

**Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub>, a monoclinic variant of the hexagonal In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> type**

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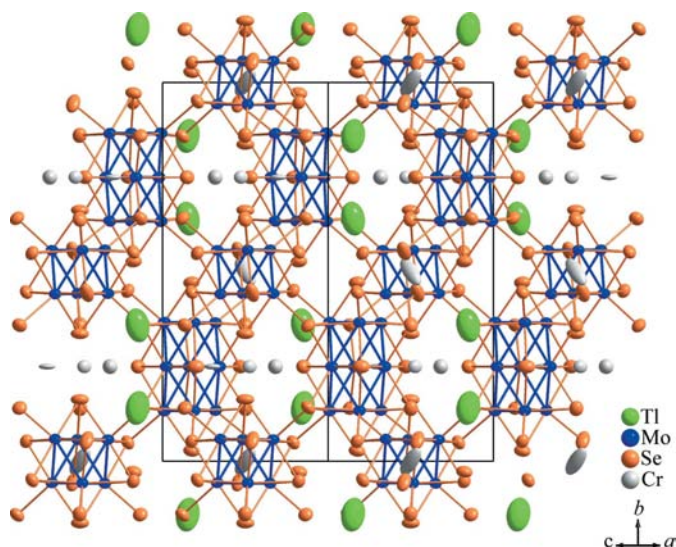
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The monoclinic compound Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub> (chromium thallium pentadecamolybdenum nonadecaselenide) represents a variant of the hexagonal In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> structure type. Its crystal structure consists of an equal mixture of Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> and Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> cluster units. The Mo and Se atoms of the median plane of the Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> unit, as well as three Cr ions, lie on sites with *m* symmetry (Wyckoff site 2*e*). The fourth Cr ion is in a 2*b* Wyckoff position with  $\bar{1}$  site symmetry.

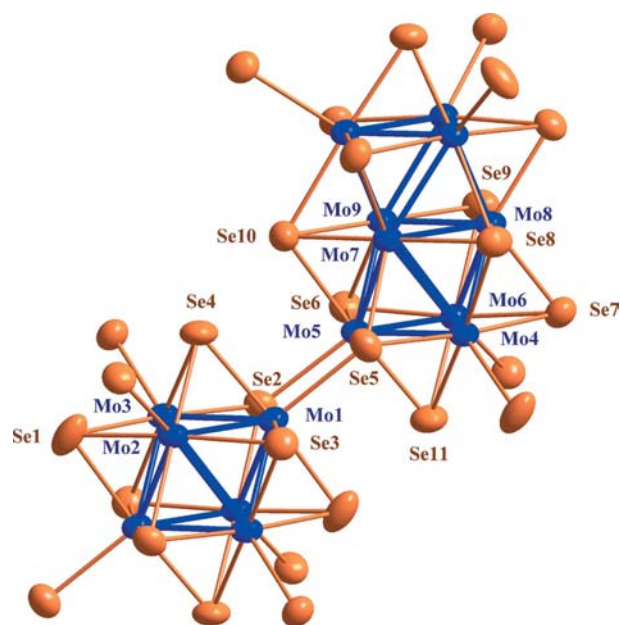
**Comment**

Thirty years ago, Grüttner *et al.* (1979) reported the crystal structures of the hexagonal compounds In<sub>2.9</sub>Mo<sub>15</sub>Se<sub>19</sub> and In<sub>3.3</sub>Mo<sub>15</sub>Se<sub>19</sub>, which were the first compounds containing a transition metal cluster with a nuclearity higher than six, namely the bioctahedral Mo<sub>9</sub> cluster. This cluster, which results from the face-sharing of two Mo<sub>6</sub> octahedra, co-exists with the octahedral Mo<sub>6</sub> cluster in equal proportions. Both clusters are surrounded by Se atoms to form Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> and Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> units that share some of their Se atoms to create the three-dimensional Mo–Se framework. The In atoms occupy two crystallographically different positions, depending on their formal oxidation state of +1 or +3. While the In<sup>+</sup> site is fully occupied, the In<sup>3+</sup> site presents a nonstoichiometry that leads to compositions ranging from In<sub>0.9</sub>In<sub>2</sub><sup>III</sup>Mo<sub>15</sub>Se<sub>19</sub> to In<sub>1.3</sub>In<sub>2</sub><sup>III</sup>Mo<sub>15</sub>Se<sub>19</sub>. Interest in these Mo cluster compounds lies not only in their structural aspects but also in their physical properties, because they show superconductivity with high critical magnetic fields at about 4 K. We present here the synthesis and the crystal structure of Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub>, which constitutes a monoclinic variant of the In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> structure type.

A view of the crystal structure of Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub> is shown in Fig. 1. The Mo–Se framework is similar to that of the In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> compounds and consists of an equal mixture of Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> and Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> cluster units interconnected through Mo–Se bonds (Fig. 2). The first unit can be described

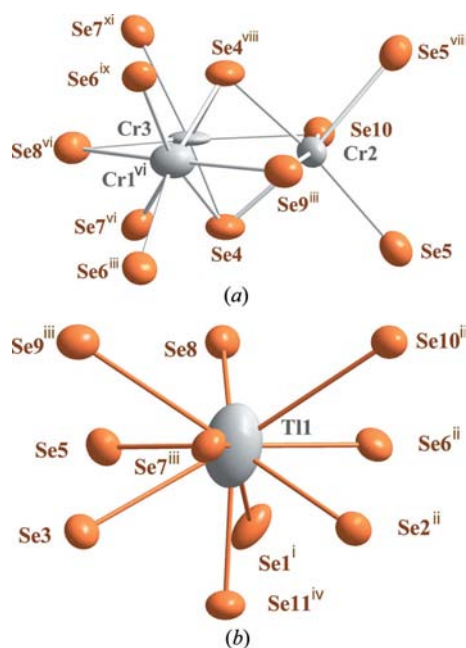
**Figure 1**

A view of Cr<sub>1.45</sub>(2)Tl<sub>1.870(4)</sub>Mo<sub>15</sub>Se<sub>19</sub>, along [101]. Displacement ellipsoids are drawn at the 97% probability level.

**Figure 2**

Plot showing the atom-numbering scheme and the inter-unit linkage of the Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> and Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> cluster units. Displacement ellipsoids are drawn at the 97% probability level.

as an Mo<sub>6</sub> octahedron surrounded by eight face-capping inner Se<sup>i</sup> and six apical Se<sup>a</sup> ligands; for details of the i- and a-type ligand notation, see Schäfer & Schnering (1964). The Mo<sub>9</sub> core of the second unit results from the one-dimensional *trans* face-sharing of two octahedral Mo<sub>6</sub> clusters. The Mo<sub>9</sub> cluster is surrounded by 11 Se<sup>i</sup> atoms capping the faces of the dioctahedron and six apical Se<sup>a</sup> ligands above the terminal Mo atoms. The Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> and Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> units are centred at 2*a* and 2*e* Wyckoff positions and thus have point group symmetries  $\bar{1}$  and *m*, respectively, instead of  $\bar{3}$ .. and  $\bar{6}$ .. in the hexagonal In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> compounds. The Mo–Mo distances within the Mo<sub>6</sub> clusters are between 2.6769 (8) and 2.7252 (7) Å, and

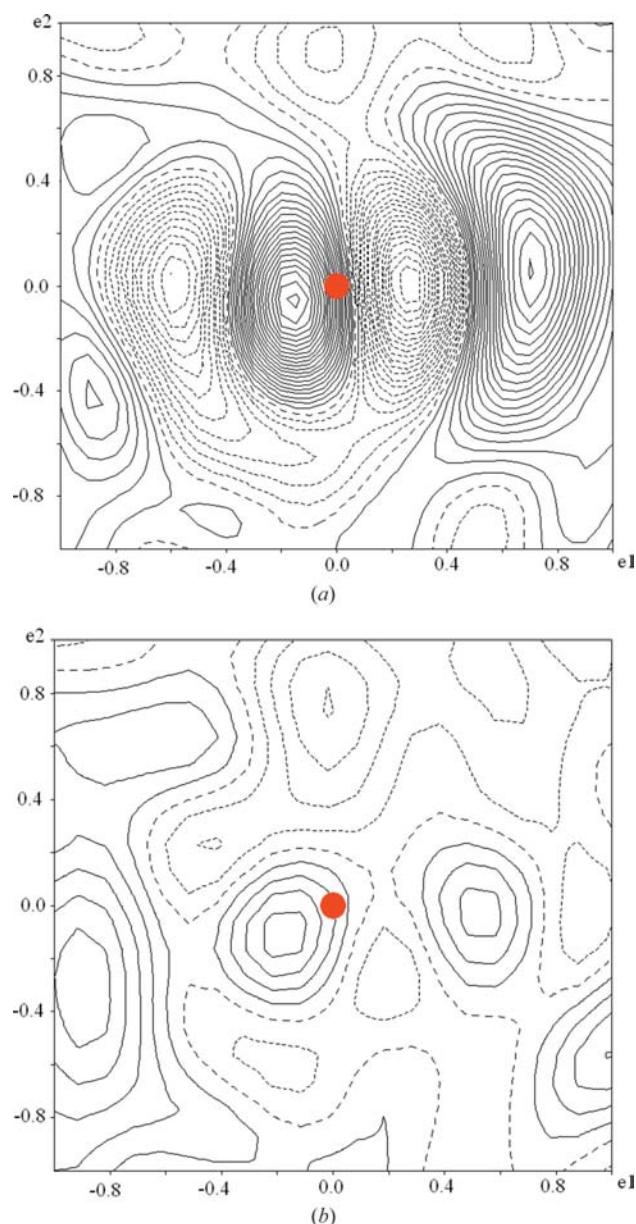


**Figure 3**  
 (a) The distribution of Cr1, Cr2 and Cr3. (b) The environment of TII. [Symmetry codes are given in Table 1. Additionally: (viii)  $x, -y + \frac{1}{2}, z$ ; (ix)  $x + 1, -y + \frac{1}{2}, z$ ; (xi)  $x + 1, -y + \frac{1}{2}, z + 1$ .]

those within the Mo<sub>9</sub> clusters are between 2.6278 (9) and 2.7491 (7) Å. These values are comparable with those observed in the In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> compounds.

The Se atoms bridge either one (Se1 to Se7) or two (Se8, Se9 and Se10) Mo triangular faces of the clusters. Moreover, atoms Se1, Se2, Se3, Se5, Se6 and Se7 are linked to an Mo atom of a neighbouring cluster. The Mo–Se bond distances range from 2.5436 (9) to 2.6610 (6) Å within the Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> unit and from 2.5421 (7) to 2.7001 (8) Å within the Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> unit. Each Mo<sub>9</sub>Se<sub>11</sub>Se<sub>6</sub> unit is connected to six Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> units (and *vice versa*) via Mo–Se<sup>a-i</sup> bonds to form the three-dimensional Mo–Se framework, the connective formula of which is Mo<sub>9</sub>Se<sub>5</sub>Se<sub>6/2</sub><sup>i-a</sup>Se<sub>6/2</sub><sup>a-i</sup>, Mo<sub>6</sub>Se<sub>2</sub>Se<sub>6/2</sub><sup>i-a</sup>Se<sub>6/2</sub><sup>a-i</sup>. As a result of this arrangement, the shortest intercluster Mo···Mo distance between the Mo<sub>6</sub> and Mo<sub>9</sub> clusters is 3.4409 (8) Å, indicating only a weak metal–metal interaction. This distance is a little shorter than the value of 3.512 (8) Å observed for In<sub>3,3</sub>Mo<sub>15</sub>Se<sub>19</sub>.

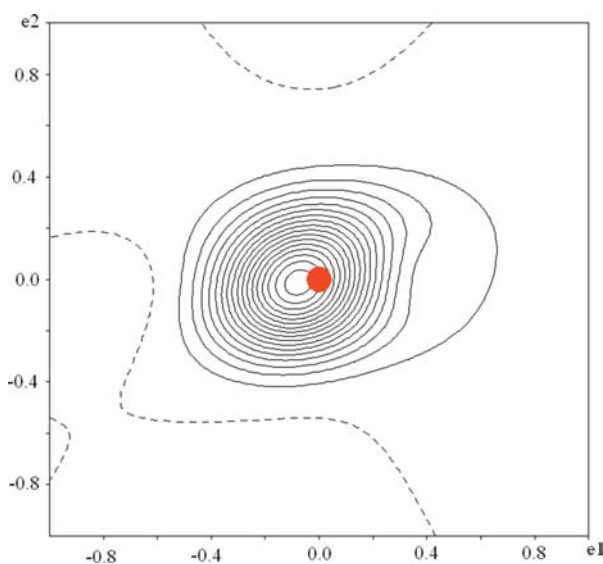
The main difference between Cr<sub>1.45</sub>Tl<sub>2</sub>Mo<sub>15</sub>Se<sub>19</sub> and the indium compounds lies in the distribution of the trivalent cations. Indeed, in addition to the triangular group of distorted octahedral sites located between two consecutive Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> units, the Cr atoms partially occupy a supplementary site located at the mid-point of the *a* axis (*2b* position). In the monoclinic compound Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub>, atoms Cr1, Cr2 and Cr3 occupy the distorted octahedral sites located between two consecutive Mo<sub>6</sub>Se<sub>8</sub>Se<sub>6</sub> units that are occupied by trivalent indium in the In<sub>3</sub>Mo<sub>15</sub>Se<sub>19</sub> compounds (Fig. 3*a*). The loss of the threefold axis leads to three crystallographically independent sites with site-occupancy factors of 0.619 (6),



**Figure 4**  
 Difference Fourier maps around the TII mean position (large central circles), showing (a) harmonic refinement (minimum and maximum densities:  $-9.84$  and  $11.02 \text{ e \AA}^{-3}$ , respectively; step:  $0.5 \text{ e \AA}^{-3}$ ), and (b) anharmonic refinement (minimum and maximum densities:  $-2.08$  and  $2.92 \text{ e \AA}^{-3}$ , respectively; step:  $0.5 \text{ e \AA}^{-3}$ ).

0.313 (6) and 0.125 (6), compared with the trivalent indium with site occupancies of 0.29 (5) for In<sub>2,9</sub>Mo<sub>15</sub>Se<sub>19</sub> and 0.44 (2) for In<sub>3,3</sub>Mo<sub>15</sub>Se<sub>19</sub>. The Cr–Se distances are in the ranges 2.515 (2)–2.850 (3), 2.524 (3)–2.798 (2) and 2.501 (8)–2.9635 (6) Å for the Cr1, Cr2 and Cr3 sites, respectively. Atoms Cr4 in the *2b* Wyckoff position also occupy a distorted octahedral site of Se atoms with Cr–Se distances ranging from 2.3096 (6) to 2.9635 (6) Å.

The Tl<sup>+</sup> cation of Cr<sub>1.45</sub>Tl<sub>1.87</sub>Mo<sub>15</sub>Se<sub>19</sub> replaces the monovalent In<sup>+</sup> in the large ten-coordinated sites of the Se atoms (Fig. 3*b*). While the In<sup>+</sup> site centred on the threefold axis has 3<sub>.</sub> symmetry, the Tl<sup>+</sup> site has no imposed symmetry. The Tl–



**Figure 5**

Anharmonic probability density function map of Tl1 in the *bc* plane (minimum and maximum densities:  $-0.086$  and  $8.7905 \text{ \AA}^{-3}$ , respectively; step:  $0.5 \text{ \AA}^{-3}$ ).

Se distances range from 3.1152 (11) to 4.2214 (9) Å. The large  $U_{\text{iso}}$  value for Tl, which is about five times higher than those for the Mo atoms and three to four times higher than those for the Se and Cr atoms, is due to the large voids that it occupies and does not result from an artefact due to the correlations (0.506 in our case) between the atomic displacement parameters (ADPs) and the site-occupation factor of the Tl atom. This is corroborated by the fact that similar ratios are observed in other reduced molybdenum chalcogenides such as  $\text{Tl}_2\text{Mo}_6\text{Se}_6$  (Potel *et al.*, 1980a) or  $\text{Tl}_2\text{Mo}_9\text{S}_{11}$  (Potel *et al.*, 1980b), in which the Tl sites are fully occupied. While in the  $\text{In}_3\text{Mo}_{15}\text{Se}_{19}$  structure type, the monovalent  $\text{In}^+$  site is fully occupied, in the title compound the  $\text{Tl}^+$  site is only 0.935 (2) occupied. This probably results from the higher temperature used during the crystal growth process, which leads to a loss of TlSe because of its high volatility at 1773 K. It is interesting to note that a similar site-occupation factor of 0.94 (1) is found when refining using *SHELXL* (Sheldrick, 2008) a model with two Tl positions and harmonic ADPs and with only one twinning matrix.

## Experimental

Single crystals of  $\text{Cr}_{1.453}\text{Tl}_{1.87}\text{Mo}_{15}\text{Se}_{19}$  were prepared from a mixture of  $\text{Cr}_2\text{Se}_3$ ,  $\text{MoSe}_2$ , TlSe and Mo with the nominal composition  $\text{Cr}_{1.5}\text{Tl}_2\text{Mo}_{15}\text{Se}_{19}$ . Before use, Mo powder was reduced under flowing  $\text{H}_2$  gas at 1273 K for 10 h in order to eliminate any trace of oxygen. The binaries  $\text{Cr}_2\text{Se}_3$ ,  $\text{MoSe}_2$  and TlSe were obtained by heating stoichiometric mixtures of the elements in sealed evacuated silica tubes for about 2 d. All handling of materials was carried out in an argon-filled glove-box. The initial mixture (*ca* 5 g) was cold pressed and loaded into a molybdenum crucible, which was then sealed under a low argon pressure using an arc-welding system. The charge was heated at a rate of  $300 \text{ K h}^{-1}$  to 1773 K, held at that temperature for 48 h, cooled at  $100 \text{ K h}^{-1}$  to 1373 K and finally furnace cooled.

## Crystal data

$\text{Cr}_{1.453}\text{Tl}_{1.87}\text{Mo}_{15}\text{Se}_{19}$   
 $M_r = 3396.7$   
 Monoclinic,  $P2_1/m$   
 $a = 9.7347 (1) \text{ \AA}$   
 $b = 19.2639 (3) \text{ \AA}$   
 $c = 9.7987 (1) \text{ \AA}$   
 $\beta = 118.9305 (8)^\circ$

$V = 1608.22 (4) \text{ \AA}^3$   
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 36.91 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 $0.10 \times 0.05 \times 0.04 \text{ mm}$

## Data collection

Nonius KappaCCD area-detector diffractometer  
 Absorption correction: analytical (de Meulenaer & Tompa, 1965)  
 $T_{\text{min}} = 0.097$ ,  $T_{\text{max}} = 0.331$

36020 measured reflections  
 10161 independent reflections  
 6942 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.087$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.087$   
 $S = 1.49$   
 10161 reflections

231 parameters  
 $\Delta\rho_{\text{max}} = 4.01 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -3.97 \text{ e \AA}^{-3}$

**Table 1**

Selected bond lengths (Å).

Tl1—Se1 <sup>i</sup>	3.6290 (11)	Mo5—Mo9	2.7300 (7)
Tl1—Se2 <sup>ii</sup>	3.4600 (11)	Mo5—Se2	2.6890 (6)
Tl1—Se3	3.7414 (8)	Mo5—Se5	2.6337 (10)
Tl1—Se5	3.2114 (11)	Mo5—Se6	2.6285 (11)
Tl1—Se6 <sup>iii</sup>	3.1803 (7)	Mo5—Se10	2.6808 (5)
Tl1—Se7 <sup>iiii</sup>	3.1152 (11)	Mo5—Se11	2.5421 (7)
Tl1—Se8	4.1426 (13)	Mo6—Mo7	3.8222 (9)
Tl1—Se10 <sup>ii</sup>	4.1250 (12)	Mo6—Mo8	2.7491 (7)
Tl1—Se11 <sup>iv</sup>	3.2515 (11)	Mo6—Mo9	2.7032 (5)
Mo1—Mo1 <sup>v</sup>	3.8353 (6)	Mo6—Se3 <sup>vii</sup>	2.6742 (9)
Mo1—Mo2	2.6769 (8)	Mo6—Se6	2.6536 (7)
Mo1—Mo2 <sup>v</sup>	2.7252 (7)	Mo6—Se7	2.5952 (6)
Mo1—Mo3	2.6992 (6)	Mo6—Se9	2.7001 (8)
Mo1—Mo3 <sup>v</sup>	2.7098 (8)	Mo6—Se11	2.5511 (8)
Mo1—Mo5	3.4409 (8)	Mo7—Mo8	2.7127 (10)
Mo1—Se1 <sup>v</sup>	2.5771 (8)	Mo7—Mo9	2.6851 (13)
Mo1—Se2	2.6214 (10)	Mo7—Se5	2.5885 (8)
Mo1—Se3	2.5704 (10)	Mo7—Se5 <sup>viii</sup>	2.5885 (8)
Mo1—Se4	2.5575 (7)	Mo7—Se8	2.6093 (9)
Mo1—Se5	2.6610 (6)	Mo7—Se10	2.5939 (10)
Mo2—Mo2 <sup>v</sup>	3.8047 (9)	Mo8—Mo9	2.6863 (9)
Mo2—Mo3	2.6967 (9)	Mo8—Se7	2.5594 (6)
Mo2—Mo3 <sup>v</sup>	2.6906 (8)	Mo8—Se7 <sup>viii</sup>	2.5594 (6)
Mo2—Mo6 <sup>iii</sup>	3.4797 (8)	Mo8—Se8	2.5981 (15)
Mo2—Se1	2.5574 (7)	Mo8—Se9	2.6152 (15)
Mo2—Se2 <sup>v</sup>	2.5654 (9)	Mo9—Se6	2.5775 (8)
Mo2—Se3	2.6055 (6)	Mo9—Se6 <sup>viii</sup>	2.5775 (8)
Mo2—Se4	2.5760 (9)	Mo9—Se9	2.6073 (11)
Mo2—Se6 <sup>iii</sup>	2.6456 (9)	Mo9—Se10	2.6096 (11)
Mo3—Mo3 <sup>v</sup>	3.8142 (8)	Cr1—Se4 <sup>i</sup>	2.515 (2)
Mo3—Mo4 <sup>vi</sup>	3.5231 (6)	Cr1—Se6 <sup>ii</sup>	2.750 (2)
Mo3—Se1	2.6149 (8)	Cr1—Se8	2.850 (3)
Mo3—Se2	2.5821 (8)	Cr1—Se9 <sup>ii</sup>	2.821 (3)
Mo3—Se3 <sup>v</sup>	2.5555 (8)	Cr2—Se4	2.524 (3)
Mo3—Se4	2.5436 (9)	Cr2—Se5	2.798 (2)
Mo3—Se7 <sup>vi</sup>	2.6346 (9)	Cr2—Se9 <sup>iii</sup>	2.788 (6)
Mo4—Mo5	2.6441 (6)	Cr2—Se10	2.738 (6)
Mo4—Mo6	2.6278 (9)	Cr3—Se4	2.501 (8)
Mo4—Mo7	2.7347 (5)	Cr3—Se7 <sup>vi</sup>	2.782 (7)
Mo4—Mo8	2.6931 (7)	Cr3—Se8 <sup>vi</sup>	2.859 (8)
Mo4—Se1 <sup>i</sup>	2.6523 (9)	Cr3—Se10	2.886 (8)
Mo4—Se5	2.6306 (8)	Cr4—Se1	2.3096 (6)
Mo4—Se7	2.6382 (8)	Cr4—Se5 <sup>vi</sup>	2.9635 (6)
Mo4—Se8	2.6873 (7)	Cr4—Se11 <sup>vi</sup>	2.7815 (8)
Mo4—Se11	2.5507 (8)	Cr1—Cr2 <sup>i</sup>	3.231 (5)
Mo5—Mo6	2.6431 (8)	Cr1—Cr3 <sup>i</sup>	3.216 (12)
Mo5—Mo7	2.7095 (6)	Cr2—Cr3	3.295 (10)
Mo5—Mo8	3.8244 (6)		

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

Numerous crystals were tested on a Nonius KappaCCD diffractometer. Most of them presented a substantial mosaic spread, leading to unreliable lattice parameters having values close to those of a hexagonal system. For these crystals, the mosaicity as calculated by *DENZO* (Otwinowski & Minor, 1997) was of about  $2^\circ$ , indicating that they are strongly twinned. For the studied crystal, the mosaicity was only  $0.496(1)^\circ$ , as usually observed for good single crystals. On the other hand, all the Bragg spots found using the  $\varphi/\chi$  procedure (Duisenberg *et al.*, 2000) could be indexed, indicating that if the crystal were twinned the twinning was very minor. Consequently, the integration of the Bragg spots from the collected frames was made with the orientation matrix deduced from the  $\varphi/\chi$  procedure and, in order to take into account the possible existence of non-overlapping spots at high diffraction angles, large integration boxes were used. The structure was solved using *SIR97* (Altomare *et al.*, 1999) and subsequent difference Fourier syntheses, and was refined in an anisotropic approximation down to  $R = 0.077$ . Because the lattice parameters are close to those of a hexagonal system, the existence of twin domains was envisaged in the studied crystal. As the space group  $P2_1/m$  is a subgroup of index 3 of the space group  $P6_3/m$ , six twin domains can exist, according to the procedure implemented in *JANA2006* (Petříček *et al.*, 2006), which decomposes a point group into co-sets with respect to a subgroup. Consequently, six twin matrices corresponding to the identity (**T1**) and the clockwise (**T2**) and anticlockwise (**T3**) threefold rotations about  $b$ , and the three inverse matrices of these (**T4**, **T5**, and **T6**), were introduced into the refinement. This lowered the  $R$  factor to 0.055. At this stage, analysis of the difference Fourier map revealed positive and negative residual peaks of  $11.02$  and  $-9.84 \text{ e } \text{Å}^{-3}$ , respectively, around the Tl1 atom (Fig. 4a). Fourth-order tensors in the Gram–Charlier expansion (Johnson & Levy, 1974) of the thallium displacement factor were used to better describe the electronic density around the cationic site. The residual  $R$  value dropped to 0.0489 and the residual difference electron-density extrema in the vicinity of Tl1 dropped to 2.92 and  $-2.08 \text{ e } \text{Å}^{-3}$  (Fig. 4b). Fig. 5 shows the anharmonic probability density function map for Tl1 in the  $bc$  plane. The absence of a significant negative region indicates that the refined model can be considered valid (Bachmann & Schulz, 1984). At the end of the refinement, the fractional volumes of the twin domains were

0.947 (2), 0.008 (1), 0.011 (1), 0.0262 (4), 0 and 0.0079 (4) for matrices **T1–T6**, respectively. Refinement of the occupancy factors of Tl and Cr led to the final stoichiometry  $\text{Cr}_{1.45(2)}\text{Tl}_{1.870(4)}\text{Mo}_{15}\text{Se}_{19}$ .

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *EVALCCD* (Duisenberg, 1998); 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*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LG3024). Services for accessing these data are described at the back of the journal.

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