

$\text{Li}_3\text{Sc}(\text{MoO}_4)_3$: substitutional disorder on three (Li,Sc) sites

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Key indicators

Single-crystal X-ray study

$T = 293 \text{ K}$

Mean $\sigma(\text{Mo}-\text{O}) = 0.002 \text{ \AA}$

Disorder in main residue

R factor = 0.023

wR factor = 0.058

Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

$\text{Li}_3\text{Sc}(\text{MoO}_4)_3$, trilithium scandium trimolybdate(VI), is isotopic with its Fe^{III} analogue and a number of $\text{Li}_3M^{\text{III}}(\text{MoO}_4)_3$ and $\text{Li}_2M^{\text{II}}_2(\text{MoO}_4)_3$ compounds. It contains three substitutionally disordered (Li,Sc) sites; two of these show octahedral coordination and contain about 42 and 25% Sc, whereas the third site has a trigonal-prismatic environment and contains only about 8% Sc. The average (Li,Sc)–O bond lengths are 2.092, 2.129, and 2.195 Å, respectively. The (Li,Sc) O_6 polyhedra share either faces or edges with equivalent polyhedra to form columns and zigzag chains, all running parallel to [100]. These building units are connected, *via* two non-equivalent MoO_4 tetrahedra (average Mo–O bond length 1.771 Å), into a three-dimensional framework.

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Comment

During flux-growth preparation in air of compounds in the system $\text{Sc}_2\text{O}_3\text{--Al}_2\text{O}_3\text{--TiO}_2\text{--SiO}_2$, the title compound was obtained as a by-product from an Li-rich molybdate flux. $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ crystallizes in space group $Pnma$ and is isotopic with $\text{Li}_3\text{Fe}(\text{MoO}_4)_3$ (Klevtsova & Magarill, 1970). The pale green Fe^{III} member forms a solid solution series with black

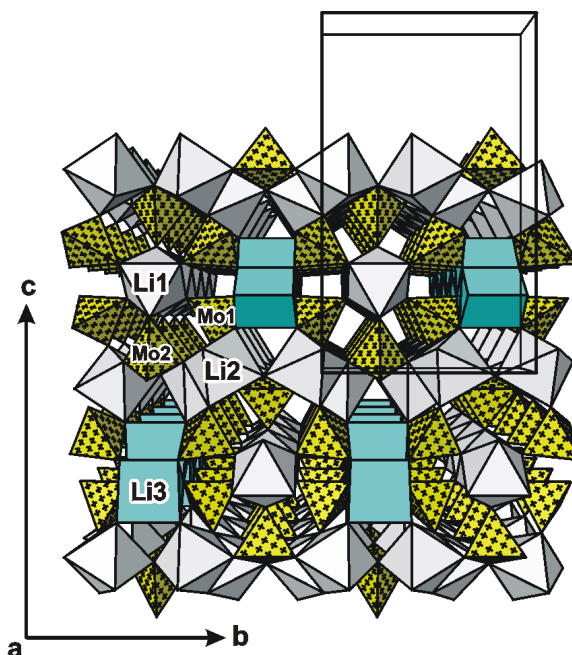


Figure 1

Perspective view of the framework structure of $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ along [100], the direction of the polyhedral columns and zigzag chains of the (Li1,Sc1) O_6 and (Li2,Sc2) O_6 octahedra (grey), and (Li3,Sc3) O_6 trigonal pyramids (blue). MoO_4 tetrahedra are yellow and marked with crosses. The unit cell is outlined.

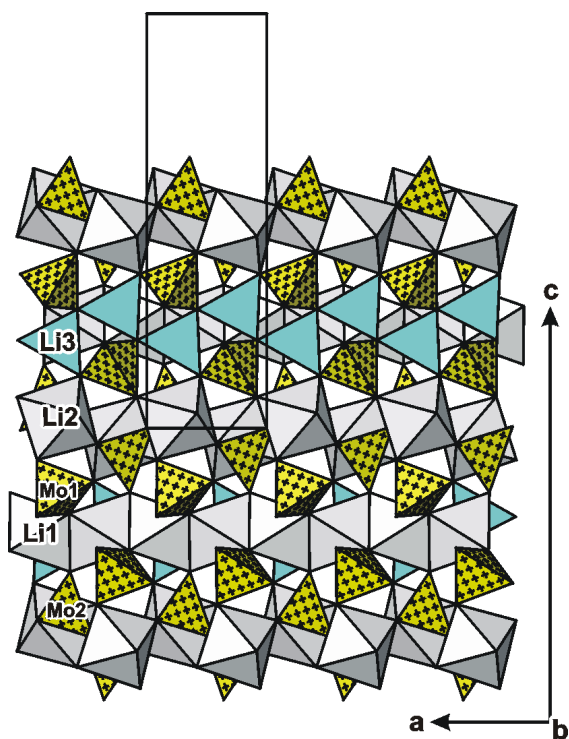


Figure 2

The structure of $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ projected along $[010]$, allowing a view perpendicular to the polyhedral columns and zigzag chains of the $(\text{Li}1, \text{Sc}1)\text{O}_6$ and $(\text{Li}2, \text{Sc}2)\text{O}_6$ octahedra (grey), and $(\text{Li}3, \text{Sc}3)\text{O}_6$ trigonal pyramids (blue).

$\text{Li}_2\text{Fe}^{\text{II}}_2(\text{MoO}_4)_3$ (Klevtsov, 1970; Klevtsova & Magarill, 1970). In fact, a number of isotypic (space group *Pnma*) $\text{Li}_3M^{\text{III}}(\text{MoO}_4)_3$ and $\text{Li}_2M^{\text{II}}_2(\text{MoO}_4)_3$ compounds are known, although the crystal structures of the M^{III} members have only been refined for the above-mentioned Fe^{III} representative.

$\text{Li}_3M^{\text{III}}(\text{MoO}_4)_3$ members with $M^{\text{III}} = \text{Fe}, \text{Al}, \text{Sc}, \text{Ga}, \text{In}$, and Cr were prepared by Klevtsov (1970) and Trunov & Efremov (1971). The In member has also been studied by Velikodnyi *et al.* (1970). $\text{Li}_3\text{Cr}(\text{MoO}_4)_3$ has been further characterized by Butukhanov *et al.* (1972), Trunov & Velikodnyi (1972), and Mokhosoev *et al.* (1973), and the members for $M^{\text{III}} = \text{Al}$ and In were described by Kozhevnikova *et al.* (1980). $\text{Li}_3\text{Ga}(\text{MoO}_4)_3$ was observed by Alekseev *et al.* (1982) as one of the products of the reaction between Li_2MoO_4 and $\text{Ga}_2(\text{SO}_4)_3$. Porotnikov *et al.* (1982) prepared $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ and stated that it melts congruently at 1109 K, and decomposes to Li_2MoO_4 and $\text{LiSc}(\text{MoO}_4)_2$ at less than 810 K. IR spectroscopic studies of $\text{Li}_3M^{\text{III}}(\text{MoO}_4)_3$ members with $M^{\text{III}} = \text{Al}, \text{In}, \text{Cr}, \text{Fe}$, and Co were reported by Juri *et al.* (1984, 1992).

$\text{Li}_2M^{\text{II}}_2(\text{MoO}_4)_3$ compounds with $M^{\text{II}} = \text{Mg}, \text{Fe}, \text{Co}, \text{Ni}, \text{Cu}$, and Zn have been prepared by Efremov & Trunov (1975) and Penkova & Klevtsov (1977). Independently of the latter authors, the structure of $\text{Li}_2\text{Ni}_2(\text{MoO}_4)_3$ was reported by Ozima *et al.* (1977), who also presented a detailed discussion of the structure type. [Note that the z coordinate for the site designated 'M1—Li' is misprinted in the original publication on $\text{Li}_2\text{Ni}_2(\text{MoO}_4)_3$ (Ozima *et al.*, 1977), and also in the ICSD entry 1082 (Belsky *et al.*, 2002) for this structure; the correct

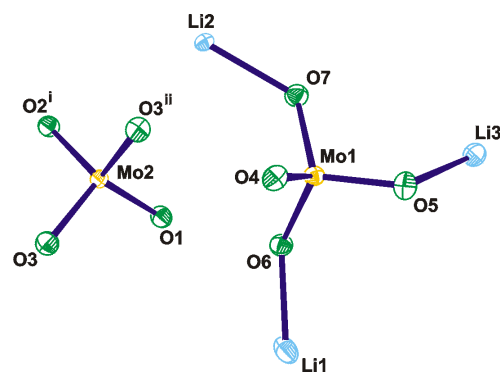


Figure 3

View of the atoms in the asymmetric unit of $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$, shown with anisotropic displacement ellipsoids at the 50% probability level. [Symmetry codes: (i) $x + 1, y, z$; (ii) $x, -y + \frac{1}{2}, z$.]

value, found by trial and error, must be 0.7568 (instead of 0.5568).] The crystal structure of $\text{Li}_{2-2x}\text{Mn}_{2+x}(\text{MoO}_4)_3$ ($x = 0.2$) was determined by Solodovnikov *et al.* (1994), and $\text{Li}_2\text{Co}_2(\text{MoO}_4)_3$ was structurally characterized by Wiesmann *et al.* (1995). Results from both studies confirm the previous results of Ozima *et al.* (1977).

$\text{Li}_2\text{Cu}_2(\text{MoO}_4)_3$ was reported to have the monoclinic space group *P2₁/c* (Wiesmann *et al.*, 1994), but a close examination showed that the topology is identical to that of the *Pnma*-type $\text{Li}_2M^{\text{II}}_2(\text{MoO}_4)_3$ compounds, and a search for higher symmetry using *PLATON* (Spek, 2000, 2003) clearly suggested that the correct space group for $\text{Li}_2\text{Cu}_2(\text{MoO}_4)_3$ is probably also *Pnma*. A dimorph of $\text{Li}_2\text{Fe}_2(\text{MoO}_4)_3$, garnet-related and crystallizing in space group *Pbcn*, was structurally characterized by Torardi & Prince (1986) and Torardi *et al.* (1988). We also point out that 'NaCo_{2.31}(MoO₄)₃' (Ibers & Smith, 1964; later shown to be $\text{Na}_2\text{Co}^{\text{II}}_2(\text{MoO}_4)_3$ by Klevtsova & Magarill, 1970) is isostructural with the *Pnma*-type $\text{Li}_3M^{\text{III}}(\text{MoO}_4)_3$ and $\text{Li}_2M^{\text{II}}_2(\text{MoO}_4)_3$ compounds listed above, and in fact represents the first solution of the common crystal structure.

The atomic arrangement in $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ is based on a three-dimensional framework of $(\text{Li}, \text{Sc})\text{O}_6$ polyhedra linked to MoO_4 tetrahedra (Figs. 1–3). There are two distinct types of $(\text{Li}, \text{Sc})\text{O}_6$ polyhedra: the first two (Li, Sc) sites, Li1 and Li2, show a nearly regular octahedral coordination (Table 1), whereas the third site, Li3, has a regular trigonal-prismatic environment (Figs. 1 and 2). The Li1 and Li2 sites contain about 42 and 25% Sc, whereas the trigonal-prismatic site (Li3) contains only about 8% Sc. The average $(\text{Li}, \text{Sc})\text{—O}$ bond lengths for the octahedrally coordinated Li1 and Li2 sites are 2.092 and 2.129 Å, respectively, whereas the corresponding value in the trigonal-prismatic $(\text{Li}3, \text{Sc})\text{O}_6$ polyhedron is 2.195 Å. Thus, an increasing Li content on a specific site causes a slight increase in average $(\text{Li}, \text{Sc})\text{—O}$ bond lengths, if the influences of the polyhedral geometry and distortion are neglected. This increase is in agreement with literature data on the size and geometry of LiO_6 and ScO_6 polyhedra; the average Li—O bond length for (more or less) octahedrally coordinated Li sites in a large number of Li oxide compounds

was calculated to be 2.15 Å (Wenger & Armbruster, 1991), whereas octahedrally coordinated Sc has an average Sc—O bond length of 2.105 Å (Baur, 1981). In $\text{Li}_3\text{Fe}(\text{MoO}_4)_3$ (Klevtsova & Magarill, 1970), the corresponding values for the (Li1,Fe), (Li2,Fe), and (Li3,Fe) sites are 2.046, 2.091, and 2.164 Å, respectively. Thus, the overall trend is identical.

A closer look at the connectivity within the framework structure of the title compound (Figs. 1 and 2) shows that it contains chains of face-sharing, fairly regular (Li1,Sc) O_6 octahedra which run parallel to [100], zigzag chains of edge-sharing, somewhat distorted, (Li2,Sc) O_6 octahedra, which also run parallel to [100], and finally (Li3,Sc) O_6 regular trigonal prisms which share prism edges to form zigzag chains, again parallel to [100]. These three (Li,Sc) O_6 polyhedra share corners with two non-equivalent MoO_4 tetrahedra. The latter show relatively small angular distortions (Table 1) and similar average Mo—O bond lengths of 1.769 (Mo1) and 1.772 Å (Mo2), respectively. In $\text{Li}_3\text{Fe}(\text{MoO}_4)_3$ (Klevtsova & Magarill, 1970), the average Mo—O bond length is only slightly larger, 1.781 Å.

The topology of the structure type can also be described as close-packed sheets of O atoms, composed of three-square-wide bands of the No. 9 regular net of Wells (1952), held together by semi-regular single chains of triangles; regular and semi-regular voids between the O atoms are filled by the Li and $M^{\text{III}}/M^{\text{II}}$ cations (see Ozima *et al.*, 1977, for further details).

The fact that the trigonal-prismatic site in $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$, Li3, contains about 8% Sc seems to distinguish it from $\text{Li}_3\text{Fe}(\text{MoO}_4)_3$, for which the equivalent site was reported to be occupied by Li only (Klevtsova & Magarill, 1970). Comparable minor discrepancies were noted for $\text{Li}_2\text{Co}_2(\text{MoO}_4)_3$ by Wiesmann *et al.* (1995) who stated that three positions for Co were determined for their compound instead of two as reported by Penkova & Klevtsov (1977); all three positions are partially occupied by Co and Li with different site occupations, with the highest Li:Co ratio being 0.79:0.21 on the trigonal-prismatic site, and the lowest Li:Co ratio being 0.34:0.66 on the edge-sharing octahedral site. Nonetheless, in $\text{Li}_2\text{Ni}_2(\text{MoO}_4)_3$, the trigonal-prismatic site was found to be occupied by Li only (Ozima *et al.*, 1977), and in 'NaCo_{2.31}(MoO₄)₃' [= Na₂Co^{II}₂(MoO₄)₃; Klevtsova & Magarill, 1970], the same site hosted Na only (Ibers & Smith, 1964). Although modern data collections and refinements might reveal small amounts of M cations on the trigonal-prismatic site in all these structures, the respective occupancies may all be dependent on temperature of formation or kinetic influences, availability of cations, and also possible oxidation of M^{II} cations. The common framework structure type is relatively flexible in adapting to different Li:M ratios on the three different (Li/M) sites (Ozima *et al.*, 1977).

By comparison with the isostructural $\text{Li}_3\text{Fe}(\text{MoO}_4)_3$ (Klevtsova & Magarill, 1970), all unit-cell parameters of the Sc member are enlarged, and the cell volume is increased by 2.56%. This is expected from the average M—O bond lengths of six-coordinated Sc and Fe^{III} (2.105 and 2.011 Å, respectively; Baur, 1981). The presently determined unit-cell parameters of the title compound are, except for the parameter *a*,

slightly smaller than those refined earlier from X-ray powder diffraction data, *a* = 5.12 (10), *b* = 10.65 (7), *c* = 17.86 (5) Å and *V* = 974 Å³ (Klevtsov, 1970; note: s.u. values estimated from error bars given in Fig. 2 of Klevtsov's paper). Unit-cell parameters given for 'LiSc(MoO₄)₂', *a* = 5.12 (1), *b* = 10.52 (2), *c* = 17.86 (2) Å and *V* = 962 Å³ (Porotnikov *et al.*, 1982; ICDD-PDF 36–207), suggest that this compound is, in fact, more likely to be $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$.

Although $\text{Li}_3\text{Al}(\text{MoO}_4)_3$ is known to exist (Klevtsov, 1970; Trunov & Efremov, 1971; Kozhevnikova *et al.*, 1980), and the flux used for crystal growth contained dissolved Al^{III} ions, the chemical analyses showed that the crystals of the title compound contain no Al. Thus, Sc strongly partitions into $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$, whereas Al was completely used up by (earlier?) crystallization of the accompanying LiAl_5O_8 octahedra (see *Experimental*).

Experimental

The title compound crystallized as a very minor component from an Li-rich flux (experimental parameters: 0.6 g LiF, 1.67 g MoO_3 , 0.1726 g Sc_2O_3 , 0.1276 g Al_2O_3 , 0.1999 g TiO_2 ; Pt crucible covered with a lid, $T_{\text{max}} = 1423$ K, holding time 6 h, cooling rate 1.5 K min⁻¹, $T_{\text{min}} = 1173$ K, slow cooling to room temperature after switching off furnace). $\text{Li}_3\text{Sc}(\text{MoO}_4)_3$ formed colourless-to-white acicular crystals (up to ca 0.5 mm in length), often in bundle-like, approximately parallel intergrowths. The crystals were accompanied by large amounts of LiAl_5O_8 (colourless sharp octahedra), and minor amounts of an Al-rich member of the solid-solution series Sc_2TiO_5 – Al_2TiO_5 (Kolitsch & Tillmanns, 2003). Chemical analyses of the title compound with standard semiquantitative SEM–EDS showed the presence of only Sc and Mo metals (Li cannot be detected under these conditions).

Crystal data

$\text{Li}_3\text{Mo}_3\text{O}_{12}\text{Sc}$	Mo <i>K</i> α radiation
$M_r = 545.60$	Cell parameters from 1670 reflections
Orthorhombic, <i>Pnma</i>	$\theta = 2.0$ – 30.0°
$a = 5.130$ (1) Å	$\mu = 4.56$ mm ⁻¹
$b = 10.560$ (2) Å	$T = 293$ (2) K
$c = 17.745$ (4) Å	Fragment, colourless
$V = 961.3$ (3) Å ³	0.10 × 0.05 × 0.05 mm
$Z = 4$	
$D_x = 3.770$ Mg m ⁻³	

Data collection

Nonius KappaCCD diffractometer	1477 independent reflections
φ and ω scans	1326 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{\text{int}} = 0.011$
(<i>HKL SCALEPACK</i> ;	$\theta_{\text{max}} = 30.1^\circ$
Otwinowski & Minor, 1997)	$h = -7 \rightarrow 7$
$T_{\text{min}} = 0.659$, $T_{\text{max}} = 0.804$	$k = -14 \rightarrow 14$
2635 measured reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.024P)^2 + 2P]$
$R[F^2 > 2\sigma(F^2)] = 0.023$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.058$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.12$	$\Delta\rho_{\text{max}} = 0.96$ e Å ⁻³
1477 reflections	$\Delta\rho_{\text{min}} = -0.84$ e Å ⁻³
99 parameters	Extinction correction: <i>SHELXL97</i>
	Extinction coefficient: 0.0018 (3)

Table 1
Selected geometric parameters (Å).

Mo1—O5	1.740 (2)	Li1—O1 ^{iv}	2.136 (4)
Mo1—O7	1.772 (2)	Li2—O7	2.085 (3)
Mo1—O4	1.781 (2)	Li2—O4 ⁱ	2.093 (3)
Mo1—O6	1.785 (2)	Li2—O4 ^v	2.122 (3)
Mo2—O3	1.762 (2)	Li2—O3 ^{vi}	2.125 (2)
Mo2—O1	1.774 (3)	Li2—O2 ^v	2.139 (2)
Mo2—O2 ⁱ	1.790 (3)	Li2—O3 ^{viii}	2.207 (3)
Li1—O6	2.067 (3)	Li3—O7 ^{viii}	2.153 (4)
Li1—O6 ⁱⁱ	2.076 (3)	Li3—O5 ^{ix}	2.193 (4)
Li1—O1 ⁱⁱⁱ	2.119 (4)	Li3—O5	2.237 (5)

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

A freely refined model gave Li:Sc occupancy ratios of 0.639 (3):0.361 (3), 0.751 (3):0.249 (3) and 0.925 (3):0.075 (3) for the Li1, Li2 and Li3 sites, respectively, which would correspond to 5.88 positive charges and 6.00 negative charges in the formula unit. However, the Li1 and, to a lesser extent, the Li3 site show somewhat anisotropic displacement parameters, an observation which indicates that the refined Li:Sc occupancy ratios are certainly influenced by the anisotropic behaviour of the atoms on these sites (neglecting all other possible influences). In the final refinement, the Li:Sc ratio on the Li1 site was slightly modified and fixed at 58:42, in order to achieve a charge-balanced formula.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS* (Shape Software, 1999) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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