sticking out of the furnace, at about room temperature. The furnace was then heated at 523 K for 96 h. At the end of the reaction, crystals of InI_3 and I_2 were obtained at the cooler end of the tube. Finally, $HgMo_6S_8$ was prepared by diffusion of mercury into crystals of Mo_6S_8 in a silica glass tube sealed under vacuum at 673 K during 96 h.

Refinement

The structure was refined using an anisotropic approximation and converged at an reliability factor $R(F) = 0.034$. Analyses of the difference Fourier maps revealed positive and negative residual peaks around the Hg atom. Fourth-order tensors in the Gram-Charlier expansion (Johnson & Levy, 1974) of the mercury displacement factor were used to describe the electron density around this site. The resulting R value dropped to 0.025 for only five additional parameters. Refinement of the occupancy factor of the Hg atom led to the final composition $Hg_{0.973\ (3)}Mo_6S_8$.

Figures

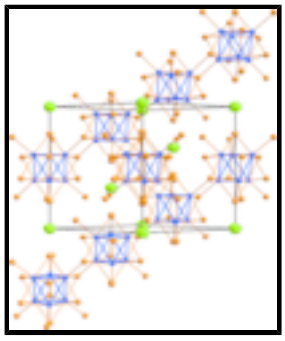

Fig. 1. : View of $HgMo_6S_8$ along [110].

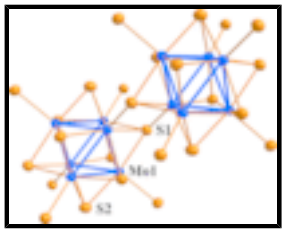

Fig. 2. : Plot showing the atom-numbering scheme and the interunit linkage of the $(Mo_6S_8)S_6$ cluster units. Displacement ellipsoids are drawn at the 97% probability level.

(I)

Crystal data

$Hg_{0.973}Mo_6S_8$	$Z = 3$
$M_r = 1027.3$	$F_{000} = 1374$
Trigonal, $R\bar{3}$	$D_x = 6.204$ (1) Mg m^{-3}
Hall symbol: -R 3	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 9.4319$ (3) Å	Cell parameters from 7043 reflections
$b = 9.4319$ (3) Å	$\theta = 2.0$–$42.1°$
$c = 10.7028$ (3) Å	$\mu = 21.62$ mm^{-1}
$\alpha = 90°$	$T = 293$ K
$\beta = 90°$	Truncated cube, black
$\gamma = 120°$	0.08 × 0.07 × 0.06 mm
$V = 824.57$ (4) Å^3	

Data collection

Nonius KappaCCD diffractometer	1121 independent reflections
Radiation source: fine-focus sealed tube	1069 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.044$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 39.8°$
$T = 293$ K	$\theta_{min} = 3.1°$
ω– and φ–scans	$h = -16 \rightarrow 16$
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	$k = -16 \rightarrow 16$
$T_{min} = 0.298$, $T_{max} = 0.384$	$l = -13 \rightarrow 19$
5784 measured reflections	

Refinement

Refinement on F	Weighting scheme based on measured s.u.'s $w = 1/\sigma^2(F)$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.026$	$\Delta\rho_{max} = 2.64$ e Å^{-3}
$S = 1.74$	$\Delta\rho_{min} = -1.57$ e Å^{-3}
1121 reflections	Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
31 parameters	Extinction coefficient: 0.020681

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Hg1	0	0	0	0.0339 (4)	0.973 (3)
Mo1	−0.01555 (2)	−0.17363 (2)	−0.394419 (15)	0.00748 (7)	
S1	0	0	−0.22344 (8)	0.0113 (2)	
S2	−0.03460 (6)	−0.31591 (7)	−0.58775 (4)	0.00933 (17)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.0384 (4)	0.0384 (4)	0.0249 (6)	0.0192 (2)	0	0
Mo1	0.00780 (9)	0.00831 (9)	0.00617 (10)	0.00391 (6)	0.00003 (5)	−0.00036 (5)
S1	0.0126 (2)	0.0126 (2)	0.0088 (3)	0.00628 (12)	0	0
S2	0.0097 (2)	0.0096 (2)	0.0087 (2)	0.00476 (17)	0.00067 (15)	−0.00032 (15)

Geometric parameters (Å, °)

Hg1—S1	2.3914 (8)	Mo1—Mo1^{ix}	2.7184 (3)
Hg1—S1^{i}	2.3914 (8)	Mo1—Mo1^{x}	2.7515 (3)
Hg1—S2^{ii}	3.2056 (4)	Mo1—Mo1^{xi}	2.7184 (4)

Hg1—S2^{iii}	3.2056 (4)	Mo1—Mo1^{xii}	2.7515 (2)
Hg1—S2^{iv}	3.2056 (7)	Mo1—S1	2.4108 (7)
Hg1—S2^{v}	3.2056 (7)	Mo1—S2	2.4236 (6)
Hg1—S2^{vi}	3.2056 (8)	Mo1—S2^{xiii}	2.4896 (8)
Hg1—S2^{vii}	3.2056 (8)	Mo1—S2^{x}	2.4933 (6)
Mo1—Mo1^{viii}	3.8679 (3)	Mo1—S2^{xii}	2.4340 (8)
Mo1—Mo1^{iii}	3.2131 (2)		
S1—Hg1—S1^{i}	180	Mo1^{x}—Mo1—Mo1^{iii}	97.693 (7)
S1—Hg1—S2^{ii}	105.278 (8)	Mo1^{x}—Mo1—Mo1^{ix}	90
S1—Hg1—S2^{iii}	74.722 (8)	Mo1^{x}—Mo1—Mo1^{xi}	60.398 (8)
S1—Hg1—S2^{iv}	105.278 (9)	Mo1^{x}—Mo1—Mo1^{xii}	59.205 (7)
S1—Hg1—S2^{v}	74.722 (9)	Mo1^{x}—Mo1—S1	115.964 (15)
S1—Hg1—S2^{vi}	105.278 (9)	Mo1^{x}—Mo1—S2	55.677 (18)
S1—Hg1—S2^{vii}	74.722 (9)	Mo1^{x}—Mo1—S2^{xiii}	138.626 (14)
S1^{i}—Hg1—S1	180	Mo1^{x}—Mo1—S2^{x}	54.776 (13)
S1^{i}—Hg1—S2^{ii}	74.722 (8)	Mo1^{x}—Mo1—S2^{xii}	114.515 (14)
S1^{i}—Hg1—S2^{iii}	105.278 (8)	Mo1^{xi}—Mo1—Mo1^{iii}	96.739 (8)
S1^{i}—Hg1—S2^{iv}	74.722 (9)	Mo1^{xi}—Mo1—Mo1^{ix}	60.000 (8)
S1^{i}—Hg1—S2^{v}	105.278 (9)	Mo1^{xi}—Mo1—Mo1^{x}	60.398 (8)
S1^{i}—Hg1—S2^{vi}	74.722 (9)	Mo1^{xi}—Mo1—Mo1^{xii}	90
S1^{i}—Hg1—S2^{vii}	105.278 (9)	Mo1^{xi}—Mo1—S1	55.682 (12)
S2^{ii}—Hg1—S2^{iii}	180	Mo1^{xi}—Mo1—S2	116.065 (18)
S2^{ii}—Hg1—S2^{iv}	113.319 (18)	Mo1^{xi}—Mo1—S2^{xiii}	135.971 (18)
S2^{ii}—Hg1—S2^{v}	66.681 (18)	Mo1^{xi}—Mo1—S2^{x}	55.48 (2)
S2^{ii}—Hg1—S2^{vi}	113.319 (17)	Mo1^{xi}—Mo1—S2^{xii}	117.362 (19)
S2^{ii}—Hg1—S2^{vii}	66.681 (17)	Mo1^{xii}—Mo1—Mo1^{iii}	148.317 (7)
S2^{iii}—Hg1—S2^{ii}	180	Mo1^{xii}—Mo1—Mo1^{ix}	60.398 (6)
S2^{iii}—Hg1—S2^{iv}	66.681 (18)	Mo1^{xii}—Mo1—Mo1^{x}	59.205 (7)
S2^{iii}—Hg1—S2^{v}	113.319 (18)	Mo1^{xii}—Mo1—Mo1^{xi}	90
S2^{iii}—Hg1—S2^{vi}	66.681 (17)	Mo1^{xii}—Mo1—S1	115.964 (13)
S2^{iii}—Hg1—S2^{vii}	113.319 (17)	Mo1^{xii}—Mo1—S2	57.184 (12)
S2^{iv}—Hg1—S2^{ii}	113.319 (18)	Mo1^{xii}—Mo1—S2^{xiii}	133.837 (19)
S2^{iv}—Hg1—S2^{iii}	66.681 (18)	Mo1^{xii}—Mo1—S2^{x}	113.894 (15)
S2^{iv}—Hg1—S2^{v}	180	Mo1^{xii}—Mo1—S2^{xii}	55.318 (14)
S2^{iv}—Hg1—S2^{vi}	113.319 (19)	S1—Mo1—S2	170.65 (2)
S2^{iv}—Hg1—S2^{vii}	66.681 (19)	S1—Mo1—S2^{xiii}	93.53 (2)
S2^{v}—Hg1—S2^{ii}	66.681 (18)	S1—Mo1—S2^{x}	90.323 (17)
S2^{v}—Hg1—S2^{iii}	113.319 (18)	S1—Mo1—S2^{xii}	91.758 (14)
S2^{v}—Hg1—S2^{iv}	180	S2—Mo1—S2^{xiii}	95.79 (2)
S2^{v}—Hg1—S2^{vi}	66.681 (19)	S2—Mo1—S2^{x}	87.39 (2)

$S2^{v}$—Hg1—$S2^{vii}$	113.319 (19)	S2—Mo1—$S2^{xii}$	88.750 (19)
$S2^{vi}$—Hg1—$S2^{ii}$	113.319 (17)	$S2^{xiii}$—Mo1—S2	95.79 (2)
$S2^{vi}$—Hg1—$S2^{iii}$	66.681 (17)	$S2^{xiii}$—Mo1—$S2^{x}$	99.70 (2)
$S2^{vi}$—Hg1—$S2^{iv}$	113.319 (19)	$S2^{xiii}$—Mo1—$S2^{xii}$	91.39 (2)
$S2^{vi}$—Hg1—$S2^{v}$	66.681 (19)	$S2^{x}$—Mo1—S2	87.39 (2)
$S2^{vi}$—Hg1—$S2^{vii}$	180	$S2^{x}$—Mo1—$S2^{xiii}$	99.70 (2)
$S2^{vii}$—Hg1—$S2^{ii}$	66.681 (17)	$S2^{x}$—Mo1—$S2^{xii}$	168.58 (2)
$S2^{vii}$—Hg1—$S2^{iii}$	113.319 (17)	$S2^{xii}$—Mo1—S2	88.750 (19)
$S2^{vii}$—Hg1—$S2^{iv}$	66.681 (19)	$S2^{xii}$—Mo1—$S2^{xiii}$	91.39 (2)
$S2^{vii}$—Hg1—$S2^{v}$	113.319 (19)	$S2^{xii}$—Mo1—$S2^{x}$	168.58 (2)
$S2^{vii}$—Hg1—$S2^{vi}$	180	Hg1—S1—Mo1	139.382 (14)
$Mo1^{viii}$—Mo1—$Mo1^{iii}$	133.459 (8)	Hg1—S1—$Mo1^{ix}$	139.382 (13)
$Mo1^{viii}$—Mo1—S1	85.136 (14)	Hg1—S1—$Mo1^{xi}$	139.382 (14)
$Mo1^{viii}$—Mo1—S2	85.600 (16)	Mo1—S1—$Mo1^{ix}$	68.64 (2)
$Mo1^{viii}$—Mo1—$S2^{xiii}$	176.394 (13)	Mo1—S1—$Mo1^{xi}$	68.64 (2)
$Mo1^{viii}$—Mo1—$S2^{x}$	83.677 (18)	$Mo1^{ix}$—S1—Mo1	68.64 (2)
$Mo1^{viii}$—Mo1—$S2^{xii}$	85.310 (16)	$Mo1^{ix}$—S1—$Mo1^{xi}$	68.64 (2)
$Mo1^{iii}$—Mo1—$Mo1^{viii}$	133.459 (8)	$Mo1^{xi}$—S1—Mo1	68.64 (2)
$Mo1^{iii}$—Mo1—$Mo1^{ix}$	147.479 (10)	$Mo1^{xi}$—S1—$Mo1^{ix}$	68.64 (2)
$Mo1^{iii}$—Mo1—$Mo1^{x}$	97.693 (7)	$Hg1^{xiv}$—S2—Mo1	125.450 (18)
$Mo1^{iii}$—Mo1—$Mo1^{xi}$	96.739 (8)	$Hg1^{xiv}$—S2—$Mo1^{x}$	98.407 (18)
$Mo1^{iii}$—Mo1—$Mo1^{xii}$	148.317 (7)	$Hg1^{xiv}$—S2—$Mo1^{xv}$	97.225 (18)
$Mo1^{iii}$—Mo1—S1	92.988 (11)	$Hg1^{xiv}$—S2—$Mo1^{xii}$	156.59 (2)
$Mo1^{iii}$—Mo1—S2	92.457 (12)	Mo1—S2—$Mo1^{x}$	69.005 (19)
$Mo1^{iii}$—Mo1—$S2^{xiii}$	49.898 (13)	Mo1—S2—$Mo1^{xv}$	132.74 (2)
$Mo1^{iii}$—Mo1—$S2^{x}$	49.797 (18)	Mo1—S2—$Mo1^{xii}$	68.041 (15)
$Mo1^{iii}$—Mo1—$S2^{xii}$	141.203 (18)	$Mo1^{x}$—S2—Mo1	69.005 (19)
$Mo1^{ix}$—Mo1—$Mo1^{iii}$	147.479 (10)	$Mo1^{x}$—S2—$Mo1^{xv}$	129.09 (2)
$Mo1^{ix}$—Mo1—$Mo1^{x}$	90	$Mo1^{x}$—S2—$Mo1^{xii}$	66.955 (19)
$Mo1^{ix}$—Mo1—$Mo1^{xi}$	60.000 (8)	$Mo1^{xv}$—S2—Mo1	132.74 (2)
$Mo1^{ix}$—Mo1—$Mo1^{xii}$	60.398 (6)	$Mo1^{xv}$—S2—$Mo1^{x}$	129.09 (2)
$Mo1^{ix}$—Mo1—S1	55.682 (11)	$Mo1^{xv}$—S2—$Mo1^{xii}$	80.305 (15)
$Mo1^{ix}$—Mo1—S2	117.489 (13)	$Mo1^{xii}$—S2—Mo1	68.041 (15)
$Mo1^{ix}$—Mo1—$S2^{xiii}$	131.337 (14)	$Mo1^{xii}$—S2—$Mo1^{x}$	66.955 (19)
$Mo1^{ix}$—Mo1—$S2^{x}$	115.28 (2)	$Mo1^{xii}$—S2—$Mo1^{xv}$	80.305 (15)
$Mo1^{ix}$—Mo1—$S2^{xii}$	57.566 (18)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $x+1/3, y+2/3, z+2/3$; (iii) $-x-1/3, -y-2/3, -z-2/3$; (iv) $-y-2/3, x-y-1/3, z+2/3$; (v) $y+2/3, -x+y+1/3, -z-2/3$; (vi) $-x+y+1/3, -x-1/3, z+2/3$; (vii) $x-y-1/3, x+1/3, -z-2/3$; (viii) $-x, -y, -z-1$; (ix) $-y, x-y, z$; (x) $y, -x+y, -z-1$; (xi) $-x+y, -x, z$; (xii) $x-y, x, -z-1$; (xiii) $-y-1/3, x-y-2/3, z+1/3$; (xiv) $x-1/3, y-2/3, z-2/3$; (xv) $-x+y+1/3, -x-1/3, z-1/3$.

Fig. 1

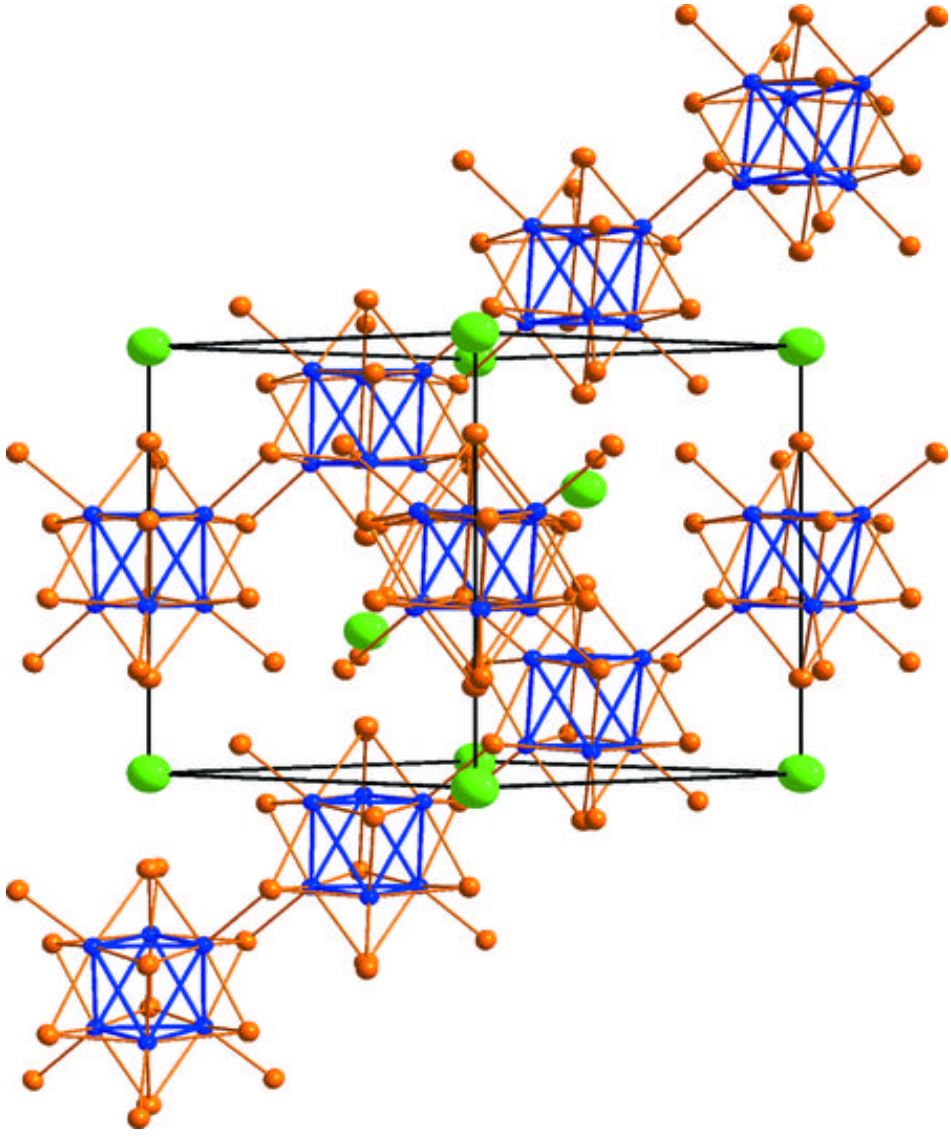
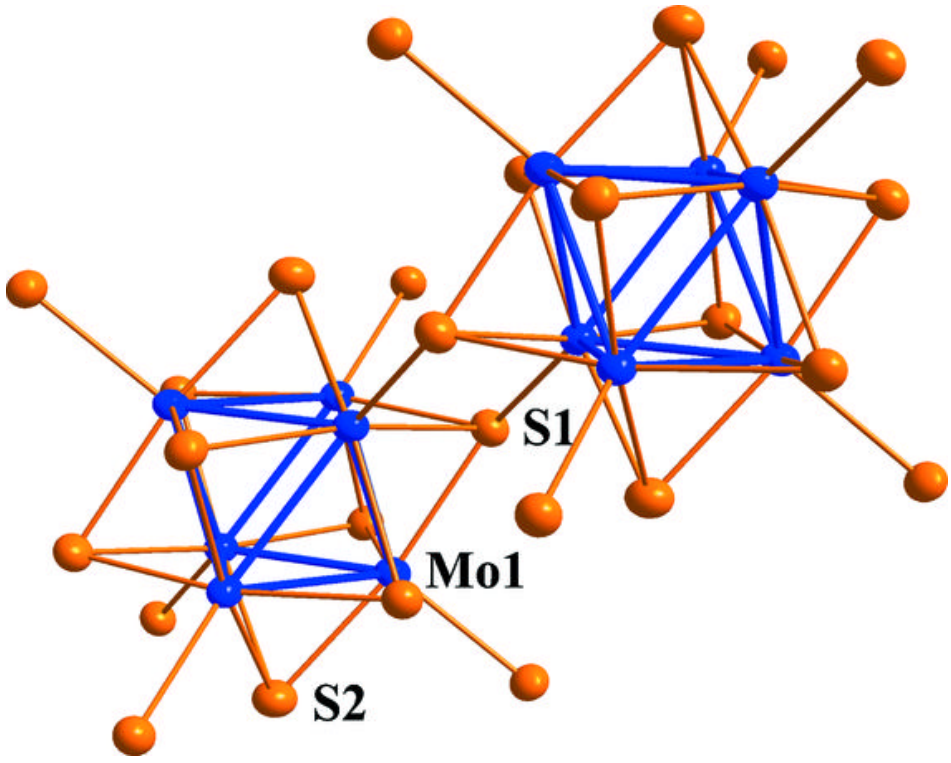
Fig. 2