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$Li_2GeMo_3O_8$: a novel reduced molybdenum oxide containing Mo_3O_{13} cluster units

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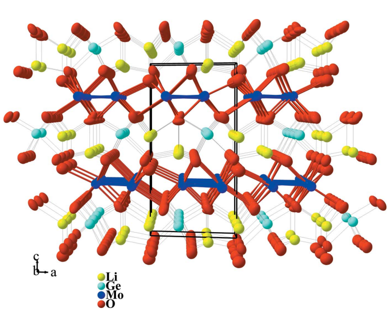
The crystal structure of the title compound, dilithium germanium trimolybdenum octaoxide, consists of distorted hexagonal-close-packed oxygen layers with stacking sequence *ABAC* along [001] that are held together by alternating lithium–germanium and molybdenum layers. The two Li^+ and Ge^{4+} ions all have site symmetry $3m$. and occupy, respectively, tetrahedral and octahedral sites in the ratio 2:1. The Mo atom has a formal oxidation state of +3.3 and occupies an octahedral site (site symmetry $.m.$) and forms strongly bonded triangular cluster units [Mo—Mo distance = 2.4728 (8) Å] involving three MoO_6 octahedra that are each shared along two edges, constituting an Mo_3O_{13} unit.



1. Chemical context

Reduced molybdenum oxides containing the Mo_3O_{13} cluster unit crystallize either in the hexagonal space group type $P6_3mc$ ($a \sim 5.7$–5.8 Å, $c \sim 10.0$–10.2 Å) or in the trigonal space group types $P3m1$ ($a \sim 5.7$–5.8 Å, $c \sim 4.9$–5.3 Å) or $R\overline{3}m$ ($a \sim 5.8$–5.9 Å, $c \sim 30.0$–30.1 Å). Representatives of the first family are the ternary compounds $M_2Mo_3O_8$ (McCarroll *et al.*, 1957) where M is a divalent metal such as Mg, Zn, Fe, Co, Ni, Zn and Cd as well as the quaternary compounds $ScZnMo_3O_8$ and $Li_2MMo_3O_8$ (M = Sn, In) (Gall *et al.*, 2013*a*,*b*). The $LiRMo_3O_8$ series (R = Sc, Y, In, Sm, Gd, Tb, Dy, Ho, Er and Yb) (DeBenedittis & Katz, 1965; McCarroll, 1977) crystallize in the $P3m1$ space group and finally, $LiZn_2Mo_3O_8$ and $Zn_3Mo_3O_8$ (Torardi & McCarley, 1985) crystallize in space group $R\overline{3}m$. The crystal structures of all these compounds consist of distorted hexagonal-close-packed oxygen layers with stacking sequences *ABAC*, *ABAB* and *ABC* for compounds crystallizing in the space groups $P6_3mc$, $P3m1$ and $R\overline{3}m$, respectively. The oxygen layers are separated by alternating mixed-metal atom (Li, M, or R) layers and molybdenum layers. The metal atoms occupy both tetrahedral and octahedral sites in a ratio of 1:1 ($M_2Mo_3O_8$ and $LiRMo_3O_8$) or 2:1 ($LiZn_2Mo_3O_8$ and $Zn_3Mo_3O_8$) between two adjacent oxygen layers. The molybdenum atoms occupy three quarters of the octahedral sites and form strongly bonded triangular cluster units involving three MoO_6 octahedra that are each shared along two edges, the whole constituting an Mo_3O_{13} unit. The Mo—Mo bonds within the trinuclear cluster units range from about 2.5 to 2.6 Å, and the number of electrons available for Mo–Mo bonding is six in $M_2Mo_3O_8$ and $LiRMo_3O_8$, seven in $LiZn_2Mo_3O_8$ and $Li_2InMo_3O_8$, and eight in $Zn_3Mo_3O_8$ and $Li_2SnMo_3O_8$. The energy-level diagram deduced from LCAO–MO calculations on the Mo_3O_{13} unit shows three bonding orbitals (*a*1 and *e*), a non-bonding level (*a*1), and five anti-

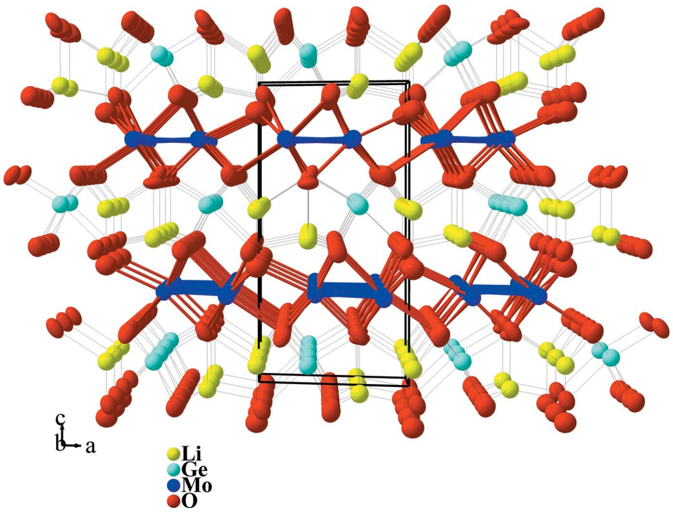


Figure 1
View of the crystal structure of $Li_2GeMo_3O_8$ in a projection approximately along [010]. Displacement ellipsoids are drawn at the 97% probability level.

Table 1
Selected bond lengths (Å).

Li1—O3	1.84 (2)	Ge1—O1^{ix}	2.016 (5)
Li1—O2^{i}	2.012 (13)	Ge1—O1^{x}	2.016 (5)
Li1—O2^{ii}	2.012 (13)	Ge1—O1^{xi}	2.016 (4)
Li1—O2^{iii}	2.012 (13)	Mo1—O4^{xii}	2.004 (6)
Li2—O4	1.78 (3)	Mo1—O1^{xii}	2.039 (4)
Li2—O1^{iv}	1.892 (6)	Mo1—O1^{v}	2.039 (4)
Li2—O1^{v}	1.892 (6)	Mo1—O3	2.076 (3)
Li2—O1^{vi}	1.892 (6)	Mo1—O2	2.146 (3)
Ge1—O2	1.883 (5)	Mo1—O2^{xiii}	2.146 (3)
Ge1—O2^{vii}	1.883 (5)	Mo1—Mo1^{xiv}	2.4728 (8)
Ge1—O2^{viii}	1.883 (5)		

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

bonding orbitals ($2e$ and $a2$) (Cotton, 1964). This explains why the compounds with seven electrons per Mo_3 cluster unit are paramagnetic with moments corresponding to one unpaired electron per Mo_3 cluster unit, and those with six and eight electrons per Mo_3 show temperature-independent paramagnetism.

We present here the crystal structure of the new quaternary compound $Li_2GeMo_3O_8$ in which the Mo_3 cluster unit has eight electrons available for bonding.

2. Structural commentary

$Li_2GeMo_3O_8$ is isotypic with the $Li_2MMo_3O_8$ (M = Sn, In) compounds (Gall *et al.*, 2013*a*,*b*). Its crystal structure consists of distorted hexagonal-close-packed oxygen layers with stacking sequence *ABAC* along [001] that are held together by alternating lithium–germanium and molybdenum layers (Fig. 1). The Li^+ and Ge^{4+} ions occupy, respectively, tetrahedral and octahedral sites in the ratio 2:1. The Mo atoms occupy octahedral sites and form strongly bonded triangular cluster units involving three MoO_6 octahedra that are each shared along two edges, constituting an Mo_3O_{13} unit (Fig. 2). The Mo—Mo distance within the Mo_3 triangle is 2.4728 (8) Å compared to 2.5036 (7) and 2.5455 (4) Å found in the tin and indium analogues, respectively. The Mo—O distances range from 2.004 (6) to 2.146 (3) Å (Table 1) while in $Li_2InMo_3O_8$ they range from 2.0212 (17) to 2.1241 (16) Å and in $Li_2SnMo_3O_8$ from 2.020 (6) to 2.122 (3) Å. The Li—O distances in the title structure range from 1.78 (2) to 2.012 (13) Å with average distances of 1.97 and 1.86 Å for the Li1 and Li2 sites, respectively. Both Li sites have site symmetry $3m$.. For the Ge site, likewise with site symmetry $3m$., the Ge—O distances are 3×1.883 (5) and 3×2.016 (5) Å. The average distance of 1.95 Å is close to the value of 1.92 Å calculated from the sum of the ionic radii of O^{2-} and Ge^{4+} in octahedral coordination according to Shannon & Prewitt (1969). The oxidation state of +4 for the Ge atoms was also confirmed from the Ge—O bond lengths by using the relationship of Brown & Wu (1976) $\{s = [d(\mathrm{Ge—O})/1.746]^{-6.05}\}$ which leads to a value of +3.5 (1). The latter relationship applied to Mo—O bonds $\{s = [d(\mathrm{Mo—O})/1.882]^{-6}\}$ yield an oxidation state of +3.38 for the Mo atom, and thus 7.86 electrons per Mo_3 cluster unit, close to the expected value of 8. This is consistent with the chemical composition $Li_2{}^{+}Ge^{4+}Mo_3{}^{3.33+}O_8{}^{2-}$.

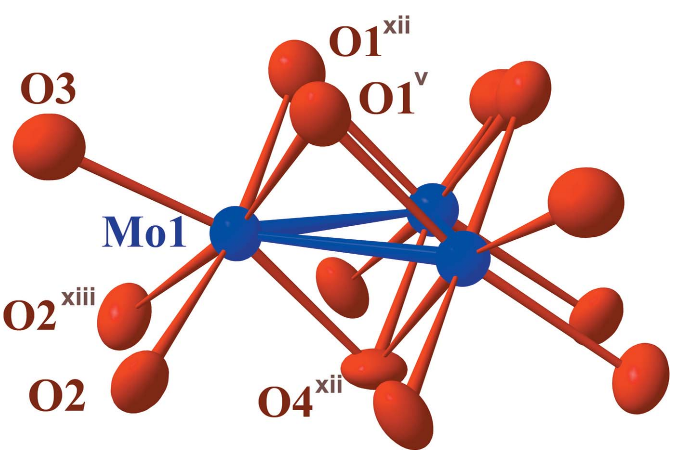


Figure 2
The Mo_3O_{13} cluster unit with its numbering scheme, with ellipsoids drawn at the 97% probability level. [Symmetry codes: (v) $y+1, -x+y+2, z-\frac{1}{2}$; (xii) $-x+3, -y+2, z-\frac{1}{2}$; (xiii) $-y+2, x-y+1, z$; (xiv) $-x+y+2, -x+3, z$.]

3. Database survey

The $M_2Mo_3O_8$ (Mg, Zn, Fe, Co, Ni, Zn and Cd) compounds containing triangular Mo_3 clusters were first synthesized by McCarroll *et al.* (1957). They presented the results of a structure determination on $Zn_2Mo_3O_8$ from photographic data (R = 0.118). Later, a refinement of the structure was accomplished by Ansell & Katz (1966) with an R factor of 0.069. Among the above compounds, it is interesting to note that $Fe_2Mo_3O_8$ is a mineral known as kamiokite (Kanazawa & Sasaki, 1986). Later, DeBenedittis & Katz (1965) reported the

Table 2
Experimental details.

Crystal data	
Chemical formula	$Li_2GeMo_3O_8$
M_r	502.29
Crystal system, space group	Hexagonal, $P6_3mc$
Temperature (K)	293
a, c (Å)	5.7268 (3), 9.9841 (6)
V (Å^3)	283.57 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	11.74
Crystal size (mm)	0.21 × 0.13 × 0.07
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Analytical (de Meulenaar & Tompa, 1965)
T_{min}, T_{max}	0.048, 0.157
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4457, 522, 501
R_{int}	0.063
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.807
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.076, 1.10
No. of reflections	522
No. of parameters	31
No. of restraints	1
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.43, −1.33
Absolute structure	Flack (1983), 247 Friedel pairs
Absolute structure parameter	0.01 (3)

Computer programs: *COLLECT* (Nonius, 1998), *EVALCCD* (Duisenberg *et al.*, 2003), *SIR97* (Altomare *et al.*, 1999), *SHELXL97* (Sheldrick, 2008) and *DIAMOND* (Bergerhoff, 1996).

existence of the LiRMo$_3$O$_8$ (R = Sc and Y) compounds. Subsequently, McCarroll (1977) obtained isotypic compounds with R = In, Sm, Gd, Tb, Dy, Ho, Er, and Yb. In 1985, Torardi & McCarley (1985) described the new Mo_3 cluster compounds $LiZn_2Mo_3O_8$, $Zn_3Mo_3O_8$ and $ScZnMo_3O_8$ and, in 2013, Gall *et al.* (2013*a*,*b*), the quaternary compounds $Li_2MMo_3O_8$ (M = Sn and In).

4. Synthesis and crystallization

Single crystals of $Li_2GeMo_3O_8$ were obtained by heating a mixture of Li_2MoO_4, O_2, MoO_3 and Mo with the nominal composition $Li_2GeMo_6O_{12}$ at 1923 K for 72 h in a molybdenum crucible sealed under low argon pressure using an arc-welding system. The molybdate Li_2MoO_4 was synthesized by heating an equimolar ratio of MoO_3 (CERAC 99.95%) and Li_2CO_3 (CERAC 99.9%) in an alumina vessel at 873 K in air over 12 h. Before use, the Mo powder was heated under a hydrogen flow at 1273 K for 6 h. The composition of the final crystals thus obtained was determined after a complete X-ray structural study on one of them.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All atoms were refined with anisotropic displacement parameters, except for the Li atoms, which were refined isotropically.

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supporting information

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Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT* (Nonius, 1998); 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* (Sheldrick, 2008).

Dilithium germanium trimolybdenum octaoxide

Crystal data

$Li_2GeMo_3O_8$
$M_r = 502.29$
Hexagonal, $P6_3mc$
Hall symbol: P 6c -2c
$a = 5.7268$ (3) Å
$c = 9.9841$ (6) Å
$V = 283.57$ (3) Å^3
$Z = 2$
$F(000) = 456$
$D_x = 5.883$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4457 reflections
$\theta = 4.1–35.0°$
$\mu = 11.74$ mm^{-1}
$T = 293$ K
Irregular block, black
0.21 × 0.13 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans ($\kappa = 0$) + additional ω scans
Absorption correction: analytical (de Meulenaar & Tompa, 1965)
$T_{min} = 0.048$, $T_{max} = 0.157$
4457 measured reflections
522 independent reflections
501 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 35.0°$, $\theta_{min} = 4.1°$
$h = -7 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.076$
$S = 1.10$
522 reflections
31 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.3716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.43$ e Å^{-3}
$\Delta\rho_{min} = -1.33$ e Å^{-3}
Absolute structure: Flack (1983), 247 Friedel pairs
Absolute structure parameter: 0.01 (3)

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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
Li1	1.0000	1.0000	1.581 (2)	0.014 (3)*
Li2	1.3333	0.6667	1.490 (2)	0.014 (3)*
Ge1	1.3333	0.6667	1.09391 (9)	0.0076 (2)
Mo1	1.37881 (9)	1.18940 (4)	1.30891 (9)	0.00660 (13)
O1	1.4795 (4)	0.5205 (4)	1.9536 (5)	0.0081 (8)
O2	1.1704 (6)	0.8296 (6)	1.1906 (5)	0.0081 (8)
O3	1.0000	1.0000	1.3974 (7)	0.0110 (14)
O4	1.3333	0.6667	1.6680 (8)	0.0080 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ge1	0.0076 (3)	0.0076 (3)	0.0076 (4)	0.00381 (14)	0.000	0.000
Mo1	0.00622 (19)	0.00662 (16)	0.00683 (19)	0.00311 (10)	−0.00015 (18)	−0.00007 (9)
O1	0.0079 (13)	0.0079 (13)	0.0094 (18)	0.0045 (14)	0.0007 (8)	−0.0007 (8)
O2	0.0071 (12)	0.0071 (12)	0.010 (2)	0.0034 (13)	0.0017 (9)	−0.0017 (9)
O3	0.012 (2)	0.012 (2)	0.010 (3)	0.0058 (10)	0.000	0.000
O4	0.0102 (19)	0.0102 (19)	0.004 (3)	0.0051 (9)	0.000	0.000

Geometric parameters (Å, º)

Li1—O3	1.84 (2)	Ge1—O1^{xv}	2.016 (5)
Li1—O2^{i}	2.012 (13)	Ge1—O1^{xvi}	2.016 (4)
Li1—O2^{ii}	2.012 (13)	Mo1—O4^{xvii}	2.004 (6)
Li1—O2^{iii}	2.012 (13)	Mo1—O1^{xvii}	2.039 (4)
Li1—Mo1^{i}	2.949 (17)	Mo1—O1^{viii}	2.039 (4)
Li1—Mo1^{iii}	2.949 (17)	Mo1—O3	2.076 (3)
Li1—Mo1^{ii}	2.949 (17)	Mo1—O2	2.146 (3)
Li1—Mo1^{iv}	3.305 (18)	Mo1—O2^{v}	2.146 (3)
Li1—Mo1^{v}	3.305 (18)	Mo1—Mo1^{xviii}	2.4728 (8)
Li1—Mo1	3.305 (18)	Mo1—Mo1^{xix}	2.4728 (8)
Li1—Li2	3.430 (9)	Mo1—Li1^{xx}	2.949 (17)
Li1—Li2^{vi}	3.430 (9)	O1—Li2^{xxi}	1.892 (6)
Li2—O4	1.78 (3)	O1—Ge1^{xxii}	2.016 (4)
Li2—O1^{vii}	1.892 (6)	O1—Mo1^{xxiii}	2.039 (4)
Li2—O1^{viii}	1.892 (6)	O1—Mo1^{xxiv}	2.039 (4)

Li2—O1^{ix}	1.892 (6)	O2—Li1^{xx}	2.012 (13)
Li2—Li1^{x}	3.430 (9)	O2—Mo1^{iv}	2.146 (3)
Li2—Li1^{xi}	3.430 (9)	O3—Mo1^{iv}	2.076 (3)
Ge1—O2	1.883 (5)	O3—Mo1^{v}	2.076 (3)
Ge1—O2^{xii}	1.883 (5)	O4—Mo1^{iii}	2.004 (5)
Ge1—O2^{xiii}	1.883 (5)	O4—Mo1^{xxiv}	2.004 (5)
Ge1—O1^{xiv}	2.016 (5)	O4—Mo1^{xxiii}	2.004 (5)
O3—Li1—O2^{i}	122.9 (5)	Li1^{x}—Li2—Li1^{xi}	113.2 (5)
O3—Li1—O2^{ii}	122.9 (5)	O2—Ge1—O2^{xii}	96.1 (2)
O2^{i}—Li1—O2^{ii}	93.3 (7)	O2—Ge1—O2^{xiii}	96.1 (2)
O3—Li1—O2^{iii}	122.9 (5)	O2^{xii}—Ge1—O2^{xiii}	96.1 (2)
O2^{i}—Li1—O2^{iii}	93.3 (7)	O2—Ge1—O1^{xiv}	92.73 (17)
O2^{ii}—Li1—O2^{iii}	93.3 (7)	O2^{xii}—Ge1—O1^{xiv}	166.8 (2)
O3—Li1—Mo1^{i}	140.4 (3)	O2^{xiii}—Ge1—O1^{xiv}	92.73 (18)
O2^{i}—Li1—Mo1^{i}	46.7 (4)	O2—Ge1—O1^{xv}	92.73 (17)
O2^{ii}—Li1—Mo1^{i}	46.7 (4)	O2^{xii}—Ge1—O1^{xv}	92.73 (17)
O2^{iii}—Li1—Mo1^{i}	96.7 (8)	O2^{xiii}—Ge1—O1^{xv}	166.8 (2)
O3—Li1—Mo1^{iii}	140.4 (3)	O1^{xiv}—Ge1—O1^{xv}	77.0 (2)
O2^{i}—Li1—Mo1^{iii}	46.7 (4)	O2—Ge1—O1^{xvi}	166.8 (2)
O2^{ii}—Li1—Mo1^{iii}	96.7 (8)	O2^{xii}—Ge1—O1^{xvi}	92.73 (17)
O2^{iii}—Li1—Mo1^{iii}	46.7 (4)	O2^{xiii}—Ge1—O1^{xvi}	92.73 (17)
Mo1^{i}—Li1—Mo1^{iii}	67.0 (4)	O1^{xiv}—Ge1—O1^{xvi}	77.0 (2)
O3—Li1—Mo1^{ii}	140.4 (3)	O1^{xv}—Ge1—O1^{xvi}	77.0 (2)
O2^{i}—Li1—Mo1^{ii}	96.7 (8)	O4^{xvii}—Mo1—O1^{xvii}	104.58 (15)
O2^{ii}—Li1—Mo1^{ii}	46.7 (4)	O4^{xvii}—Mo1—O1^{viii}	104.58 (15)
O2^{iii}—Li1—Mo1^{ii}	46.7 (4)	O1^{xvii}—Mo1—O1^{viii}	76.0 (2)
Mo1^{i}—Li1—Mo1^{ii}	67.0 (4)	O4^{xvii}—Mo1—O3	160.6 (3)
Mo1^{iii}—Li1—Mo1^{ii}	67.0 (4)	O1^{xvii}—Mo1—O3	90.60 (16)
O3—Li1—Mo1^{iv}	34.6 (2)	O1^{viii}—Mo1—O3	90.60 (16)
O2^{i}—Li1—Mo1^{iv}	133.2 (4)	O4^{xvii}—Mo1—O2	87.53 (16)
O2^{ii}—Li1—Mo1^{iv}	133.2 (4)	O1^{xvii}—Mo1—O2	167.40 (14)
O2^{iii}—Li1—Mo1^{iv}	88.2 (3)	O1^{viii}—Mo1—O2	97.8 (2)
Mo1^{i}—Li1—Mo1^{iv}	175.1 (5)	O3—Mo1—O2	78.36 (18)
Mo1^{iii}—Li1—Mo1^{iv}	116.94 (6)	O4^{xvii}—Mo1—O2^{v}	87.53 (16)
Mo1^{ii}—Li1—Mo1^{iv}	116.94 (6)	O1^{xvii}—Mo1—O2^{v}	97.8 (2)
O3—Li1—Mo1^{v}	34.6 (2)	O1^{viii}—Mo1—O2^{v}	167.40 (14)
O2^{i}—Li1—Mo1^{v}	133.2 (4)	O3—Mo1—O2^{v}	78.36 (18)
O2^{ii}—Li1—Mo1^{v}	88.2 (3)	O2—Mo1—O2^{v}	86.0 (3)
O2^{iii}—Li1—Mo1^{v}	133.2 (4)	O4^{xvii}—Mo1—Mo1^{xviii}	51.91 (12)
Mo1^{i}—Li1—Mo1^{v}	116.94 (6)	O1^{xvii}—Mo1—Mo1^{xviii}	52.67 (9)
Mo1^{iii}—Li1—Mo1^{v}	175.1 (5)	O1^{viii}—Mo1—Mo1^{xviii}	90.54 (10)
Mo1^{ii}—Li1—Mo1^{v}	116.94 (6)	O3—Mo1—Mo1^{xviii}	141.60 (11)
Mo1^{iv}—Li1—Mo1^{v}	59.0 (4)	O2—Mo1—Mo1^{xviii}	139.30 (11)
O3—Li1—Mo1	34.6 (2)	O2^{v}—Mo1—Mo1^{xviii}	94.37 (14)
O2^{i}—Li1—Mo1	88.2 (3)	O4^{xvii}—Mo1—Mo1^{xix}	51.91 (12)
O2^{ii}—Li1—Mo1	133.2 (4)	O1^{xvii}—Mo1—Mo1^{xix}	90.54 (9)
O2^{iii}—Li1—Mo1	133.2 (4)	O1^{viii}—Mo1—Mo1^{xix}	52.67 (9)

Mo1^{i}—Li1—Mo1	116.94 (6)	O3—Mo1—Mo1^{xix}	141.60 (11)
Mo1^{iii}—Li1—Mo1	116.94 (6)	O2—Mo1—Mo1^{xix}	94.37 (13)
Mo1^{ii}—Li1—Mo1	175.1 (5)	O2^{v}—Mo1—Mo1^{xix}	139.30 (11)
Mo1^{iv}—Li1—Mo1	59.0 (4)	Mo1^{xviii}—Mo1—Mo1^{xix}	60.0
Mo1^{v}—Li1—Mo1	59.0 (4)	O4^{xvii}—Mo1—Li1^{xx}	85.0 (3)
O3—Li1—Li2	74.6 (5)	O1^{xvii}—Mo1—Li1^{xx}	139.97 (14)
O2^{i}—Li1—Li2	74.9 (3)	O1^{viii}—Mo1—Li1^{xx}	139.97 (15)
O2^{ii}—Li1—Li2	162.6 (10)	O3—Mo1—Li1^{xx}	75.6 (3)
O2^{iii}—Li1—Li2	74.9 (3)	O2—Mo1—Li1^{xx}	43.02 (16)
Mo1^{i}—Li1—Li2	120.8 (5)	O2^{v}—Mo1—Li1^{xx}	43.02 (16)
Mo1^{iii}—Li1—Li2	65.8 (4)	Mo1^{xviii}—Mo1—Li1^{xx}	123.5 (2)
Mo1^{ii}—Li1—Li2	120.8 (5)	Mo1^{xix}—Mo1—Li1^{xx}	123.5 (2)
Mo1^{iv}—Li1—Li2	60.5 (4)	O4^{xvii}—Mo1—Li1	169.2 (3)
Mo1^{v}—Li1—Li2	109.2 (7)	O1^{xvii}—Mo1—Li1	67.23 (18)
Mo1—Li1—Li2	60.5 (4)	O1^{viii}—Mo1—Li1	67.23 (18)
O3—Li1—Li2^{vi}	74.6 (5)	O3—Mo1—Li1	30.2 (3)
O2^{i}—Li1—Li2^{vi}	162.6 (10)	O2—Mo1—Li1	100.31 (19)
O2^{ii}—Li1—Li2^{vi}	74.9 (3)	O2^{v}—Mo1—Li1	100.31 (19)
O2^{iii}—Li1—Li2^{vi}	74.9 (3)	Mo1^{xviii}—Mo1—Li1	119.49 (18)
Mo1^{i}—Li1—Li2^{vi}	120.8 (5)	Mo1^{xix}—Mo1—Li1	119.49 (18)
Mo1^{iii}—Li1—Li2^{vi}	120.8 (5)	Li1^{xx}—Mo1—Li1	105.78 (5)
Mo1^{ii}—Li1—Li2^{vi}	65.8 (4)	Li2^{xxi}—O1—Ge1^{xxii}	124.9 (8)
Mo1^{iv}—Li1—Li2^{vi}	60.5 (4)	Li2^{xxi}—O1—Mo1^{xxiii}	119.4 (6)
Mo1^{v}—Li1—Li2^{vi}	60.5 (4)	Ge1^{xxii}—O1—Mo1^{xxiii}	103.46 (15)
Mo1—Li1—Li2^{vi}	109.2 (7)	Li2^{xxi}—O1—Mo1^{xxiv}	119.4 (6)
Li2—Li1—Li2^{vi}	113.2 (5)	Ge1^{xxii}—O1—Mo1^{xxiv}	103.46 (15)
O4—Li2—O1^{vii}	101.1 (7)	Mo1^{xxiii}—O1—Mo1^{xxiv}	74.66 (17)
O4—Li2—O1^{viii}	101.1 (7)	Ge1—O2—Li1^{xx}	116.3 (6)
O1^{vii}—Li2—O1^{viii}	116.4 (5)	Ge1—O2—Mo1	125.62 (15)
O4—Li2—O1^{ix}	101.1 (7)	Li1^{xx}—O2—Mo1	90.3 (4)
O1^{vii}—Li2—O1^{ix}	116.4 (5)	Ge1—O2—Mo1^{iv}	125.62 (15)
O1^{viii}—Li2—O1^{ix}	116.4 (5)	Li1^{xx}—O2—Mo1^{iv}	90.3 (4)
O4—Li2—Li1	74.6 (5)	Mo1—O2—Mo1^{iv}	98.6 (2)
O1^{vii}—Li2—Li1	65.0 (2)	Li1—O3—Mo1^{iv}	115.18 (19)
O1^{viii}—Li2—Li1	65.0 (2)	Li1—O3—Mo1	115.18 (19)
O1^{ix}—Li2—Li1	175.6 (12)	Mo1^{iv}—O3—Mo1	103.2 (2)
O4—Li2—Li1^{x}	74.6 (5)	Li1—O3—Mo1^{v}	115.18 (19)
O1^{vii}—Li2—Li1^{x}	175.6 (12)	Mo1^{iv}—O3—Mo1^{v}	103.2 (2)
O1^{viii}—Li2—Li1^{x}	65.0 (2)	Mo1—O3—Mo1^{v}	103.2 (2)
O1^{ix}—Li2—Li1^{x}	65.0 (2)	Li2—O4—Mo1^{iii}	134.58 (16)
Li1—Li2—Li1^{x}	113.2 (5)	Li2—O4—Mo1^{xxiv}	134.58 (16)
O4—Li2—Li1^{xi}	74.6 (5)	Mo1^{iii}—O4—Mo1^{xxiv}	76.2 (2)
O1^{vii}—Li2—Li1^{xi}	65.0 (2)	Li2—O4—Mo1^{xxiii}	134.58 (16)
O1^{viii}—Li2—Li1^{xi}	175.6 (12)	Mo1^{iii}—O4—Mo1^{xxiii}	76.2 (2)

O1^{ix}—Li2—Li1^{xi}	65.0 (2)	Mo1^{xxiv}—O4—Mo1^{xxiii}	76.2 (2)
Li1—Li2—Li1^{xi}	113.2 (5)		

Symmetry codes: (i) $x-y+1, x, z+1/2$; (ii) $-x+2, -y+2, z+1/2$; (iii) $y, -x+y+1, z+1/2$; (iv) $-x+y+1, -x+2, z$; (v) $-y+2, x-y+1, z$; (vi) $x-1, y, z$; (vii) $x-y, x-1, z-1/2$; (viii) $y+1, -x+y+2, z-1/2$; (ix) $-x+3, -y+1, z-1/2$; (x) $x+1, y, z$; (xi) $x, y-1, z$; (xii) $-x+y+2, -x+2, z$; (xiii) $-y+2, x-y, z$; (xiv) $-x+y+2, -x+2, z-1$; (xv) $-y+2, x-y, z-1$; (xvi) $x, y, z-1$; (xvii) $-x+3, -y+2, z-1/2$; (xviii) $-x+y+2, -x+3, z$; (xix) $-y+3, x-y+1, z$; (xx) $-x+2, -y+2, z-1/2$; (xxi) $-x+3, -y+1, z+1/2$; (xxii) $x, y, z+1$; (xxiii) $x-y+1, x-1, z+1/2$; (xxiv) $-x+3, -y+2, z+1/2$.