

Tl₂Mo₉Se₁₁

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Key indicators: single-crystal X-ray study; *T* = 293 K; mean $\sigma(\text{Mo}-\text{Se}) = 0.001 \text{ \AA}$; *R* factor = 0.043; *wR* factor = 0.092; data-to-parameter ratio = 59.4.

The structure of Tl₂Mo₉Se₁₁, dithallium nonamolybdenum undecaselesenide, is isotypic with Tl₂Mo₉S₁₁ [Potel *et al.* (1980). *Acta Cryst. B* **36**, 1319–1322]. The structural set-up is characterized by a mixture of Mo₆Se₈ⁱSe₆^a and Mo₁₂Se₁₄Se₆^a cluster units in a 1:1 ratio. Both components are interconnected through interunit Mo–Se bonds. The cluster units are centered at Wyckoff positions *3a* and *3b* (point-group symmetry $\bar{3}$). The two Tl^I atoms are situated in the voids of the three-dimensional arrangement. Two of the five independent Se atoms and the Tl atoms lie on sites with 3. symmetry (Wyckoff site 6c).

Related literature

For the crystal structure of Tl₂Mo₉S₁₁, see: Potel *et al.* (1980). For details of the *i*- and *a*-type ligand notation, see: Schäfer & von Schnering (1964). Ionic radii were compiled by Shannon (1976). For background to the extinction correction, see: Becker & Coppens (1974).

Experimental

Crystal data

Tl₂Mo₉Se₁₁ *Z* = 6
M_r = 2140.8 Mo *K*α radiation
 Trigonal, *R*³ μ = 42.73 mm⁻¹
a = 9.6212 (1) Å *T* = 293 K
c = 36.3316 (7) Å 0.13 × 0.12 × 0.11 mm
V = 2912.55 (7) Å³

Data collection

Nonius KappaCCD diffractometer 21097 measured reflections
 Absorption correction: multi-scan 4097 independent reflections
 (PLATON; Spek, 2009) 3156 reflections with *I* > 2σ(*I*)
*T*_{min} = 0.033, *T*_{max} = 0.108 *R*_{int} = 0.087

Refinement

R[*F*² > 2σ(*F*²)] = 0.043 69 parameters
wR(*F*²) = 0.092 Δ*ρ*_{max} = 3.92 e Å⁻³
S = 1.37 Δ*ρ*_{min} = -2.89 e Å⁻³
 4097 reflections

Table 1

Selected bond lengths (Å).

Tl1–Tl2 ⁱ	3.5164 (7)	Mo1–Mo1 ^x	2.7609 (6)
Tl1–Se2	4.3543 (6)	Mo1–Mo2 ^{viii}	3.4239 (8)
Tl1–Se2 ⁱⁱ	4.3543 (7)	Mo1–Se1	2.5540 (8)
Tl1–Se2 ⁱⁱⁱ	4.3543 (7)	Mo1–Se1 ^x	2.6143 (8)
Tl1–Se3	3.5840 (6)	Mo1–Se1 ^{xiii}	2.5757 (6)
Tl1–Se3 ⁱⁱ	3.5840 (5)	Mo1–Se2	2.6247 (8)
Tl1–Se3 ⁱⁱⁱ	3.5840 (8)	Mo1–Se4	2.5269 (8)
Tl1–Se3 ^{iv}	3.4547 (6)	Mo2–Mo2 ^{ix}	2.6382 (5)
Tl1–Se3 ^v	3.4547 (8)	Mo2–Mo3	2.7397 (7)
Tl1–Se3 ^{vi}	3.4547 (5)	Mo2–Mo3 ^{viii}	2.7901 (7)
Tl1–Se4 ^{vii}	3.0737 (10)	Mo2–Se1 ⁱ	2.6597 (6)
Tl2–Se1	3.4032 (7)	Mo2–Se2	2.6034 (9)
Tl2–Se1 ^{viii}	3.4032 (6)	Mo2–Se2 ^{ix}	2.6124 (10)
Tl2–Se1 ^{ix}	3.4032 (8)	Mo2–Se3	2.6721 (6)
Tl2–Se2 ⁱ	3.1441 (4)	Mo2–Se5	2.5129 (8)
Tl2–Se2 ^x	3.1441 (7)	Mo3–Mo3 ^{ix}	2.6780 (7)
Tl2–Se2 ^{xi}	3.1441 (7)	Mo3–Mo3 ^{xiv}	2.6796 (6)
Tl2–Se3 ⁱ	4.2446 (7)	Mo3–Se2 ^{ix}	2.5634 (8)
Tl2–Se3 ^x	4.2446 (6)	Mo3–Se3	2.6090 (6)
Tl2–Se3 ^{xi}	4.2446 (8)	Mo3–Se3 ^{ix}	2.6112 (6)
Tl2–Se5	3.0483 (9)	Mo3–Se3 ^{xiv}	2.7025 (8)
Mo1–Mo1 ^{xii}	2.6897 (7)		

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

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: JANA2006 (Petříček *et al.*, 2006); molecular graphics: DIAMOND (Brandenburg, 2001); software used to prepare material for publication: JANA2006.

Intensity data were collected at 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: WM2368).

References

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Tl₂Mo₉Se₁₁

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Comment

Potel *et al.* (1980) reported the crystal structure of Tl₂Mo₉S₁₁ that was the first compound containing a transition metal cluster with a nuclearity higher than 9, namely the trioctahedral Mo₁₂ cluster. The latter cluster, which results from the uniaxial face-sharing of three Mo₆ octahedra, coexists with the octahedral Mo₆ cluster in equal proportion. We present here the crystal structure of the isotypic selenide, Tl₂Mo₉Se₁₁.

The Mo—Se framework of Tl₂Mo₉Se₁₁ consists of an equal mixture of Mo₆Se^{*i*}₈Se^{*a*}₆ and Mo₁₂Se^{*i*}₁₄Se^{*a*}₆ cluster units interconnected through Mo—Se bonds (Fig.1 and 2). Details of the *i*- and *a*-type ligand notation were reported by Schäfer & von Schnering (1964). The first unit can be described as an Mo₆ octahedron surrounded by 8 face-capping inner Se^{*i*} and 6 apical Se^{*a*} ligands. The Mo₁₂ core of the second unit results from the face sharing of 3 octahedral Mo₆ clusters. The Mo₁₂ cluster is surrounded by 14 Se^{*i*} atoms capping the faces of the bioctahedron and 6 apical Se^{*a*} ligands above the ending Mo atoms. The Mo₆Se^{*i*}₈Se^{*a*}₆ and Mo₁₂Se^{*i*}₁₄Se^{*a*}₆ units are centered at 3*a* and 3*b* positions and have point-group symmetry $\bar{3}$. The Mo—Mo distances within the Mo₆ clusters are 2.6897 (7) Å for the intra-triangle distances (distances within the Mo₃ triangles formed by the Mo1 atoms related through the threefold axis) and 2.7609 (6) Å for the inter-triangle distances. In the sulfide, the two later values are slightly larger, *viz.* 2.693 (1) and 2.780 (5) Å. The Mo—Mo distances within the Mo₁₂ clusters are 2.6382 (5) and 2.6780 (7) Å for the distances in the triangles formed by the Mo2 and Mo3 atoms, respectively. In Tl₂Mo₉S₁₁, the corresponding distances are equal to 2.658 (1) and 2.688 (1) Å, respectively. The distances between the triangles formed by the Mo2 and Mo3 atoms are 2.7397 (7) and 2.7901 (7) Å and those between the Mo₃₃ triangles, 2.6796 (6) Å. The average Mo—Mo distance within the Mo₁₂ cluster is similar in Tl₂Mo₉S₁₁ and Tl₂Mo₉Se₁₁ and amounts to 2.705 Å. The Se atoms bridge either one (Se1, Se2, Se4 and Se5) or two (Se3) Mo triangular faces of the clusters. Moreover, the Se1 and Se2 atoms are linked to a Mo atom of a neighboring cluster. The Mo—Se bond distances range from 2.5269 (8) to 2.6247 (8) Å within the Mo₆Se^{*i*}₈Se^{*a*}₆ unit and from 2.5129 (8) to 2.7025 (8) Å within the Mo₁₂Se^{*i*}₁₄Se^{*a*}₆ unit. Each Mo₁₂Se^{*i*}₁₄Se^{*a*}₆ unit is interconnected to 6 Mo₆Se^{*i*}₈Se^{*a*}₆ units (and *vice versa*) via Mo2—Se1 bonds (respectively Mo1—Se2) to form the three-dimensional Mo—Se framework, the overall connectivity formula of which is Mo₁₂Se^{*i*}₈Se^{*i-a*}_{6/2}Se^{*a-i*}_{6/2}, Mo₆Se^{*i*}₂Se^{*i-a*}_{6/2}Se^{*a-i*}_{6/2}. It results from this arrangement that the shortest intercluster Mo1—Mo2 distance between the Mo₆ and Mo₁₂ clusters is 3.4239 (8) Å compared to 3.217 (1) in Tl₂Mo₉S₁₁, indicating only weak metal-metal interaction. The Tl atoms occupy large voids delimited by four Mo₆Se^{*i*}₈Se^{*a*}₆ units and four Mo₁₂Se^{*i*}₁₄Se^{*a*}₆ units. The Tl1 and Tl2 cations have a very similar environment which consists of seven Se atoms as nearest neighbors with Tl—Se distances in the ranges 3.0737 (10) - 3.5840 (6) Å and 3.0483 (9) - 3.4032 (6) Å for the Tl1 and Tl2 sites, respectively. These seven Se atoms form a monocapped octahedron compressed along the threefold axis. Three additional Se atoms at 4.3543 (6) and 4.2446 (6) Å from Tl1 and Tl2, respectively, are also observed (Figure 3). The

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average Tl—Se values are 3.72 and 3.54 Å for the Tl1 and Tl2 sites, respectively. Values of 3.57 and 3.68 Å are expected from the sum of the ionic radii for Se^{2-} and Tl^+ for coordination numbers 8 and 12 (Shannon, 1976).

Experimental

Single crystals of $\text{Tl}_2\text{Mo}_9\text{Se}_{11}$ were prepared from a mixture of MoSe_2 , TlSe and Mo with the nominal composition $\text{Tl}_2\text{Mo}_{12}\text{Se}_{14}$. Before use, Mo powder was reduced under H_2 flowing gas at 1273 K during ten hours in order to eliminate any trace of oxygen. The binaries MoSe_2 and TlSe were obtained by heating stoichiometric mixtures of the elements in sealed evacuated silica tubes during about 2 days at 1073 and 573 K, respectively. 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 48 h, then cooled at 100 K/h down to 1373 K and finally furnace cooled.

Refinement

The highest peak and the deepest hole in the final Fourier map are at 1.67 and 0.83 Å from Tl1 and Se5, respectively.

Figures

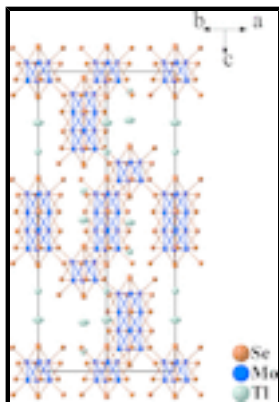


Fig. 1. : View of $\text{Tl}_2\text{Mo}_9\text{Se}_{11}$ along [110].

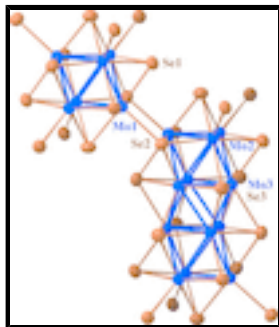


Fig. 2. : Plot showing the atom-numbering scheme and the linkage of the $\text{Mo}_{12}\text{Se}_{14}\text{Se}_6$ and $\text{Mo}_6\text{Se}_8\text{Se}_6$ cluster units. Displacement ellipsoids are drawn at the 97% probability level. Symmetry codes are as in Table 1.

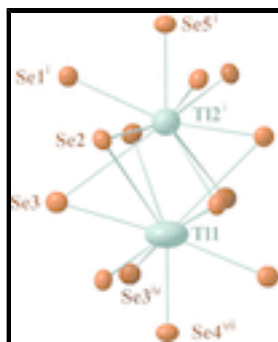


Fig. 3. : Environment of the Tl atoms. Symmetry codes are as in Table 1.

dithallium nonamolybdenum undecaselenide

Crystal data

$\text{Tl}_2\text{Mo}_9\text{Se}_{11}$

$M_r = 2140.8$

Trigonal, $R\bar{3}$

Hall symbol: $-R\ 3$

$a = 9.6212(1)\ \text{\AA}$

$c = 36.3316(7)\ \text{\AA}$

$V = 2912.55(7)\ \text{\AA}^3$

$Z = 6$

$F(000) = 5484$

$D_x = 7.321\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71069\ \text{\AA}$

Cell parameters from 10017 reflections

$\theta = 1\text{--}40.3^\circ$

$\mu = 42.73\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Irregular block, black

$0.13 \times 0.12 \times 0.11\ \text{mm}$

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube
horizontally mounted graphite crystal

Detector resolution: $9\ \text{pixels mm}^{-1}$

φ - and ω -scans

Absorption correction: multi-scan
(*PLATON*; Spek, 2009)

$T_{\min} = 0.033$, $T_{\max} = 0.108$

21097 measured reflections

4097 independent reflections

3156 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

$\theta_{\max} = 40.2^\circ$, $\theta_{\min} = 1.7^\circ$

$h = -16 \rightarrow 17$

$k = -16 \rightarrow 17$

$l = -58 \rightarrow 66$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.092$

$S = 1.37$

4097 reflections

0 constraints

Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0004I^2]$

$(\Delta/\sigma)_{\max} = 0.033$

$\Delta\rho_{\max} = 3.92\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -2.89\ \text{e \AA}^{-3}$

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69 parameters

Extinction correction: B-C type 1 Gaussian isotropic
(Becker & Coppens, 1974)

0 restraints

Extinction coefficient: 2320 (40)

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Tl1	0.333333	0.666667	0.495781 (14)	0.04933 (17)
Tl2	0	0	0.267672 (12)	0.02611 (11)
Mo1	-0.18022 (5)	0.31787 (5)	0.364749 (12)	0.01129 (13)
Mo2	0.16529 (5)	0.15027 (5)	0.406587 (12)	0.01120 (13)
Mo3	0.15606 (5)	-0.00891 (5)	0.469882 (12)	0.01087 (13)
Se1	-0.04118 (6)	0.29911 (6)	0.306626 (15)	0.01295 (17)
Se2	0.03067 (6)	0.32633 (6)	0.411871 (15)	0.01342 (17)
Se3	0.32150 (6)	0.30388 (6)	0.467628 (15)	0.01463 (17)
Se4	-0.333333	0.333333	0.41962 (2)	0.01559 (19)
Se5	0	0	0.35157 (2)	0.01474 (19)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Tl1	0.0620 (2)	0.0620 (2)	0.0239 (2)	0.03102 (12)	0	0
Tl2	0.02704 (13)	0.02704 (13)	0.0242 (2)	0.01352 (7)	0	0
Mo1	0.01119 (17)	0.01064 (17)	0.01168 (17)	0.00518 (13)	0.00061 (13)	-0.00028 (13)
Mo2	0.01129 (17)	0.01096 (17)	0.01107 (17)	0.00535 (14)	0.00023 (13)	0.00051 (13)
Mo3	0.01081 (16)	0.01111 (16)	0.01077 (17)	0.00553 (13)	0.00040 (13)	0.00014 (13)
Se1	0.0120 (2)	0.0136 (2)	0.0140 (2)	0.00691 (17)	0.00193 (16)	0.00090 (16)
Se2	0.0145 (2)	0.0121 (2)	0.0140 (2)	0.00685 (17)	-0.00087 (17)	0.00125 (16)
Se3	0.0121 (2)	0.0135 (2)	0.0151 (2)	0.00408 (17)	0.00057 (17)	0.00032 (17)
Se4	0.0181 (2)	0.0181 (2)	0.0106 (3)	0.00905 (12)	0	0
Se5	0.0167 (2)	0.0167 (2)	0.0108 (3)	0.00835 (11)	0	0

Geometric parameters (\AA , $^\circ$)

Tl1—Tl2 ⁱ	3.5164 (7)	Mo1—Mo1 ^{xiv}	2.7609 (7)
Tl1—Se2	4.3543 (6)	Mo1—Mo2 ^{viii}	3.4239 (8)
Tl1—Se2 ⁱⁱ	4.3543 (7)	Mo1—Se1	2.5540 (8)
Tl1—Se2 ⁱⁱⁱ	4.3543 (7)	Mo1—Se1 ^x	2.6143 (8)
Tl1—Se3	3.5840 (6)	Mo1—Se1 ^{xiv}	2.5757 (6)
Tl1—Se3 ⁱⁱ	3.5840 (5)	Mo1—Se2	2.6247 (8)
Tl1—Se3 ⁱⁱⁱ	3.5840 (8)	Mo1—Se4	2.5269 (8)
Tl1—Se3 ^{iv}	3.4547 (6)	Mo2—Mo2 ^{viii}	2.6382 (6)
Tl1—Se3 ^v	3.4547 (8)	Mo2—Mo2 ^{ix}	2.6382 (5)
Tl1—Se3 ^{vi}	3.4547 (5)	Mo2—Mo3	2.7397 (7)
Tl1—Se4 ^{vii}	3.0737 (10)	Mo2—Mo3 ^{viii}	2.7901 (7)
Tl2—Se1	3.4032 (7)	Mo2—Se1 ⁱ	2.6597 (6)

Tl2—Se1 ^{viii}	3.4032 (6)	Mo2—Se2	2.6034 (9)
Tl2—Se1 ^{ix}	3.4032 (8)	Mo2—Se2 ^{ix}	2.6124 (10)
Tl2—Se2 ⁱ	3.1441 (4)	Mo2—Se3	2.6721 (6)
Tl2—Se2 ^x	3.1441 (7)	Mo2—Se5	2.5129 (8)
Tl2—Se2 ^{xi}	3.1441 (7)	Mo3—Mo3 ^{viii}	2.6780 (9)
Tl2—Se3 ⁱ	4.2446 (7)	Mo3—Mo3 ^{ix}	2.6780 (7)
Tl2—Se3 ^x	4.2446 (6)	Mo3—Mo3 ^{xv}	2.6796 (6)
Tl2—Se3 ^{xi}	4.2446 (8)	Mo3—Mo3 ^{vi}	2.6796 (7)
Tl2—Se5	3.0483 (9)	Mo3—Se2 ^{ix}	2.5634 (8)
Mo1—Mo1 ^{xii}	2.6897 (9)	Mo3—Se3	2.6090 (6)
Mo1—Mo1 ^{xiii}	2.6897 (7)	Mo3—Se3 ^{ix}	2.6112 (6)
Mo1—Mo1 ^x	2.7609 (6)	Mo3—Se3 ^{xv}	2.7025 (8)
Mo1 ^{xii} —Mo1—Mo1 ^{xiii}	60.000 (18)	Mo2—Mo3—Mo3 ^{ix}	90.11 (2)
Mo1 ^{xii} —Mo1—Mo1 ^x	90	Mo2—Mo3—Mo3 ^{xv}	147.49 (3)
Mo1 ^{xii} —Mo1—Mo1 ^{xiv}	60.85 (2)	Mo2—Mo3—Mo3 ^{vi}	111.83 (2)
Mo1 ^{xiii} —Mo1—Mo1 ^x	60.849 (16)	Mo2 ^{ix} —Mo3—Mo3 ^{viii}	89.04 (2)
Mo1 ^{xiii} —Mo1—Mo1 ^{xiv}	90	Mo2 ^{ix} —Mo3—Mo3 ^{ix}	60.094 (17)
Mo1 ^x —Mo1—Mo1 ^{xiv}	58.302 (17)	Mo2 ^{ix} —Mo3—Mo3 ^{xv}	110.275 (17)
Mo2 ^{viii} —Mo2—Mo2 ^{ix}	60.00 (2)	Mo2 ^{ix} —Mo3—Mo3 ^{vi}	145.90 (3)
Mo2 ^{viii} —Mo2—Mo3	90.953 (18)	Mo3 ^{viii} —Mo3—Mo3 ^{ix}	60.000 (18)
Mo2 ^{viii} —Mo2—Mo3 ^{viii}	60.546 (16)	Mo3 ^{viii} —Mo3—Mo3 ^{xv}	90
Mo2 ^{ix} —Mo2—Mo3	62.472 (16)	Mo3 ^{viii} —Mo3—Mo3 ^{vi}	60.02 (2)
Mo2 ^{ix} —Mo2—Mo3 ^{viii}	89.850 (19)	Mo3 ^{ix} —Mo3—Mo3 ^{xv}	60.019 (16)
Mo3—Mo2—Mo3 ^{viii}	57.92 (2)	Mo3 ^{ix} —Mo3—Mo3 ^{vi}	90
Mo2—Mo3—Mo2 ^{ix}	56.983 (15)	Mo3 ^{xv} —Mo3—Mo3 ^{vi}	59.962 (18)
Mo2—Mo3—Mo3 ^{viii}	61.982 (19)		

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

Fig. 1

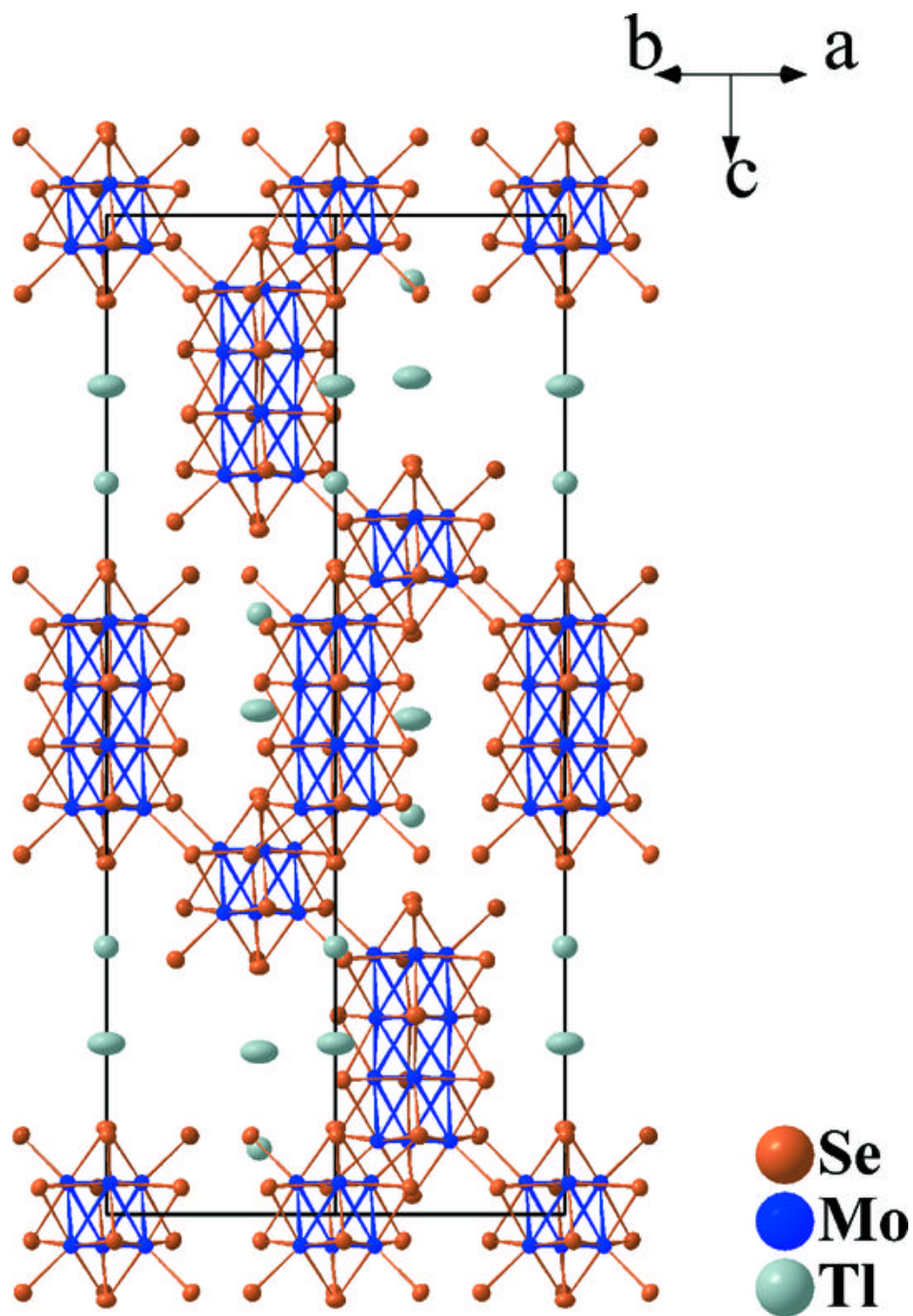


Fig. 2

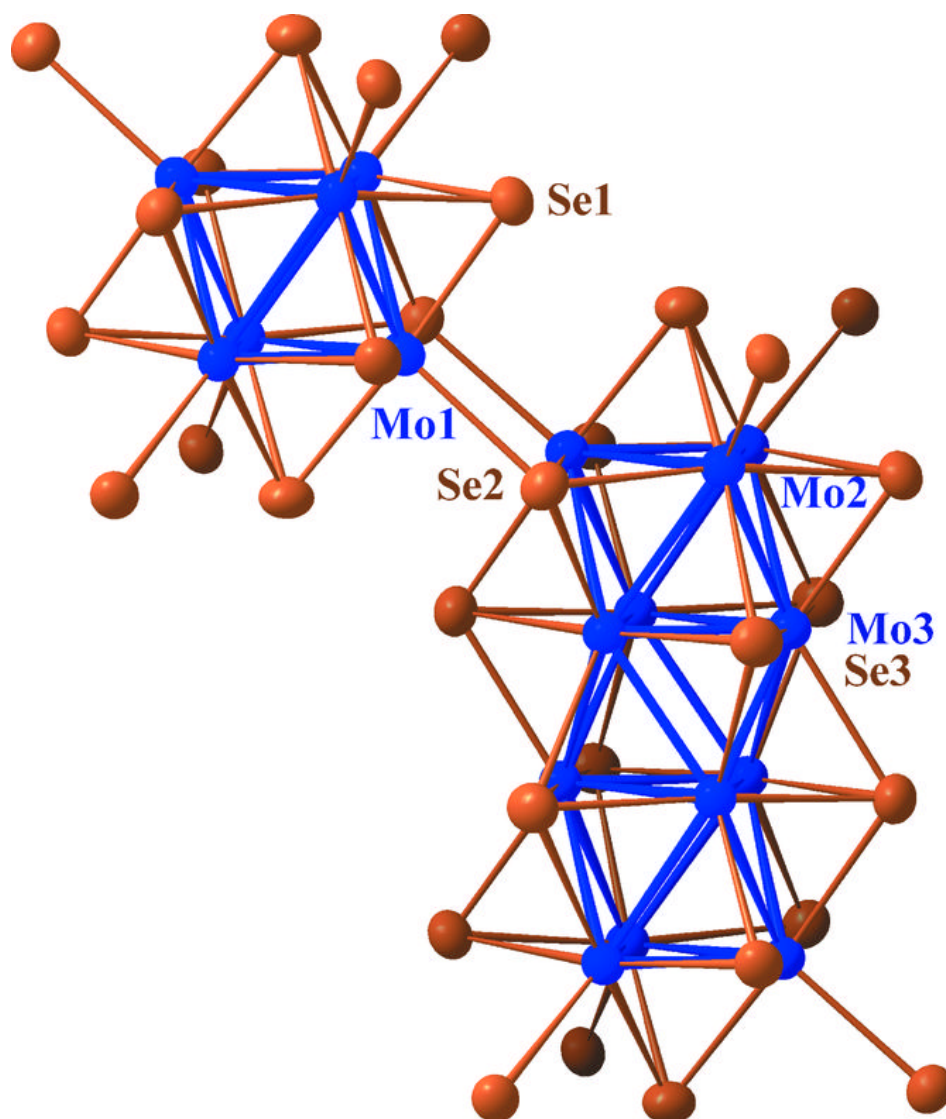


Fig. 3

