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V_{1.42}In_{1.83}Mo₁₅Se₁₉

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{Mo}–\text{Se}) = 0.001$ Å; disorder in main residue; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 35.7.

The structure of the title compound, vanadium indium pentadecamolybdenum nonadecaselelide, V_{1.42}In_{1.83}Mo₁₅Se₁₉, is isotopic with In_{2.9}Mo₁₅Se₁₉ [Grüttner *et al.* (1979). *Acta Cryst. B* **35**, 285–292]. It is characterized by two cluster units Mo₆Se₈Se₆^o and Mo₉Se₁₁Se₆ⁱ (where *i* represents inner and *a* apical atoms) that are present in a 1:1 ratio. The cluster units are centered at Wyckoff positions 2*b* and 2*c* and have point-group symmetry $\bar{3}$ and $\bar{6}$, respectively. The clusters are interconnected through additional Mo–Se bonds. In the title compound, the V³⁺ cations replace the trivalent indium atoms present in In_{2.9}Mo₁₅Se₁₉, and a deficiency is observed on the monovalent indium site. One Mo, one Se and the V atom are situated on mirror planes, and two other Se atoms and the In atom are situated on threefold rotation axes.

Related literature

For previous reports on the crystal structure of In_{~3}Mo₁₅Se₁₉ compounds, see: Grüttner *et al.* (1979). For physical properties of this type of compound, see: Seeber *et al.* (1979). The crystal structures of the substituted compounds Ho_{0.76}In_{1.68}Mo₁₅Se₁₉ and In_{0.87}K₂Mo₁₅Se₁₉ were reported by Salloum *et al.* (2006; 2007). For details of the *i*- and *a*-type ligand notation, see: Schäfer & von Schnering (1964).

Experimental

Crystal data

V_{1.42}In_{1.83}Mo₁₅Se₁₉
 $M_r = 3221.80$
Hexagonal, $P6_3/m$
 $a = 9.7361$ (1) Å
 $c = 19.3090$ (4) Å
 $V = 1585.11$ (4) Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 29.21$ mm⁻¹
 $T = 293$ K
 $0.09 \times 0.07 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: analytical
(de Meulenaar & Tompa, 1965)
 $T_{\min} = 0.161$, $T_{\max} = 0.329$
27560 measured reflections
2390 independent reflections
1634 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.093$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.087$
 $S = 1.08$
2390 reflections
67 parameters
 $\Delta\rho_{\max} = 3.22$ e Å⁻³
 $\Delta\rho_{\min} = -2.57$ e Å⁻³

Table 1

Selected bond lengths (Å).

Mo2–Se5	2.5259 (8)	Mo3–Se2	2.5631 (6)
Mo2–Se2	2.5974 (6)	Mo3–Se2 ^{vii}	2.5631 (6)
Mo2–Se2 ⁱ	2.6290 (6)	Mo3–Se3	2.5965 (9)
Mo2–Mo2 ⁱ	2.6474 (7)	Mo3–Se3 ⁱ	2.6095 (9)
Mo2–Se1 ⁱⁱ	2.6504 (6)	Mo3–Mo3 ^{viii}	2.7056 (10)
Mo2–Se3 ⁱ	2.6966 (6)	Mo3–Mo2 ^{ix}	2.7136 (5)
Mo2–Mo3 ⁱ	2.7136 (5)	In1–Se5	3.0759 (15)
Mo2–Mo3	2.7557 (5)	In1–Se2 ^x	3.1221 (5)
Mo1–Se1 ⁱⁱⁱ	2.5467 (7)	In1–Se1 ^x	3.4904 (7)
Mo1–Se4	2.5481 (8)	V1–Se4	2.510 (2)
Mo1–Se1	2.5717 (6)	V1–Se3 ^{iv}	2.730 (3)
Mo1–Se1 ^{iv}	2.6111 (6)	V1–Se2 ^v	2.7981 (18)
Mo1–Se2	2.6378 (7)	V1–Se3 ^v	2.831 (3)
Mo1–Mo1 ^v	2.6992 (7)	V1–Mo3 ^v	2.865 (3)
Mo1–Mo1 ^{vi}	2.7223 (8)		

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

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: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Bergerhoff, 1996); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2403).

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supplementary materials

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V_{1.42}In_{1.83}Mo₁₅Se₁₉

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Comment

From the point of view of crystal chemistry and physical properties, the reduced molybdenum selenides In₃Mo₁₅Se₁₉ (Grüttner *et al.*, 1979) constitute an interesting family of compounds. Indeed, their crystal structure contains an equal mixture of Mo₆ and Mo₉ cluster units and the In atoms occupy two crystallographically different positions depending on their formal oxidation state of +1 or +3. Recently, we have shown that the In³⁺ cation can be replaced by other trivalent cations such as Ho³⁺ (Salloum *et al.*, 2006) and the In⁺ cation by K⁺ (Salloum *et al.*, 2007). Interest in these Mo cluster compounds also lies in their physical properties, because they become superconductors with high critical magnetic fields at about 4 K (Seeber *et al.*, 1979). We present here the crystal structure of V_{1.42}In_{1.83}Mo₁₅Se₁₉ in which a 3d element replaces the trivalent indium atom.

The Mo—Se framework of the title compound consists of the cluster units Mo₆Se^{*i*}₈Se^{*a*}₆ and Mo₉Se^{*i*}₁₁Se^{*a*}₆ in a 1:1 ratio (for details of the *i*- and *a*-type ligand notation, see: Schäfer & von Schnering (1964)). Both cluster units are interconnected through additional Mo—Se bonds (Figs. 1 and 2). The first unit can be described as an Mo₆ octahedron surrounded by eight face-capping inner Se^{*i*} and six apical Se^{*a*} ligands. The Mo₉ cluster is surrounded by 11 Se^{*i*} atoms capping one or two faces of the bioctahedron and six Se^{*a*} ligands above the apical Mo atoms. The Mo₆Se^{*i*}₈Se^{*a*}₆ and Mo₉Se^{*i*}₁₁Se^{*a*}₆ units are centered at Wyckoff positions 2*b* and 2*c* and have point-group symmetry $\bar{3}$ and $\bar{6}$, respectively. The Mo—Mo distances within the Mo₆ cluster are 2.6992 (7) Å for the distances of the Mo triangles formed by the Mo1 atoms related through the threefold axis, and 2.7223 (8) Å for the distances between these triangles. The Mo—Mo distances within the Mo₉ clusters are 2.6474 (7) and 2.7056 (10) Å in the triangles formed by the atoms Mo2 and Mo3, respectively, and 2.7136 (5) and 2.7557 (5) Å for those between the Mo₂₃ and Mo₃₃ triangles. The Se atoms bridge either one (Se1, Se2, Se4 and Se5) or two (Se3) triangular faces of the Mo clusters. Moreover, atoms Se1 and Se2 are linked to an Mo atom of a neighboring cluster. The Mo—Se bond lengths range from 2.5467 (7) to 2.6378 (7) Å within the Mo₆Se^{*i*}₈Se^{*a*}₆ unit, and from 2.5259 (8) to 2.6966 (6) Å within the Mo₉Se^{*i*}₁₁Se^{*a*}₆ unit. Each Mo₉Se^{*i*}₁₁Se^{*a*}₆ cluster is interconnected by six Mo₆Se^{*i*}₈Se^{*a*}₆ units (and vice versa) via Mo2—Se1 bonds (and Mo1—Se2 bonds, respectively), forming the three-dimensional Mo—Se framework, the connectivity formula of which is Mo₉Se^{*i*}₅Se^{*i-a*}_{6/2}Se^{*-ai*}_{6/2}, Mo₆Se^{*i*}₂Se^{*i-a*}_{6/2}Se^{*-ai*}_{6/2}. It results from this arrangement that the shortest intercluster Mo1—Mo2 distance is 3.4216 (6) Å, indicating only weak metal—metal interaction. The In⁺ cations are surrounded by seven Se atoms forming a distorted tricapped tetrahedron, as is the case in In_{2.9}Mo₁₅Se₁₉. The Se5 and Se2 atoms forming the tetrahedron are at 3.0759 (15) and 3.1221 (5) Å from the In atom, and the capping Se1 atoms are at 3.4904 (7) Å. The V³⁺ cations, as the In³⁺ cations in the In₃Mo₁₅Se₁₉ compounds, occupy partially at 47.4 (6)% a triangular group of distorted octahedral cavities, which are formed by two Mo₆Se^{*i*}₈Se^{*a*}₆ and three Mo₉Se^{*i*}₁₁Se^{*a*}₆ units, around the threefold rotation axis. The V—Se distances are in the 2.510 (2) - 2.831 (3) Å range.

Experimental

Single crystals of $V_{1.42}In_{1.83}Mo_{15}Se_{19}$ were prepared from a mixture of V_2Se_3 , $MoSe_2$, $InSe$ and Mo with a nominal composition $V_{1.5}In_2Mo_{15}Se_{19}$. 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 V_2Se_3 , $MoSe_2$, $InSe$ were obtained by heating stoichiometric mixtures of the elements in sealed evacuated silica tubes during about 2 days. 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 hours, then cooled at 100 K/h down to 1373 K and finally furnace cooled.

Refinement

The highest peak and the deepest hole are located 1.56 Å and 0.66 Å from Mo_3 , respectively. Refinement of the occupancy factors of the V and In atoms led to the final composition $V_{1.42(2)}In_{1.832(8)}Mo_{15}Se_{19}$.

Figures

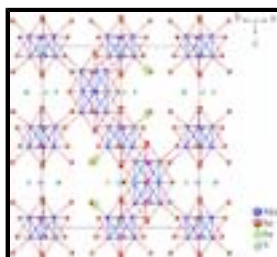


Fig. 1. View of $V_{1.42}In_{1.83}Mo_{15}Se_{19}$ along [110]. Displacement ellipsoids are drawn at the 97% probability level.

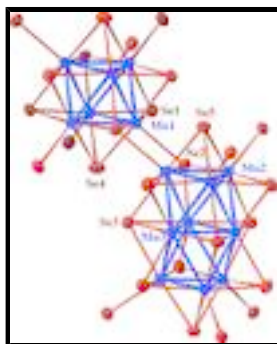


Fig. 2. Plot showing the atom-numbering scheme and the interunit linkage of the $Mo_9Se_{11}Se_6$ and $Mo_6Se_8Se_6$ cluster units. Displacement ellipsoids are drawn at the 97% probability level.

vanadium indium pentadecamolybdenum nonadecaselenide

Crystal data

$V_{1.42}In_{1.83}Mo_{15}Se_{19}$

$M_r = 3221.80$

Hexagonal, $P6_3/m$

Hall symbol: $-P\ 6c$

$a = 9.7361(1)\ \text{\AA}$

$c = 19.3090(4)\ \text{\AA}$

$D_x = 6.750\ \text{Mg m}^{-3}$

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

Cell parameters from 26495 reflections

$\theta = 2.6\text{--}35.0^\circ$

$\mu = 29.21\ \text{mm}^{-1}$

$T = 293\ \text{K}$

$V = 1585.11 (4) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 2797$

Irregular block, black
 $0.09 \times 0.07 \times 0.05 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 Radiation source: fine-focus sealed tube graphite
 φ scans ($\kappa = 0$) + additional ω scans
 Absorption correction: analytical (de Meulenaar & Tompa, 1965)
 $T_{\min} = 0.161$, $T_{\max} = 0.329$
 27560 measured reflections

2390 independent reflections
 1634 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.093$
 $\theta_{\max} = 35.0^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -15 \rightarrow 15$
 $k = -15 \rightarrow 12$
 $l = -30 \rightarrow 31$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.087$
 $S = 1.08$
 2390 reflections
 67 parameters
 0 restraints

Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 $w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.9587P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 3.22 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -2.57 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 2008),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.00025 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Mo1	0.16776 (5)	0.01669 (5)	0.55780 (2)	0.00914 (10)	
Mo2	0.68407 (5)	0.18577 (5)	0.63317 (2)	0.00965 (10)	
Mo3	0.51292 (7)	0.16694 (7)	0.7500	0.00868 (11)	

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Se1	0.03647 (6)	-0.28702 (6)	0.55134 (3)	0.01070 (11)	
Se2	0.37798 (6)	0.00665 (6)	0.64065 (3)	0.01233 (12)	
Se3	0.34594 (8)	0.30753 (9)	0.7500	0.01280 (15)	
Se4	0.0000	0.0000	0.66221 (5)	0.01643 (19)	
Se5	0.6667	0.3333	0.52902 (5)	0.01225 (17)	
In1	0.6667	0.3333	0.36972 (6)	0.0374 (4)	0.916 (4)
V1	-0.2026 (4)	-0.1744 (3)	0.7500	0.0177 (9)	0.474 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00996 (18)	0.00853 (18)	0.0089 (2)	0.00456 (15)	0.00070 (14)	-0.00012 (14)
Mo2	0.00995 (18)	0.00993 (18)	0.0089 (2)	0.00488 (15)	0.00013 (14)	-0.00037 (14)
Mo3	0.0096 (2)	0.0099 (3)	0.0071 (3)	0.0053 (2)	0.000	0.000
Se1	0.0110 (2)	0.0097 (2)	0.0116 (2)	0.00543 (19)	0.00107 (17)	0.00229 (17)
Se2	0.0115 (2)	0.0119 (2)	0.0138 (3)	0.00598 (18)	-0.00305 (18)	-0.00208 (18)
Se3	0.0121 (3)	0.0158 (3)	0.0117 (3)	0.0079 (3)	0.000	0.000
Se4	0.0203 (3)	0.0203 (3)	0.0086 (4)	0.01016 (14)	0.000	0.000
Se5	0.0142 (2)	0.0142 (2)	0.0083 (4)	0.00712 (12)	0.000	0.000
In1	0.0345 (4)	0.0345 (4)	0.0431 (7)	0.0172 (2)	0.000	0.000
V1	0.0216 (16)	0.0095 (13)	0.0165 (16)	0.0038 (11)	0.000	0.000

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

Mo2—Se5	2.5259 (8)	Mo3—Mo3 ⁱⁱ	2.7056 (10)
Mo2—Se2	2.5974 (6)	Mo3—Mo3 ⁱ	2.7056 (10)
Mo2—Se2 ⁱ	2.6290 (6)	Mo3—Mo2 ^{ix}	2.7136 (5)
Mo2—Mo2 ⁱ	2.6474 (7)	Mo3—Mo2 ⁱⁱ	2.7136 (5)
Mo2—Mo2 ⁱⁱ	2.6474 (7)	Mo3—Mo2 ^{viii}	2.7557 (5)
Mo2—Se1 ⁱⁱⁱ	2.6504 (6)	In1—Se5	3.0759 (15)
Mo2—Se3 ⁱ	2.6966 (6)	In1—Se2 ^x	3.1221 (5)
Mo2—Mo3 ⁱ	2.7136 (5)	In1—Se2 ^{xi}	3.1221 (5)
Mo2—Mo3	2.7557 (5)	In1—Se2 ^{iv}	3.1221 (5)
Mo1—Se1 ^{iv}	2.5467 (7)	In1—Se1 ^x	3.4904 (7)
Mo1—Se4	2.5481 (8)	In1—Se1 ^{xi}	3.4904 (7)
Mo1—Se1	2.5717 (6)	In1—Se1 ^{iv}	3.4904 (7)
Mo1—Se1 ^v	2.6111 (6)	In1—Se3 ^{vii}	4.2444 (9)
Mo1—Se2	2.6378 (7)	In1—Se3 ^{xii}	4.2444 (9)
Mo1—Mo1 ^{vi}	2.6992 (7)	In1—Se3 ^{xiii}	4.2444 (9)
Mo1—Mo1 ^v	2.6992 (7)	V1—Se4	2.510 (2)
Mo1—Mo1 ^{vii}	2.7223 (8)	V1—Se4 ^{viii}	2.510 (2)
Mo1—Mo1 ^{iv}	2.7223 (8)	V1—Se3 ^v	2.730 (3)
Mo3—Se2	2.5631 (6)	V1—Se2 ^{vi}	2.7981 (18)
Mo3—Se2 ^{viii}	2.5631 (6)	V1—Se2 ^{xiv}	2.7981 (18)
Mo3—Se3	2.5965 (9)	V1—Se3 ^{vi}	2.831 (3)

Mo3—Se3 ⁱ	2.6095 (9)	V1—Mo3 ^{vi}	2.865 (3)
Se5—Mo2—Se2	92.603 (17)	Se2 ^{viii} —Mo3—Mo3 ⁱ	117.84 (2)
Se5—Mo2—Se2 ⁱ	91.858 (17)	Se3—Mo3—Mo3 ⁱ	118.92 (3)
Se2—Mo2—Se2 ⁱ	173.59 (3)	Se3 ⁱ —Mo3—Mo3 ⁱ	58.45 (3)
Se5—Mo2—Mo2 ⁱ	58.396 (13)	Mo3 ⁱⁱ —Mo3—Mo3 ⁱ	60.0
Se2—Mo2—Mo2 ⁱ	120.06 (2)	Se2—Mo3—Mo2 ^{ix}	149.88 (3)
Se2 ⁱ —Mo2—Mo2 ⁱ	58.978 (19)	Se2 ^{viii} —Mo3—Mo2 ^{ix}	59.685 (15)
Se5—Mo2—Mo2 ⁱⁱ	58.396 (13)	Se3—Mo3—Mo2 ^{ix}	60.991 (14)
Se2—Mo2—Mo2 ⁱⁱ	60.16 (2)	Se3 ⁱ —Mo3—Mo2 ^{ix}	118.165 (16)
Se2 ⁱ —Mo2—Mo2 ⁱⁱ	118.878 (19)	Mo3 ⁱⁱ —Mo3—Mo2 ^{ix}	61.129 (16)
Mo2 ⁱ —Mo2—Mo2 ⁱⁱ	60.0	Mo3 ⁱ —Mo3—Mo2 ^{ix}	89.824 (14)
Se5—Mo2—Se1 ⁱⁱⁱ	90.42 (2)	Se2—Mo3—Mo2 ⁱⁱ	59.685 (15)
Se2—Mo2—Se1 ⁱⁱⁱ	86.03 (2)	Se2 ^{viii} —Mo3—Mo2 ⁱⁱ	149.88 (3)
Se2 ⁱ —Mo2—Se1 ⁱⁱⁱ	98.54 (2)	Se3—Mo3—Mo2 ⁱⁱ	60.991 (14)
Mo2 ⁱ —Mo2—Se1 ⁱⁱⁱ	137.757 (18)	Se3 ⁱ —Mo3—Mo2 ⁱⁱ	118.165 (16)
Mo2 ⁱⁱ —Mo2—Se1 ⁱⁱⁱ	129.74 (2)	Mo3 ⁱⁱ —Mo3—Mo2 ⁱⁱ	61.129 (16)
Se5—Mo2—Se3 ⁱ	175.68 (2)	Mo3 ⁱ —Mo3—Mo2 ⁱⁱ	89.824 (14)
Se2—Mo2—Se3 ⁱ	85.76 (2)	Mo2 ^{ix} —Mo3—Mo2 ⁱⁱ	112.47 (3)
Se2 ⁱ —Mo2—Se3 ⁱ	89.46 (2)	Se2—Mo3—Mo2 ^{viii}	145.46 (3)
Mo2 ⁱ —Mo2—Se3 ⁱ	119.150 (18)	Se2 ^{viii} —Mo3—Mo2 ^{viii}	58.328 (15)
Mo2 ⁱⁱ —Mo2—Se3 ⁱ	117.432 (18)	Se3—Mo3—Mo2 ^{viii}	118.823 (15)
Se1 ⁱⁱⁱ —Mo2—Se3 ⁱ	93.45 (2)	Se3 ⁱ —Mo3—Mo2 ^{viii}	60.271 (14)
Se5—Mo2—Mo3 ⁱ	120.23 (2)	Mo3 ⁱⁱ —Mo3—Mo2 ^{viii}	88.941 (14)
Se2—Mo2—Mo3 ⁱ	116.35 (2)	Mo3 ⁱ —Mo3—Mo2 ^{viii}	59.578 (16)
Se2 ⁱ —Mo2—Mo3 ⁱ	57.311 (17)	Mo2 ^{ix} —Mo3—Mo2 ^{viii}	57.893 (17)
Mo2 ⁱ —Mo2—Mo3 ⁱ	61.851 (15)	Mo2 ⁱⁱ —Mo3—Mo2 ^{viii}	146.06 (3)
Mo2 ⁱⁱ —Mo2—Mo3 ⁱ	91.063 (14)	Se2—Mo3—Mo2	58.328 (15)
Se1 ⁱⁱⁱ —Mo2—Mo3 ⁱ	138.90 (2)	Se2 ^{viii} —Mo3—Mo2	145.46 (3)
Se3 ⁱ —Mo2—Mo3 ⁱ	57.36 (2)	Se3—Mo3—Mo2	118.823 (15)
Se5—Mo2—Mo3	118.64 (2)	Se3 ⁱ —Mo3—Mo2	60.271 (14)
Se2—Mo2—Mo3	57.123 (17)	Mo3 ⁱⁱ —Mo3—Mo2	88.941 (14)
Se2 ⁱ —Mo2—Mo3	116.58 (2)	Mo3 ⁱ —Mo3—Mo2	59.578 (16)
Mo2 ⁱ —Mo2—Mo3	90.142 (14)	Mo2 ^{ix} —Mo3—Mo2	146.06 (3)
Mo2 ⁱⁱ —Mo2—Mo3	60.255 (15)	Mo2 ⁱⁱ —Mo3—Mo2	57.893 (17)
Se1 ⁱⁱⁱ —Mo2—Mo3	131.66 (2)	Mo2 ^{viii} —Mo3—Mo2	109.90 (3)
Se3 ⁱ —Mo2—Mo3	57.177 (19)	Mo1 ^{vii} —Se1—Mo1	64.26 (2)
Mo3 ⁱ —Mo2—Mo3	59.29 (2)	Mo1 ^{vii} —Se1—Mo1 ^{vi}	63.70 (2)
Se1 ^{iv} —Mo1—Se4	176.34 (2)	Mo1—Se1—Mo1 ^{vi}	62.77 (2)
Se1 ^{iv} —Mo1—Se1	88.948 (17)	Mo1 ^{vii} —Se1—Mo2 ^{xv}	131.23 (2)
Se4—Mo1—Se1	91.765 (17)	Mo1—Se1—Mo2 ^{xv}	128.19 (2)

supplementary materials

Se1 ^{iv} —Mo1—Se1 ^v	88.084 (17)	Mo1 ^{vi} —Se1—Mo2 ^{xv}	81.114 (19)
Se4—Mo1—Se1 ^v	90.859 (17)	Mo3—Se2—Mo2	64.549 (18)
Se1—Mo1—Se1 ^v	173.79 (3)	Mo3—Se2—Mo2 ⁱⁱ	63.004 (18)
Se1 ^{iv} —Mo1—Se2	93.50 (2)	Mo2—Se2—Mo2 ⁱⁱ	60.86 (2)
Se4—Mo1—Se2	90.13 (2)	Mo3—Se2—Mo1	130.39 (2)
Se1—Mo1—Se2	86.306 (19)	Mo2—Se2—Mo1	126.33 (2)
Se1 ^v —Mo1—Se2	99.32 (2)	Mo2 ⁱⁱ —Se2—Mo1	81.018 (19)
Se1 ^{iv} —Mo1—Mo1 ^{vi}	119.568 (17)	Mo3—Se3—Mo3 ⁱⁱ	62.62 (3)
Se4—Mo1—Mo1 ^{vi}	58.018 (12)	Mo3—Se3—Mo2 ^{ix}	61.649 (16)
Se1—Mo1—Mo1 ^{vi}	59.33 (2)	Mo3 ⁱⁱ —Se3—Mo2 ^{ix}	62.551 (16)
Se1 ^v —Mo1—Mo1 ^{vi}	117.827 (19)	Mo3—Se3—Mo2 ⁱⁱ	61.649 (16)
Se2—Mo1—Mo1 ^{vi}	129.11 (2)	Mo3 ⁱⁱ —Se3—Mo2 ⁱⁱ	62.551 (16)
Se1 ^{iv} —Mo1—Mo1 ^v	118.595 (17)	Mo2 ^{ix} —Se3—Mo2 ⁱⁱ	113.56 (3)
Se4—Mo1—Mo1 ^v	58.018 (12)	Mo1 ^{vi} —Se4—Mo1	63.96 (2)
Se1—Mo1—Mo1 ^v	119.25 (2)	Mo1 ^{vi} —Se4—Mo1 ^v	63.96 (2)
Se1 ^v —Mo1—Mo1 ^v	57.903 (19)	Mo1—Se4—Mo1 ^v	63.96 (2)
Se2—Mo1—Mo1 ^v	137.270 (18)	Mo2 ⁱ —Se5—Mo2	63.21 (3)
Mo1 ^{vi} —Mo1—Mo1 ^v	60.0	Mo2 ⁱ —Se5—Mo2 ⁱⁱ	63.21 (3)
Se1 ^{iv} —Mo1—Mo1 ^{vii}	59.30 (2)	Mo2—Se5—Mo2 ⁱⁱ	63.21 (3)
Se4—Mo1—Mo1 ^{vii}	118.273 (15)	Mo2 ⁱ —Se5—In1	142.763 (16)
Se1—Mo1—Mo1 ^{vii}	57.423 (15)	Mo2—Se5—In1	142.763 (16)
Se1 ^v —Mo1—Mo1 ^{vii}	116.42 (2)	Mo2 ⁱⁱ —Se5—In1	142.763 (16)
Se2—Mo1—Mo1 ^{vii}	132.182 (19)	Se4 ^{viii} —V1—Se4	84.98 (9)
Mo1 ^{vi} —Mo1—Mo1 ^{vii}	60.281 (10)	Se4 ^{viii} —V1—Se3 ^v	87.27 (7)
Mo1 ^v —Mo1—Mo1 ^{vii}	90.0	Se4—V1—Se3 ^v	87.27 (7)
Se1 ^{iv} —Mo1—Mo1 ^{iv}	58.31 (2)	Se4 ^{viii} —V1—Se2 ^{vi}	166.76 (13)
Se4—Mo1—Mo1 ^{iv}	118.273 (15)	Se4—V1—Se2 ^{vi}	87.36 (2)
Se1—Mo1—Mo1 ^{iv}	116.86 (2)	Se3 ^v —V1—Se2 ^{vi}	103.16 (8)
Se1 ^v —Mo1—Mo1 ^{iv}	56.998 (14)	Se4 ^{viii} —V1—Se2 ^{xiv}	87.36 (2)
Se2—Mo1—Mo1 ^{iv}	140.74 (2)	Se4—V1—Se2 ^{xiv}	166.76 (13)
Mo1 ^{vi} —Mo1—Mo1 ^{iv}	90.0	Se3 ^v —V1—Se2 ^{xiv}	103.16 (8)
Mo1 ^v —Mo1—Mo1 ^{iv}	60.281 (10)	Se2 ^{vi} —V1—Se2 ^{xiv}	97.98 (8)
Mo1 ^{vii} —Mo1—Mo1 ^{iv}	59.44 (2)	Se4 ^{viii} —V1—Se3 ^{vi}	85.09 (8)
Se2—Mo3—Se2 ^{viii}	110.93 (3)	Se4—V1—Se3 ^{vi}	85.09 (8)
Se2—Mo3—Se3	93.18 (2)	Se3 ^v —V1—Se3 ^{vi}	169.63 (12)
Se2 ^{viii} —Mo3—Se3	93.18 (2)	Se2 ^{vi} —V1—Se3 ^{vi}	83.50 (6)
Se2—Mo3—Se3 ⁱ	88.30 (2)	Se2 ^{xiv} —V1—Se3 ^{vi}	83.50 (6)
Se2 ^{viii} —Mo3—Se3 ⁱ	88.30 (2)	Se4 ^{viii} —V1—Mo3 ^{vi}	122.97 (8)
Se3—Mo3—Se3 ⁱ	177.38 (3)	Se4—V1—Mo3 ^{vi}	122.97 (8)
Se2—Mo3—Mo3 ⁱⁱ	120.789 (19)	Se3 ^v —V1—Mo3 ^{vi}	136.14 (12)
Se2 ^{viii} —Mo3—Mo3 ⁱⁱ	120.789 (19)	Se2 ^{vi} —V1—Mo3 ^{vi}	53.80 (4)

Se ³ —Mo ³ —Mo ³ ⁱⁱ	58.92 (3)	Se ² ^{xiv} —V ¹ —Mo ³ ^{vi}	53.80 (4)
Se ³ ⁱ —Mo ³ —Mo ³ ⁱⁱ	118.45 (3)	Se ³ ^{vi} —V ¹ —Mo ³ ^{vi}	54.23 (5)
Se ² —Mo ³ —Mo ³ ⁱ	117.84 (2)		

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

Fig. 1

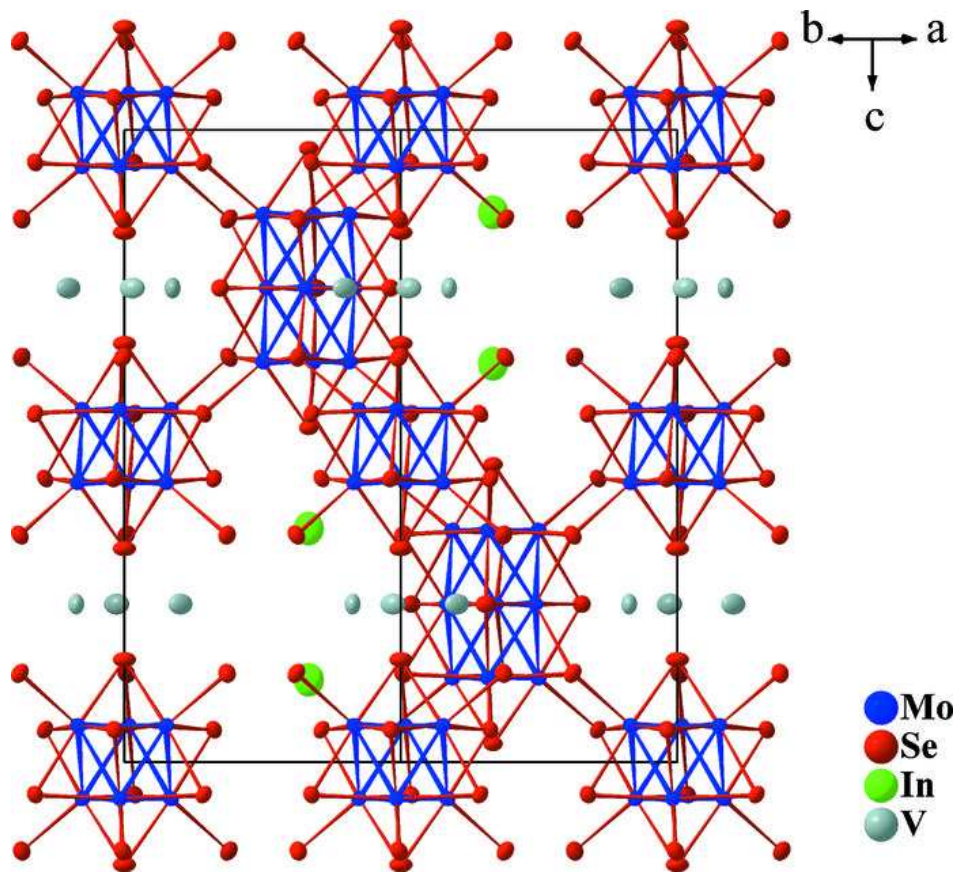


Fig. 2

