

Synthesis and crystal structure of $\text{NaMgFe}(\text{MoO}_4)_3$

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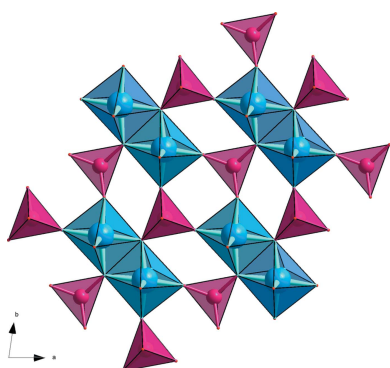
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Canada**Keywords:** crystal structure; iron molybdate;
anionic framework; Na–Fe–Mo–O system.**CCDC reference:** 1481125**Supporting information:** this article has
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The iron molybdate $\text{NaMgFe}(\text{MoO}_4)_3$ {sodium magnesium iron(III) tris-[molybdate(VI)]} has been synthesized by the flux method. This compound is isostructural with $\alpha\text{-NaFe}_2(\text{MoO}_4)_3$ and crystallizes in the triclinic space group $P\bar{1}$. Its structure is built up from $[\text{Mg,Fe}]_2\text{O}_{10}$ units of edge-sharing $[\text{Mg,Fe}]\text{O}_6$ octahedra which are linked to each other through the common corners of $[\text{MoO}_4]$ tetrahedra. The resulting anionic three-dimensional framework leads to the formation of channels along the $[101]$ direction in which the Na^+ cations are located.

1. Chemical context

Iron molybdates have been subject to very intensive research as a result of their numerous applications including as catalysts (Tian *et al.*, 2011), multiferroic properties and more recently as a possible positive electrode in rechargeable batteries (Sinyakov *et al.*, 1978; Maćzka *et al.*, 2011; Devi & Varadaraju, 2012). In these materials, the anionic framework is constructed from MoO_4 tetrahedra linked to the iron coordination polyhedra, leading to a large variety of crystal structures with a high capacity for cationic and anionic substitutions.

Until now, a total of six orthomolybdate compounds have been reported in the Na–Fe–Mo–O system: $\text{Na}_9\text{Fe}(\text{MoO}_4)_6$ (Savina *et al.*, 2013); $\text{NaFe}(\text{MoO}_4)_2$ (Klevtsova, 1975); $\alpha\text{-NaFe}_2(\text{MoO}_4)_3$, $\beta\text{-NaFe}_2(\text{MoO}_4)_3$ and $\text{Na}_3\text{Fe}_2(\text{MoO}_4)_3$ (Muessig *et al.*, 2003); $\text{NaFe}_4(\text{MoO}_4)_5$ (Ehrenberg *et al.*, 2006). Their structures are described in terms of three-dimensional networks of isolated $[\text{MoO}_4]$ tetrahedra and $[\text{FeO}_6]$ octahedra. The sodium and mixed-valence iron molybdate $\text{NaFe}_2(\text{MoO}_4)_3$ exhibits two polymorphs, both crystallizing in the triclinic system. The low-temperature α -phase changes irreversibly at high temperature into a β -phase. In addition to these orthomolybdate compounds, another phase with the formula $\text{Na}_3\text{Fe}_2\text{Mo}_5\text{O}_{16}$ and with layers of Mo_3O_{13} units consisting of $[\text{MoO}_6]$ octahedra has been synthesized and characterized (Bramnik *et al.*, 2003). In addition, Kozhevnikova & Imekhenova (2009) have investigated the $\text{Na}_2\text{MoO}_4\text{-MMoO}_4\text{-Fe}_2(\text{MoO}_4)_3$ system ($M = \text{Mg, Mn, Ni, Co}$) and have attributed the Nasicon-type structure with space group $R\bar{3}c$ (Kotova & Kozhevnikova, 2003; Kozhevnikova & Imekhenova, 2009) to the phase of variable composition $\text{Na}_{(1-x)}M_{(1-x)}\text{Fe}_{(1+x)}(\text{MoO}_4)_3$. More recently, $\text{NaNiFe}(\text{MoO}_4)_3$ and $\text{NaZnFe}(\text{MoO}_4)_3$ (Mhiri *et al.*, 2015) were found to be isostructural to $\beta\text{-NaFe}_2(\text{MoO}_4)_3$ and to have a good ionic conductivity with low activation energy, close to those of Nasicon-type compounds with similar formula such as $\text{AZr}_2(\text{PO}_4)_3$ ($A = \text{Na, Li}$). As an extension of the previous work, we report here on the synthesis and characterization by



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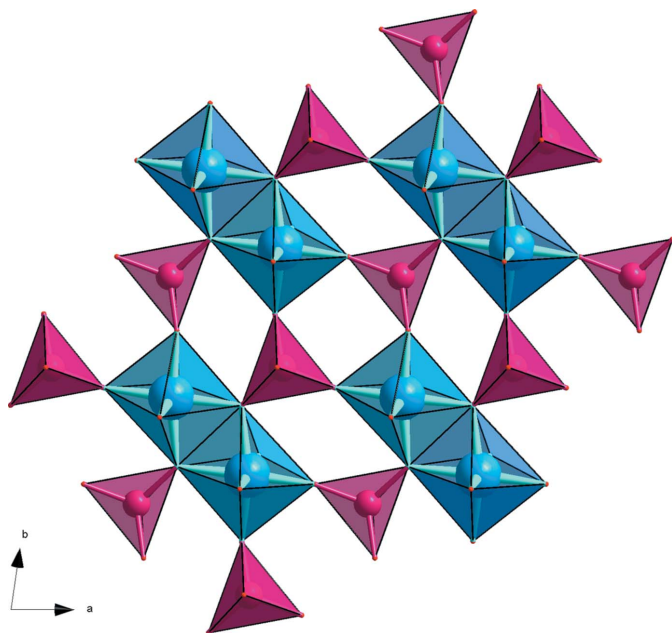


Figure 1
 $[\text{Mg,Fe}]_2\text{O}_{10}$ units parallel to $[1\bar{1}0]$ in $\text{NaMgFe}(\text{MoO}_4)_3$ structure. $[\text{Mg,Fe}]_2\text{O}_{10}$ dimers are shown in blue and MoO_4 tetrahedra in purple.

X-ray diffraction of a new compound, $\text{NaMgFe}(\text{MoO}_4)_3$, which is isostructural with $\alpha\text{-NaFe}_2(\text{MoO}_4)_3$.

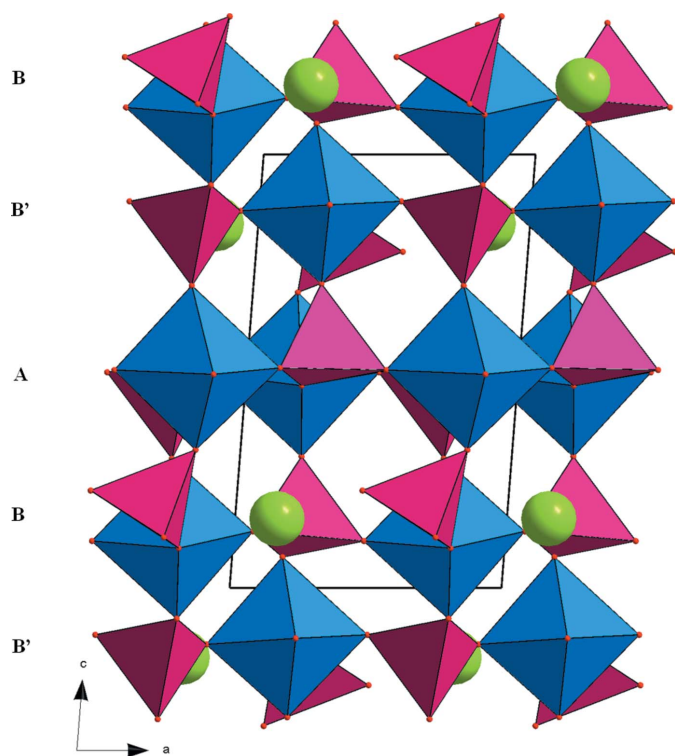


Figure 2
 Projection of the $\text{NaMgFe}(\text{MoO}_4)_3$ structure along the b axis. $[\text{Mg,Fe}]_2\text{O}_{10}$ dimers are shown in blue; MoO_4 tetrahedra in purple and Na^+ cations as green spheres.

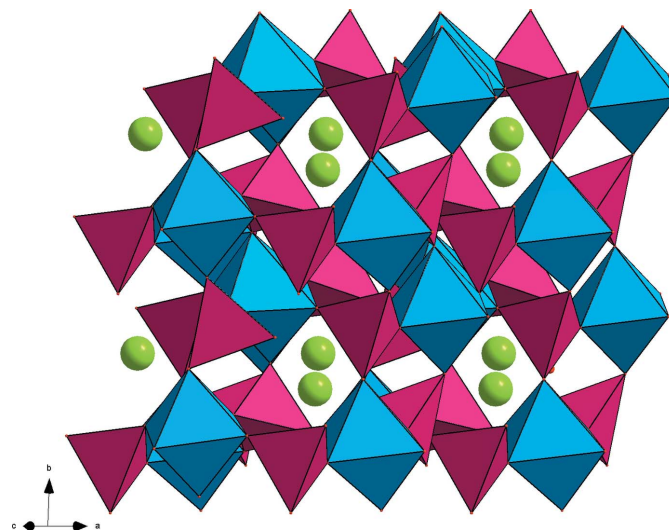


Figure 3
 Channels along $[101]$ in the structure of $\text{NaMgFe}(\text{MoO}_4)_3$. $[\text{Mg,Fe}]_2\text{O}_{10}$ dimers are shown in blue, MoO_4 tetrahedra in purple and Na^+ cations as green spheres.

2. Structural commentary

The title $\text{NaMgFe}(\text{MoO}_4)_3$ structure is based on a three-dimensional framework of $[\text{Mg,Fe}]_2\text{O}_{10}$ units of edge-sharing $[\text{Mg,Fe}]\text{O}_6$ octahedra, connected to each other through the common corners of $[\text{MoO}_4]$ tetrahedra. All $[\text{Mg,Fe}]_2\text{O}_{10}$ units are parallel to $[1\bar{1}0]$ (Fig. 1). In this structure, two types of layers (A and B), similar to those observed in $\alpha\text{-NaFe}_2(\text{MoO}_4)_3$, are aligned parallel to (110) with the sequence $-A-B-B'-A-B-B'-$ and stacked along $[001]$. B' layers are obtained from B by an inversion centre located on the A planes (Fig. 2). The resulting anionic three-dimensional framework leads to the formation of channels along $[101]$ in which the sodium ions are located (Fig. 3).

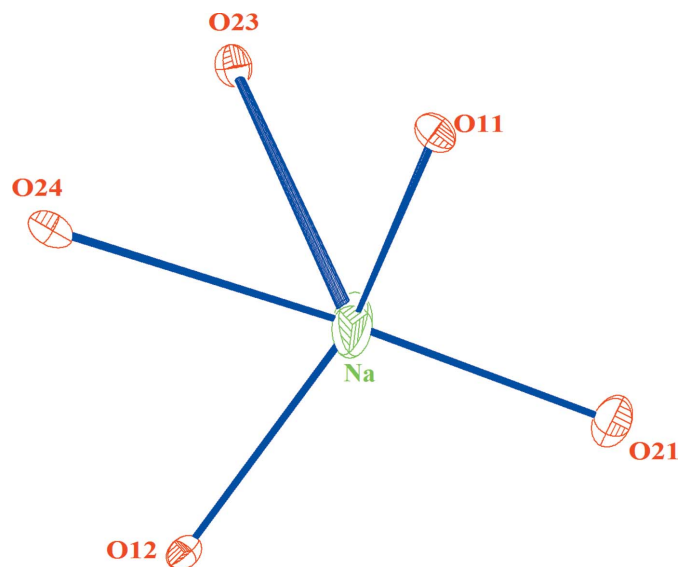


Figure 4
 The environment of the Na^+ cation showing displacement ellipsoids drawn at the 50% probability level.

Table 1
Experimental details.

Crystal data	
Chemical formula	NaMgFe(MoO ₄) ₃
<i>M_r</i>	582.97
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.900 (4), 6.928 (1), 11.055 (1)
α , β , γ (°)	80.24 (1), 83.55 (2), 80.22 (3)
<i>V</i> (Å ³)	511.3 (3)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	5.15
Crystal size (mm)	0.28 × 0.14 × 0.07
Data collection	
Diffractometer	Enraf–Nonius TurboCAD-4
Absorption correction	ψ scan (North <i>et al.</i> , 1968)
<i>T</i> _{min} , <i>T</i> _{max}	0.478, 0.695
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	3429, 2983, 2850
<i>R</i> _{int}	0.014
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.703
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.025, 0.068, 1.19
No. of reflections	2983
No. of parameters	168
No. of restraints	4
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	1.47, -1.60

Computer programs: *CAD-4 EXPRESS* (Enraf–Nonius, 1994), *XCAD4* (Harms & Wocadlo, 1995), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014/7* (Sheldrick, 2011), *DIAMOND* (Brandenburg & Putz, 1999) and *WinGX* publication routines (Farrugia, 2012).

In the title structure, all atoms are located in general positions. The three crystallographically different molybdenum atoms have a tetrahedral coordination with Mo–O distances between 1.715 (3) and 1.801 (2) Å. The mean distances (Mo1–O = 1.762, Mo2–O = 1.766 and Mo3–O = 1.760 Å) are in good accordance with those usually observed in molybdates (Abrahams *et al.*, 1967; Harrison & Cheetham, 1989; Smit *et al.*, 2006). The [Mg,Fe]–O distances and the *cis* O–[Mg,Fe]–O angles in the [Mg,Fe]₂O₁₀ units range from 2.003 (3) to 2.099 (3) Å and from 81.2 (1) to 177.8 (1)°, respectively. This dispersion reflects a slight distortion of the [Mg,Fe]O₆ octahedra. The average distances [Mg,Fe]1–O = 2.059 and [Mg,Fe]2–O = 2.013 Å lie between the values of 1.990 Å observed for six-coordinated Fe³⁺ in LiFe(MoO₄)₂ (van der Lee *et al.* 2008) and 2.072 Å reported for Mg²⁺ with the same coordination in NaMg₃Al(MoO₄)₅ (Hermanowicz *et al.*, 2006). This result is related to the disordered distribution of Fe³⁺ and Mg²⁺ in both sites. Assuming sodium–oxygen distances below 3.13 Å (Donnay & Allmann, 1970), the Na site is surrounded by five oxygen atoms (Fig. 4).

3. Synthesis and crystallization

Crystals of the title compound were grown in a flux of sodium dimolybdate Na₂Mo₂O₇ with an atomic ratio Na:Mg:Fe:Mo = 5:1:1:7. Appropriate amounts of the starting reactants NaNO₃, Mg(NO₃)₂·6H₂O, Fe(NO₃)₃·9H₂O and (NH₄)₆Mo₇O₂₄·4H₂O were dissolved in nitric acid and the resulting solution was

evaporated to dryness. The dry residue was then placed in a platinum crucible and slowly heated in air up to 673 K for 24 h to remove H₂O and NH₃. The mixture was ground in an agate mortar, melted for 2 h at 1123 K and then cooled to room temperature at a rate of 5 K h⁻¹. Crystals without regular shape were separated from the flux by washing in boiling water.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The application of the direct methods revealed two sites, labeled *M*(1) and *M*(2), statistically occupied by the Fe³⁺ and Mg²⁺ ions. This distribution was supported by the *M*1–O and *M*2–O distances which are between the classical values for pure Mg–O and Fe–O bonds. Succeeding difference Fourier synthesis led to the positions of all the remaining atoms.

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supporting information

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Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 201); molecular graphics: *DIAMOND* (Brandenburg & Putz, 1999); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012).

(I)

Crystal data

FeMgMo₃NaO₁₂

$M_r = 582.97$

Triclinic, $P\bar{1}$

$a = 6.900$ (4) Å

$b = 6.928$ (1) Å

$c = 11.055$ (1) Å

$\alpha = 80.24$ (1)°

$\beta = 83.55$ (2)°

$\gamma = 80.22$ (3)°

$V = 511.3$ (3) Å³

$Z = 2$

$F(000) = 542$

$D_x = 3.786$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9.1$ – 11.4 °

$\mu = 5.15$ mm⁻¹

$T = 293$ K

Prism, brown

$0.28 \times 0.14 \times 0.07$ mm

Data collection

Enraf–Nonius TurboCAD-4

diffractometer

Radiation source: fine-focus sealed tube

non-profiled $\omega/2\tau$ scans

Absorption correction: ψ scan

(North *et al.*, 1968)

$T_{\min} = 0.478$, $T_{\max} = 0.695$

3429 measured reflections

2983 independent reflections

2850 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 30.0$ °, $\theta_{\min} = 3.0$ °

$h = -9$ → 9

$k = -9$ → 9

$l = -1$ → 15

2 standard reflections every 120 min

intensity decay: 1.1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.068$

$S = 1.19$

2983 reflections

168 parameters

4 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 2.3858P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.47$ e Å⁻³

$\Delta\rho_{\min} = -1.60$ e Å⁻³

Extinction correction: SHELXL-2014/7
 (Sheldrick 2015),
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0074 (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)
Na	0.8586 (4)	0.5914 (4)	0.8148 (4)	0.0757 (12)	
Mg1	0.8152 (1)	0.1703 (1)	0.50854 (8)	0.00851 (16)	0.7558 (7)
Fe1	0.8152 (1)	0.1703 (1)	0.50854 (8)	0.00851 (16)	0.2442 (7)
Mg2	0.77528 (8)	0.77491 (8)	0.11025 (5)	0.00785 (12)	0.2442 (7)
Fe2	0.77528 (8)	0.77491 (8)	0.11025 (5)	0.00785 (12)	0.7558 (7)
Mo1	0.75910 (4)	0.10066 (4)	0.85110 (2)	0.00799 (8)	
O11	0.8166 (4)	0.8508 (4)	0.9264 (2)	0.0125 (5)	
O12	0.9297 (4)	0.2547 (4)	0.8737 (3)	0.0146 (5)	
O13	0.5170 (4)	0.2053 (4)	0.8938 (3)	0.0158 (5)	
O14	0.7784 (5)	0.0889 (4)	0.6953 (2)	0.0185 (5)	
Mo2	0.70522 (4)	0.28318 (4)	0.18835 (3)	0.00950 (8)	
O21	0.4579 (4)	0.3458 (5)	0.2289 (3)	0.0232 (6)	
O22	0.7436 (4)	0.0675 (4)	0.1148 (3)	0.0185 (5)	
O23	0.8372 (4)	0.2322 (4)	0.3205 (2)	0.0173 (5)	
O24	0.8015 (4)	0.4878 (4)	0.0918 (2)	0.0148 (5)	
Mo3	0.27372 (4)	0.29658 (4)	0.54507 (2)	0.00732 (8)	
O31	0.1224 (4)	0.1328 (4)	0.5056 (2)	0.0113 (4)	
O32	0.2458 (5)	0.2976 (4)	0.7045 (2)	0.0194 (5)	
O33	0.5183 (4)	0.2083 (4)	0.5042 (3)	0.0172 (5)	
O34	0.2067 (4)	0.5383 (4)	0.4690 (3)	0.0153 (5)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na	0.0293 (12)	0.0255 (11)	0.186 (4)	0.0089 (9)	-0.0496 (18)	-0.0429 (17)
Mg1	0.0079 (4)	0.0081 (4)	0.0091 (4)	-0.0007 (3)	-0.0018 (3)	0.0001 (3)
Fe1	0.0079 (4)	0.0081 (4)	0.0091 (4)	-0.0007 (3)	-0.0018 (3)	0.0001 (3)
Mg2	0.0080 (2)	0.0073 (2)	0.0079 (2)	-0.00174 (18)	-0.00177 (18)	0.00103 (18)
Fe2	0.0080 (2)	0.0073 (2)	0.0079 (2)	-0.00174 (18)	-0.00177 (18)	0.00103 (18)
Mo1	0.00697 (13)	0.00870 (13)	0.00770 (13)	-0.00175 (9)	-0.00095 (9)	0.00122 (9)
O11	0.0163 (12)	0.0106 (11)	0.0094 (11)	-0.0013 (9)	-0.0021 (9)	0.0019 (8)

O12	0.0112 (11)	0.0152 (12)	0.0182 (12)	-0.0052 (9)	-0.0040 (9)	0.0003 (10)
O13	0.0090 (11)	0.0178 (12)	0.0194 (13)	-0.0009 (9)	-0.0016 (9)	-0.0005 (10)
O14	0.0258 (14)	0.0185 (13)	0.0092 (11)	-0.0021 (11)	-0.0017 (10)	0.0018 (10)
Mo2	0.01041 (14)	0.00802 (13)	0.00985 (13)	-0.00235 (9)	-0.00173 (9)	0.00076 (9)
O21	0.0133 (12)	0.0295 (16)	0.0261 (15)	-0.0028 (11)	-0.0017 (11)	-0.0026 (12)
O22	0.0226 (14)	0.0110 (12)	0.0229 (14)	-0.0050 (10)	-0.0032 (11)	-0.0017 (10)
O23	0.0192 (13)	0.0188 (13)	0.0125 (12)	-0.0009 (10)	-0.0033 (10)	0.0005 (10)
O24	0.0203 (13)	0.0100 (11)	0.0129 (11)	-0.0016 (9)	0.0019 (9)	-0.0012 (9)
Mo3	0.00771 (13)	0.00787 (13)	0.00674 (13)	-0.00290 (9)	-0.00105 (9)	-0.00015 (9)
O31	0.0093 (10)	0.0089 (10)	0.0167 (12)	-0.0027 (8)	-0.0026 (9)	-0.0027 (9)
O32	0.0266 (15)	0.0239 (14)	0.0088 (11)	-0.0081 (11)	-0.0019 (10)	-0.0010 (10)
O33	0.0107 (11)	0.0198 (13)	0.0212 (13)	-0.0025 (10)	-0.0018 (10)	-0.0025 (10)
O34	0.0189 (13)	0.0086 (11)	0.0179 (12)	-0.0027 (9)	-0.0027 (10)	0.0008 (9)

Geometric parameters (Å, °)

Na—O21 ⁱ	2.244 (4)	Mg2—O32 ⁱ	2.019 (3)
Na—O12	2.296 (4)	Mg2—O12 ⁱⁱ	2.036 (3)
Na—O11	2.308 (4)	Mo1—O14	1.727 (3)
Na—O24 ⁱⁱ	2.604 (4)	Mo1—O13	1.751 (3)
Na—O23 ⁱⁱ	2.772 (5)	Mo1—O12	1.780 (3)
Mg1—O33	2.025 (3)	Mo1—O11 ^{vii}	1.789 (3)
Mg1—O23	2.044 (3)	Mo2—O21	1.715 (3)
Mg1—O14	2.045 (3)	Mo2—O23	1.761 (3)
Mg1—O34 ⁱ	2.054 (3)	Mo2—O22	1.787 (3)
Mg1—O31 ⁱⁱⁱ	2.089 (3)	Mo2—O24	1.799 (3)
Mg1—O31 ^{iv}	2.099 (3)	Mo3—O33	1.731 (3)
Mg2—O13 ⁱ	2.003 (3)	Mo3—O32	1.753 (3)
Mg2—O24	2.009 (3)	Mo3—O34	1.753 (3)
Mg2—O22 ^v	2.010 (3)	Mo3—O31	1.801 (2)
Mg2—O11 ^{vi}	2.012 (3)		
O21 ⁱ —Na—O12	106.29 (15)	O24—Mg2—O11 ^{vi}	90.58 (11)
O21 ⁱ —Na—O11	92.34 (14)	O22 ^v —Mg2—O11 ^{vi}	85.22 (11)
O12—Na—O11	131.5 (2)	O13 ⁱ —Mg2—O32 ⁱ	91.56 (12)
O21 ⁱ —Na—O24 ⁱⁱ	169.3 (2)	O24—Mg2—O32 ⁱ	90.53 (12)
O12—Na—O24 ⁱⁱ	71.63 (12)	O22 ^v —Mg2—O32 ⁱ	93.77 (12)
O11—Na—O24 ⁱⁱ	81.96 (12)	O11 ^{vi} —Mg2—O32 ⁱ	175.79 (12)
O21 ⁱ —Na—O23 ⁱⁱ	125.19 (19)	O13 ⁱ —Mg2—O12 ⁱⁱ	176.14 (11)
O12—Na—O23 ⁱⁱ	115.39 (14)	O24—Mg2—O12 ⁱⁱ	90.70 (12)
O11—Na—O23 ⁱⁱ	85.84 (12)	O22 ^v —Mg2—O12 ⁱⁱ	91.08 (12)
O24 ⁱⁱ —Na—O23 ⁱⁱ	63.66 (10)	O11 ^{vi} —Mg2—O12 ⁱⁱ	91.24 (11)
O33—Mg1—O23	88.07 (12)	O32 ⁱ —Mg2—O12 ⁱⁱ	84.69 (12)
O33—Mg1—O14	88.89 (12)	O14—Mo1—O13	108.16 (14)
O23—Mg1—O14	174.80 (12)	O14—Mo1—O12	106.87 (14)
O33—Mg1—O34 ⁱ	89.20 (12)	O13—Mo1—O12	110.69 (13)
O23—Mg1—O34 ⁱ	93.92 (12)	O14—Mo1—O11 ^{vii}	106.05 (13)
O14—Mg1—O34 ⁱ	90.26 (12)	O13—Mo1—O11 ^{vii}	111.66 (13)

O33—Mg1—O31 ⁱⁱⁱ	177.80 (12)	O12—Mo1—O11 ^{vii}	113.08 (12)
O23—Mg1—O31 ⁱⁱⁱ	89.73 (11)	O21—Mo2—O23	110.07 (14)
O14—Mg1—O31 ⁱⁱⁱ	93.30 (12)	O21—Mo2—O22	109.36 (15)
O34 ⁱ —Mg1—O31 ⁱⁱⁱ	91.09 (11)	O23—Mo2—O22	109.03 (13)
O33—Mg1—O31 ^{iv}	98.59 (12)	O21—Mo2—O24	110.70 (14)
O23—Mg1—O31 ^{iv}	88.75 (11)	O23—Mo2—O24	105.75 (13)
O14—Mg1—O31 ^{iv}	87.53 (11)	O22—Mo2—O24	111.86 (13)
O34 ⁱ —Mg1—O31 ^{iv}	171.86 (11)	O33—Mo3—O32	108.10 (14)
O31 ⁱⁱⁱ —Mg1—O31 ^{iv}	81.22 (11)	O33—Mo3—O34	110.71 (13)
O13 ⁱ —Mg2—O24	88.40 (12)	O32—Mo3—O34	109.21 (14)
O13 ⁱ —Mg2—O22 ^v	90.10 (12)	O33—Mo3—O31	108.43 (13)
O24—Mg2—O22 ^v	175.47 (12)	O32—Mo3—O31	109.96 (13)
O13 ⁱ —Mg2—O11 ^{vi}	92.52 (11)	O34—Mo3—O31	110.40 (12)

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