

Rb₃[PMo₁₂O₄₀], a compound containing Keggin anions

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{P}-\text{O}) = 0.005$ Å; R factor = 0.032; wR factor = 0.086; data-to-parameter ratio = 18.3.

The cubic structure of trirubidium molybdophosphate, Rb₃[PMo₁₂O₄₀], contains α -type Keggin [PMo₁₂O₄₀]³⁻ anions and Rb⁺ counter-ions located in orthogonally intersecting channels. The P and Rb atoms are located on special positions with site symmetries $\bar{4}3m$ and $\bar{4}2.m$, respectively. The three-dimensional arrangement is isostructural with that found in, for example, X₃[PMo₁₂O₄₀], with $X = \text{K}, \text{NH}_4$ and H_3O , or X₃[PW₁₂O₄₀] and X₄[SiW₁₂O₄₀], with $X = \text{Ag}$ and Tl . The compound was crystallized using a typical high-temperature solid-state reaction with GaPO₄ and X₃Mo₃O₁₀ ($X = \text{Rb}$ or Li) as starting materials.

Related literature

The synthesis of the GaPO₄ precursor is described by Beaurain *et al.* (2006). For isostructural compounds, see: Boeyens *et al.* (1976); Goubin *et al.* (2004); Kang *et al.* (2004); Parent & Moffat (1996). For properties and applications of compounds with Keggin anions, see: Bonardet *et al.* (1995); Clemente-Léon *et al.* (1997); Katsoulis (1998); López *et al.* (2001); Maestre *et al.* (2001). Keggin anions were described for the first time by Keggin (1933).

Experimental

Crystal data

Rb ₃ [PMo ₁₂ O ₄₀]	$Z = 2$
$M_r = 2078.66$	Mo $K\alpha$ radiation
Cubic, $Pn\bar{3}m$	$\mu = 9.30 \text{ mm}^{-1}$
$a = 11.67521(8) \text{ \AA}$	$T = 293 \text{ K}$
$V = 1591.45(2) \text{ \AA}^3$	$0.20 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Oxford Diffraction XCALIBUR diffractometer	$T_{\min} = 0.525, T_{\max} = 1.000$ (expected range = 0.130–0.248)
Absorption correction: multi-scan <i>CrysAlis RED</i> (Oxford Diffraction, 2007);	27390 measured reflections
	567 independent reflections
	375 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	31 parameters
$wR(F^2) = 0.086$	$\Delta\rho_{\text{max}} = 1.42 \text{ e \AA}^{-3}$
$S = 0.91$	$\Delta\rho_{\text{min}} = -3.91 \text{ e \AA}^{-3}$
567 reflections	

Table 1

Selected bond lengths (Å).

Rb1—O4 ⁱ	3.281 (5)	Mo2—O5	1.687 (5)
Rb1—O5 ⁱⁱ	3.044 (5)	Mo2—O6	2.438 (5)
Rb1—O7 ⁱ	3.244 (5)	Mo2—O7	1.920 (2)
Mo2—O7 ⁱⁱⁱ	1.920 (2)	P3—O6	1.534 (8)
Mo2—O4	1.9115 (13)		

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *Superflip* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996) and *DrawXtl* (Finger *et al.*, 2007); software used to prepare material for publication: *CRYSTALS*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2153).

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Rb₃[PMo₁₂O₄₀], a compound containing Keggin anions

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Comment

Structures containing Keggin anions (Keggin, 1933), $[XM_{12}O_{40}]^{n-}$ with X a main group or a transition metal ion and M a transition metal ion, have been largely studied from a fundamental point of view (López *et al.*, 2001; Maestre *et al.*, 2001) and also because of their applications in view of their interesting redox properties in relation to their micro-porosity in the salts of Keggin anions with various cations (Bonardet *et al.*, 1995; Clemente-Léon *et al.*, 1997; Katsoulis *et al.*, 1998).

The structure of Rb₃[PMo₁₂O₄₀] is comprised of $[PMo_{12}O_{40}]^{3-}$ anions and Rb⁺ counter-cations located in one-dimensional square channels. The anions have the α -Keggin structure type, *i.e.* they are composed of a central tetrahedrally coordinated hetero-atom, in the present case a phosphorus atom. The PO₄ tetrahedron is caged by 12 octahedral MoO₆-units linked to one another by neighboring oxygen atoms. It is noted that the MoO₆ octahedra are rather distorted, having one short distance Mo2—O5 of 1.687 (5) Å, one long distance Mo2—O6 of 2.438 (5) Å, and four intermediate distances of 1.9115 (13) Å and 1.920 (2) Å, each appearing twice. The PO₄ tetrahedron is by symmetry regular (P on a $\bar{4}3m$ site; d(P—O) = 1.534 (8) Å). The Rb atom is in 12-fold coordination by oxygen, with three different Rb—O distances of 3.044 (5) Å, 3.244 (5) Å, and 3.281 (5) Å, respectively, each distance appearing 4 times. Fig. 1 shows the coordination environment of the three cations.

Fig. 2 shows a polyhedral representation of the α -Keggin anion $[PMo_{12}O_{40}]^{3-}$. A view of the three-dimensional structure is shown in Fig. 3; there are two square channels that are occupied by Rb cations. Due to the cubic symmetry there are in fact three orthogonally intersecting Rb-containing channels.

It is noted that the title compound is isostructural with a number of other structures of rather different composition such as (K, NH₄, H₃O)₃[PMo₁₂O₄₀] (Boeyens *et al.*, 1976), (Ag, Tl)₃[PW₁₂O₄₀] and (Ag, Tl)₄[SiW₁₂O₄₀] (Parent & Moffat, 1996), K₃[PMo₁₂O₄₀] (Goubin *et al.*, 2004) and (K_{2.4}(H₃O)_{0.6})[PW₁₂O₄₀] (Kang *et al.*, 2004).

Experimental

The aim of this synthesis was to grow piezoelectric α -GaPO₄ single crystals at temperatures below their transition point (1233 K). This was achieved using the high temperature solution growth technique also known as the fluxed-melt technique. In this context, X₂Mo₃O₁₀ compounds with X = Li, K, Rb, which have relatively low melting points, were used as fluxes. The α -GaPO₄ powder was obtained by dissolving 4 N (99.99% purity) Ga metal in nitric acid followed by precipitation with phosphoric acid as described by Beaurain *et al.* (2006). Rb₃Mo₃O₁₀ and Li₂Mo₃O₁₀ used as starting materials were synthesized following the solid-state reaction: X₂CO₃ + 3MoO₃ = X₂Mo₃O₁₀ + CO₂ with X = Li, Rb. 51 wt% of Rb₃Mo₃O₁₀ and 34 wt% of Li₂Mo₃O₁₀ were mixed with 15 wt% of α -GaPO₄ and homogenized in an agate mortar. The mixture was put in a Pt crucible covered with a lid, heated from room temperature to 1223 K at a ramp rate of 150 K·h⁻¹ in a single temperature zone with a SiC resistance heater furnace, and held at this temperature during 5 h for homogenization. The

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melted charge was then slowly cooled down at a rate of 2 K h^{-1} to 873 K. After 5 h at 873 K, the charge was cooled to room temperature at 200 K h^{-1} . Yellow transparent crystals of prismatic habit were found, typically of sizes ranging from 0.1 to 1.0 mm, besides α -GaPO₄ single crystals.

Refinement

All atoms were located *ab-initio* with default values of the charge flipping parameters and an automatic determination of the static charge flip threshold δ (5.246); convergence was reached after 57 iteration cycles. The largest negative residual electron density was found 0.459 Å from P3 and the largest positive residual electron density was found 2.081 Å from O7.

Figures

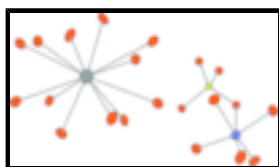


Fig. 1. The coordination environment of the three different cations in the structure of Rb₃[PMo₁₂O₄₀]. The Mo atom is in purple, the P atom in light-green, the Rb atom in grey, and the O atoms in red. Displacement ellipsoids are drawn at the 75% level.

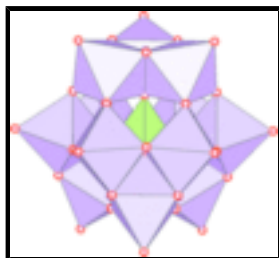


Fig. 2. Polyhedral representation of the α -Keggin [PMo₁₂O₄₀]³⁻ anion. PO₄ tetrahedra are in lime-green, whereas MoO₆ octahedra are in violet.

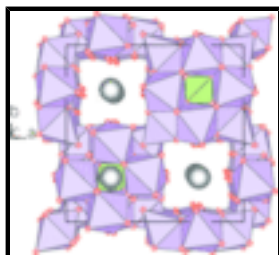


Fig. 3. Polyhedral representation of the packing of the [PMo₁₂O₄₀]³⁻ anions, which creates three orthogonally intersecting channels filled with Rb⁺ counter-cations (in grey). The colours of the polyhedra are as in Fig. 2.

trirubidium molybdophosphate

Crystal data

Rb₃[PMo₁₂O₄₀]

$M_r = 2078.66$

Cubic, $Pn\bar{3}m$

Hall symbol: -P 4bc 2bc

$a = 11.67521(8) \text{ \AA}$

$b = 11.67521(8) \text{ \AA}$

$c = 11.67521(8) \text{ \AA}$

$\alpha = 90^\circ$

$\beta = 90^\circ$

$Z = 2$

$F_{000} = 1900$

$D_x = 4.337 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 12049 reflections

$\theta = 3.0\text{--}32.5^\circ$

$\mu = 9.30 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, yellow transparent

$\gamma = 90^\circ$ $0.20 \times 0.20 \times 0.15$ mm
 $V = 1591.454 (19) \text{ \AA}^3$

Data collection

Oxford Diffraction XCALIBUR diffractometer	$R_{\text{int}} = 0.029$
Radiation source: Enhance (Mo) X-ray Source	$\theta_{\text{max}} = 32.5^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 3.0^\circ$
$T = 293$ K	$h = -17 \rightarrow 16$
ω scans	$k = -17 \rightarrow 15$
Absorption correction: multi-scan CrysAlis RED (Oxford Diffraction, 2007)	$l = -17 \rightarrow 16$
$T_{\text{min}} = 0.525$, $T_{\text{max}} = 1.000$	Standard reflections: 2 frames;
27390 measured reflections	every 50 frames reflections
567 independent reflections	intensity decay: none
375 reflections with $I > 2.0\sigma(I)$	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2]$, where $P = (\max(F_o^2, 0) + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$(\Delta/\sigma)_{\text{max}} = 0.0001$
$wR(F^2) = 0.086$	$\Delta\rho_{\text{max}} = 1.42 \text{ e \AA}^{-3}$
$S = 0.91$	$\Delta\rho_{\text{min}} = -3.91 \text{ e \AA}^{-3}$
567 reflections	Extinction correction: None
31 parameters	

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Rb1	0.7500	0.2500	0.2500	0.0233
Mo2	0.24122 (5)	0.03433 (3)	0.03433 (3)	0.0103
P3	0.2500	0.2500	0.2500	0.0059
O4	0.3472 (3)	0.1528 (3)	-0.0049 (4)	0.0136
O5	0.2680 (4)	-0.0661 (3)	-0.0661 (3)	0.0167
O6	0.1741 (4)	0.1741 (4)	0.1741 (4)	0.0078
O7	0.1232 (3)	0.1232 (3)	-0.0378 (4)	0.0134

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb1	0.0218 (8)	0.0240 (5)	0.0240 (5)	0.0000	0.0000	0.0000
Mo2	0.0106 (3)	0.01018 (19)	0.01018 (19)	0.00047 (14)	0.00047 (14)	-0.0036 (2)
P3	0.0059 (10)	0.0059 (10)	0.0059 (10)	0.0000	0.0000	0.0000

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O4	0.0143 (15)	0.0143 (15)	0.012 (2)	-0.0023 (18)	0.0014 (14)	-0.0014 (14)
O5	0.014 (3)	0.0181 (16)	0.0181 (16)	0.0010 (13)	0.0010 (13)	-0.012 (2)
O6	0.0078 (16)	0.0078 (16)	0.0078 (16)	-0.0004 (18)	-0.0004 (18)	-0.0004 (18)
O7	0.0147 (14)	0.0147 (14)	0.011 (2)	0.0022 (19)	-0.0034 (14)	-0.0034 (14)

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

Rb1—O4 ⁱ	3.281 (5)	Mo2—Mo2 ^x	3.4161 (9)
Rb1—O4 ⁱⁱ	3.281 (5)	Mo2—O7 ^{xi}	1.920 (2)
Rb1—O4 ⁱⁱⁱ	3.281 (5)	Mo2—O4 ^{xi}	1.9115 (13)
Rb1—O4 ^{iv}	3.281 (5)	Mo2—Mo2 ^{xii}	3.7060 (9)
Rb1—O5 ^v	3.044 (5)	Mo2—Mo2 ^{xiii}	3.7060 (9)
Rb1—O5 ^{vi}	3.044 (5)	Mo2—O4	1.9115 (13)
Rb1—O5 ^{vii}	3.044 (5)	Mo2—O5	1.687 (5)
Rb1—O5 ^{viii}	3.044 (5)	Mo2—O6	2.438 (5)
Rb1—O7 ⁱ	3.244 (5)	Mo2—O7	1.920 (2)
Rb1—O7 ⁱⁱ	3.244 (5)	P3—O6 ^{xiv}	1.534 (8)
Rb1—O7 ⁱⁱⁱ	3.244 (5)	P3—O6 ^{xv}	1.534 (8)
Rb1—O7 ^{iv}	3.244 (5)	P3—O6 ^{iv}	1.534 (8)
Mo2—Mo2 ^{ix}	3.4161 (9)	P3—O6	1.534 (8)
O4 ⁱ —Rb1—O4 ⁱⁱ	139.52 (11)	O7 ⁱ —Rb1—O7 ^{iv}	125.68 (10)
O4 ⁱ —Rb1—O4 ⁱⁱⁱ	58.58 (17)	O7 ⁱⁱ —Rb1—O7 ^{iv}	80.42 (16)
O4 ⁱⁱ —Rb1—O4 ⁱⁱⁱ	139.52 (11)	O7 ⁱⁱⁱ —Rb1—O7 ^{iv}	125.68 (10)
O4 ⁱ —Rb1—O4 ^{iv}	139.52 (11)	Mo2 ^{ix} —Mo2—Mo2 ^x	60.000
O4 ⁱⁱ —Rb1—O4 ^{iv}	58.58 (17)	Mo2 ^{ix} —Mo2—O7 ^{xi}	27.16 (12)
O4 ⁱⁱⁱ —Rb1—O4 ^{iv}	139.52 (11)	Mo2 ^x —Mo2—O7 ^{xi}	78.62 (14)
O4 ⁱ —Rb1—O5 ^v	93.45 (8)	Mo2 ^{ix} —Mo2—O4 ^{xi}	86.92 (14)
O4 ⁱⁱ —Rb1—O5 ^v	56.75 (12)	Mo2 ^x —Mo2—O4 ^{xi}	128.85 (15)
O4 ⁱⁱⁱ —Rb1—O5 ^v	93.45 (8)	O7 ^{xi} —Mo2—O4 ^{xi}	88.2 (2)
O4 ^{iv} —Rb1—O5 ^v	115.33 (12)	Mo2 ^{ix} —Mo2—Mo2 ^{xii}	90.000
O4 ⁱ —Rb1—O5 ^{vi}	56.75 (12)	Mo2 ^x —Mo2—Mo2 ^{xii}	120.000
O4 ⁱⁱ —Rb1—O5 ^{vi}	93.45 (8)	O7 ^{xi} —Mo2—Mo2 ^{xii}	97.22 (14)
O4 ⁱⁱⁱ —Rb1—O5 ^{vi}	115.33 (12)	O4 ^{xi} —Mo2—Mo2 ^{xii}	14.21 (15)
O4 ^{iv} —Rb1—O5 ^{vi}	93.45 (8)	Mo2 ^{ix} —Mo2—Mo2 ^{xiii}	120.000
O5 ^v —Rb1—O5 ^{vi}	90.273 (12)	Mo2 ^x —Mo2—Mo2 ^{xiii}	90.000
O4 ⁱ —Rb1—O5 ^{vii}	115.33 (12)	O7 ^{xi} —Mo2—Mo2 ^{xiii}	144.88 (12)
O4 ⁱⁱ —Rb1—O5 ^{vii}	93.45 (8)	O4 ^{xi} —Mo2—Mo2 ^{xiii}	73.23 (15)
O4 ⁱⁱⁱ —Rb1—O5 ^{vii}	56.75 (12)	Mo2 ^{xii} —Mo2—Mo2 ^{xiii}	60.000
O4 ^{iv} —Rb1—O5 ^{vii}	93.45 (8)	Mo2 ^{ix} —Mo2—O4	128.85 (15)
O5 ^v —Rb1—O5 ^{vii}	90.273 (12)	Mo2 ^x —Mo2—O4	86.92 (14)
O4 ⁱ —Rb1—O5 ^{viii}	93.45 (8)	O7 ^{xi} —Mo2—O4	155.75 (19)
O4 ⁱⁱ —Rb1—O5 ^{viii}	115.33 (12)	O4 ^{xi} —Mo2—O4	85.8 (3)

O4 ⁱⁱⁱ —Rb1—O5 ^{viii}	93.45 (8)	Mo2 ^{xii} —Mo2—O4	73.23 (15)
O4 ^{iv} —Rb1—O5 ^{viii}	56.75 (12)	Mo2 ^{ix} —Mo2—O5	128.49 (12)
O5 ^v —Rb1—O5 ^{viii}	172.08 (18)	Mo2 ^x —Mo2—O5	128.49 (12)
O4 ⁱ —Rb1—O7 ⁱ	48.24 (7)	O7 ^{xi} —Mo2—O5	101.75 (16)
O4 ⁱⁱ —Rb1—O7 ⁱ	110.50 (11)	O4 ^{xi} —Mo2—O5	102.49 (18)
O4 ⁱⁱⁱ —Rb1—O7 ⁱ	48.24 (7)	Mo2 ^{xii} —Mo2—O5	111.12 (13)
O4 ^{iv} —Rb1—O7 ⁱ	169.08 (12)	Mo2 ^{ix} —Mo2—O6	45.51 (11)
O5 ^v —Rb1—O7 ⁱ	53.75 (12)	Mo2 ^x —Mo2—O6	45.51 (11)
O4 ⁱ —Rb1—O7 ⁱⁱ	110.50 (11)	O7 ^{xi} —Mo2—O6	72.62 (16)
O4 ⁱⁱ —Rb1—O7 ⁱⁱ	48.24 (7)	O4 ^{xi} —Mo2—O6	83.34 (18)
O4 ⁱⁱⁱ —Rb1—O7 ⁱⁱ	169.08 (12)	Mo2 ^{xii} —Mo2—O6	75.75 (13)
O4 ^{iv} —Rb1—O7 ⁱⁱ	48.24 (7)	Mo2 ^{ix} —Mo2—O7	78.62 (14)
O5 ^v —Rb1—O7 ⁱⁱ	86.98 (7)	Mo2 ^x —Mo2—O7	27.16 (12)
O4 ⁱ —Rb1—O7 ⁱⁱⁱ	48.24 (7)	O7 ^{xi} —Mo2—O7	87.6 (3)
O4 ⁱⁱ —Rb1—O7 ⁱⁱⁱ	169.08 (12)	O4 ^{xi} —Mo2—O7	155.75 (19)
O4 ⁱⁱⁱ —Rb1—O7 ⁱⁱⁱ	48.24 (7)	Mo2 ^{xii} —Mo2—O7	144.88 (12)
O4 ^{iv} —Rb1—O7 ⁱⁱⁱ	110.50 (11)	Mo2 ^{xiii} —Mo2—O4	14.21 (15)
O5 ^v —Rb1—O7 ⁱⁱⁱ	134.17 (12)	Mo2 ^{xiii} —Mo2—O5	111.12 (13)
O4 ⁱ —Rb1—O7 ^{iv}	169.08 (12)	O4—Mo2—O5	102.49 (18)
O4 ⁱⁱ —Rb1—O7 ^{iv}	48.24 (7)	Mo2 ^{xiii} —Mo2—O6	75.75 (13)
O4 ⁱⁱⁱ —Rb1—O7 ^{iv}	110.50 (11)	O4—Mo2—O6	83.34 (18)
O4 ^{iv} —Rb1—O7 ^{iv}	48.24 (7)	O5—Mo2—O6	171.9 (2)
O5 ^v —Rb1—O7 ^{iv}	86.98 (7)	Mo2 ^{xiii} —Mo2—O7	97.22 (14)
O5 ^{vi} —Rb1—O5 ^{vii}	172.08 (18)	O4—Mo2—O7	88.2 (2)
O5 ^{vi} —Rb1—O5 ^{viii}	90.273 (12)	O5—Mo2—O7	101.75 (16)
O5 ^{vii} —Rb1—O5 ^{viii}	90.273 (12)	O6—Mo2—O7	72.62 (16)
O5 ^{vi} —Rb1—O7 ⁱ	86.98 (7)	O6 ^{xiv} —P3—O6 ^{xv}	109.471
O5 ^{vii} —Rb1—O7 ⁱ	86.98 (7)	O6 ^{xiv} —P3—O6 ^{iv}	109.471
O5 ^{viii} —Rb1—O7 ⁱ	134.17 (12)	O6 ^{xv} —P3—O6 ^{iv}	109.471
O5 ^{vi} —Rb1—O7 ⁱⁱ	53.75 (12)	O6 ^{xiv} —P3—O6	109.471
O5 ^{vii} —Rb1—O7 ⁱⁱ	134.17 (12)	O6 ^{xv} —P3—O6	109.471
O5 ^{viii} —Rb1—O7 ⁱⁱ	86.98 (7)	O6 ^{iv} —P3—O6	109.471
O7 ⁱ —Rb1—O7 ⁱⁱ	125.68 (10)	Mo2—O4—Mo2 ⁱⁱ	151.6 (3)
O5 ^{vi} —Rb1—O7 ⁱⁱⁱ	86.98 (7)	Mo2—O6—Mo2 ^{ix}	89.0 (2)
O5 ^{vii} —Rb1—O7 ⁱⁱⁱ	86.98 (7)	Mo2—O6—Mo2 ^x	89.0 (2)
O5 ^{viii} —Rb1—O7 ⁱⁱⁱ	53.75 (12)	Mo2 ^{ix} —O6—Mo2 ^x	89.0 (2)
O7 ⁱ —Rb1—O7 ⁱⁱⁱ	80.42 (16)	Mo2—O6—P3	125.99 (15)
O7 ⁱⁱ —Rb1—O7 ⁱⁱⁱ	125.68 (10)	Mo2 ^{ix} —O6—P3	125.99 (15)
O5 ^{vi} —Rb1—O7 ^{iv}	134.17 (12)	Mo2 ^x —O6—P3	125.99 (15)
O5 ^{vii} —Rb1—O7 ^{iv}	53.75 (12)	Mo2 ^x —O7—Mo2	125.7 (2)
O5 ^{viii} —Rb1—O7 ^{iv}	86.98 (7)		

supplementary materials

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

Fig. 1

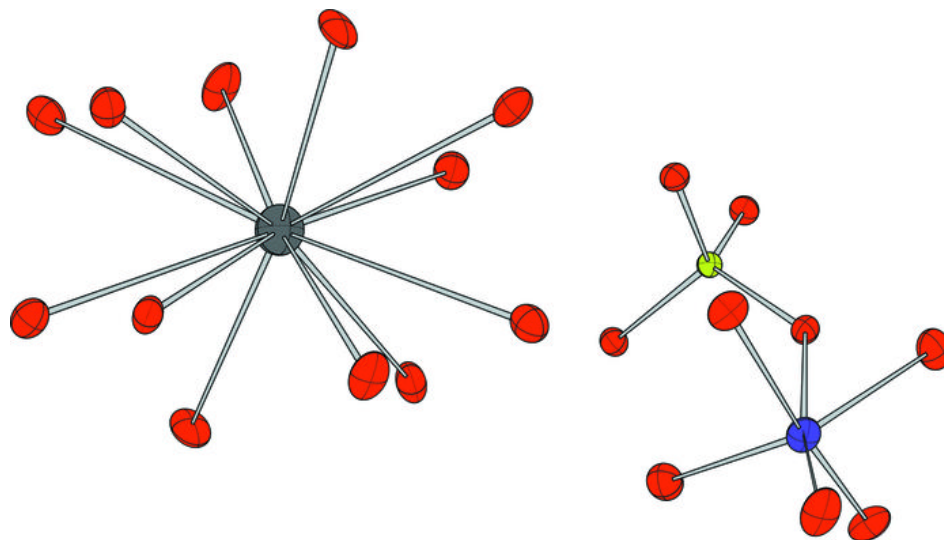


Fig. 2

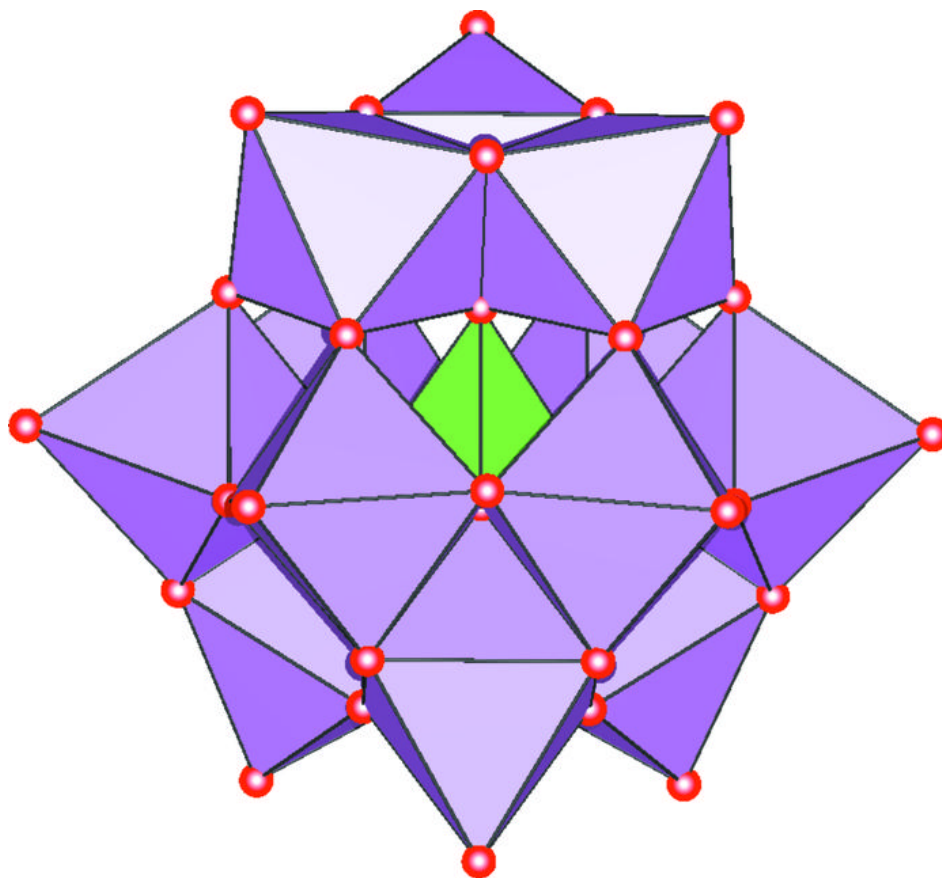


Fig. 3

