Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

β-$Nd_2Mo_4O_{15}$

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Received 4 November 2010; accepted 22 November 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Mo–O) = 0.007 Å; R factor = 0.046; wR factor = 0.128; data-to-parameter ratio = 12.5.

The title compound, dineodymium(III) tetramolybdate(VI), has been prepared by a flux technique and is the second polymorph of composition $Nd_2Mo_4O_{15}$. The crystal structure is isotypic with those of $Ce_2Mo_4O_{15}$ and $Pr_2Mo_4O_{15}$. It features a three-dimensional network composed of distorted edge- and corner-sharing NdO_7 polyhedra, NdO_8 polyhedra, MoO_4 tetrahedra and MoO_6 octahedra.

Related literature

For background to molybdates with rare earth (*RE*) cations, see: Borchardt & Bierstedt (1966); Ouwerkerk *et al.* (1982). For the α-polymorph of $Nd_2Mo_4O_{15}$, see: Naruke & Yamase (2003). Structures isotypic with β-$Nd_2Mo_4O_{15}$ were reported for the Ce (Fallon & Gatehouse, 1982) and Pr (Efremov *et al.*, 1988*a*) analogues. For the crystal structures, properties and applications of other molybdates with general formula $RE_2Mo_4O_{15}$, see: *RE* = La (Dubois *et al.*, 2001); Tb (Naruke & Yamase, 2001); La, Nd, Sm (Naruke & Yamase, 2003); Ho (Efremov *et al.*, 1988*b*).

Experimental

Crystal data

$Nd_2Mo_4O_{15}$
M_r = 912.24
Triclinic, $P\overline{1}$
a = 7.4000 (6) Å
b = 7.4992 (6) Å
c = 11.7291 (9) Å
α = 88.916 (2)°
β = 83.957 (1)°
γ = 84.196 (2)°
V = 643.94 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 11.77 mm^{-1}
T = 293 K
0.15 × 0.15 × 0.05 mm

Data collection

Bruker SMART 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.271, T_{max} = 0.591
3620 measured reflections
2390 independent reflections
2268 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.05
2390 reflections
191 parameters
$\Delta\rho_{max}$ = 3.38 e Å^{-3}
$\Delta\rho_{min}$ = −2.54 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2425).

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supporting information

Acta Cryst. (2010). E**66**, i85 [https://doi.org/10.1107/S1600536810048609]

β-$Nd_2Mo_4O_{15}$

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S1. Comment

Rare-earth molybdate compounds have been intensively studied due to their diversity and excellent chemical stabilities, as well as their potential applications as laser host phosphors, or as ferroelectric and ferroelastic materials (Borchardt & Bierstedt, 1966; Ouwerkerk *et al.*, 1982). Previous studies of the family of $RE_2Mo_4O_{15}$ (*RE* is a rare earth metal cation) compounds show that they adopt different structure types, such as monoclinic $La_2Mo_4O_{15}$ with $Z = 4$ (Dubois *et al.*, 2001; Naruke & Yamase, 2003); $Tb_2Mo_4O_{15}$ (Naruke & Yamase, 2001) and $Ho_2Mo_4O_{15}$ (Efremov *et al.*, 1988*b*) with $Z = 2$, or triclinic $Nd_2Mo_4O_{15}$ (Naruke & Yamase, 2003) with $Z = 3$. In this paper, we present synthesis and crystal structure of the β-phase of compound $Nd_2Mo_4O_{15}$ which is structurally different from the first (α-) $Nd_2Mo_4O_{15}$ polymorph (Naruke & Yamase, 2003), but is isotypic with $Ce_2Mo_4O_{15}$ (Fallon & Gatehouse, 1982) and $Pr_2Mo_4O_{15}$ (Efremov *et al.*, 1988*a*) with $Z = 2$.

The structure of β-$Nd_2Mo_4O_{15}$ features a three-dimensional framework composed of distorted NdO_7, NdO_8, MoO_4 and MoO_6 polyhedra, as shown in Fig. 1. There are four crystallographically different Mo atoms in the asymmetric unit. Mo(1), Mo(2), Mo(3) atoms are surrounded by four oxygen atoms within a tetrahedral coordination, while the Mo(4) atom is surrrounded by six oxygen atoms within a considerably distorted octahedral coordination. Two adjacent $Mo(4)O_6$ octahedra are connected through edge-sharing, forming Mo_2O_{10} units. These Mo_2O_{10} units are interconnected by $Mo(1)O_4$ tetrahedra *via* corner-sharing to form an infinite Mo_4O_{14} chain parallel to [100]. The distorted environments of the two Nd atoms Nd(1) and Nd(2) are different. While Nd(1) is coordinated by seven oxygen atoms, Nd(2) is coordinated by eight oxygen atoms. The Mo_4O_{14} chains are linked perpendicularly to the chain direction into a three-dimensional framework *via* isolated $Mo(2)O_4$ and $Mo(3)O_4$ tetrahedra and by $Nd(1)O_7$ and $Nd(2)O_8$ polyhedra sharing edges and corners (Fig. 2) .

S2. Experimental

The finely ground reagents K_2CO_3, Nd_2O_3, and MoO_3 were mixed in the molar ratio K: Nd: Mo = 3: 2: 6, were placed in a Pt crucible, and heated at 573 K for 4 h. The mixture was then re-ground and heated at 1273 K for 20 h, then cooled to 673 K at a rate of 3 K h^{-1}, and finally quenched to room temperature. A few light-red crystals of the title compound with prismatic shape were obtained.

S3. Refinement

The highest peak in the difference electron density map equals to 3.38 e/Å^3 at the distance of 0.92 Å from the Nd(1) site while the deepest hole equals to -2.54 e/Å^3 at the distance of 1.22 Å from the Nd(2) site.

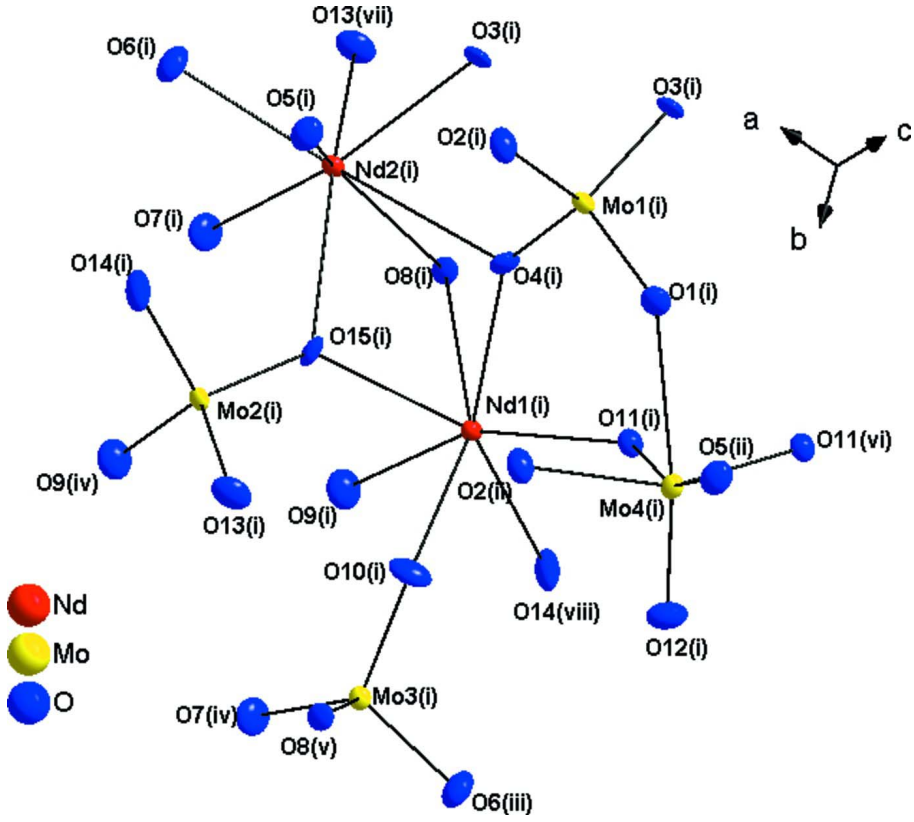

Figure 1

The expanded asymmetric unit of β-$Nd_2Mo_4O_{15}$ showing the coordination environments of the Mo and Nd atoms. [Symmetry codes: (i) *x*, *y*, *z*; (ii) 1 - *x*, 1 - *y*, 1 - *z*; (iii) -1 + *x*, 1 + *y*, *z*; (iv) 1 - *x*, 1 - *y*, -*z*; (v) *x*, 1 + *y*, *z*; (vi) -*x*, 1 - *y*, 1 - *z*; (vii) *x*, -1 + *y*, *z*; (viii) -1 + *x*, *y*, *z*.]

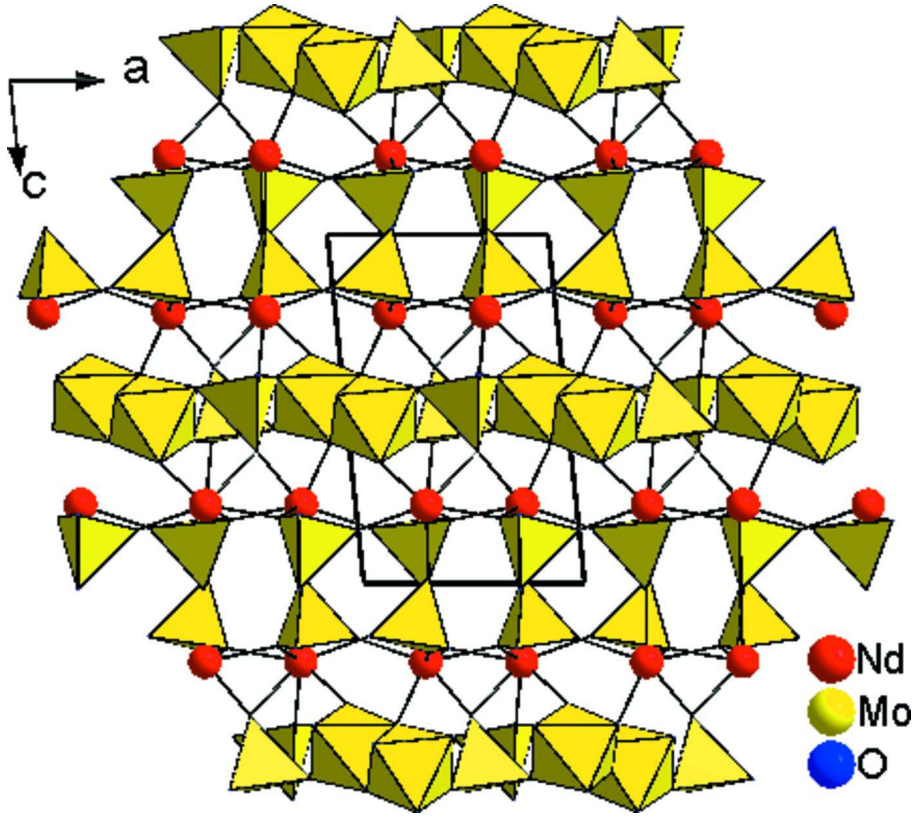

Figure 2

View of the crystal structure of β-$Nd_2Mo_4O_{15}$ along [0$\bar{1}$0]. MoO_4 and MoO6 units are given in the polyhedral representation.

dineodymium(III) tetramolybdate(VI)

Crystal data

$Nd_2Mo_4O_{15}$
M_r = 912.24
Triclinic, $P\bar{1}$
Hall symbol: -P 1
a = 7.4000 (6) Å
b = 7.4992 (6) Å
c = 11.7291 (9) Å
α = 88.916 (2)°
β = 83.957 (1)°
γ = 84.196 (2)°
V = 643.94 (9) Å^3

Z = 2
F(000) = 816
D_x = 4.705 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 487 reflections
θ = 2.1–23.0°
μ = 11.77 mm^{-1}
T = 293 K
Prism, light-red
0.15 × 0.15 × 0.05 mm

Data collection

Bruker SMART 1K CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.271, T_{max} = 0.591

3620 measured reflections
2390 independent reflections
2268 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 25.7°, θ_{min} = 1.8°
h = −9→8
k = −9→6
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.128$
$S = 1.05$
2390 reflections
191 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$w = 1/[\sigma^2(F_o^2) + (0.0986P)^2 + 6.3644P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 3.38$ e Å^{-3}
$\Delta\rho_{min} = -2.54$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0080 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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, conven tional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ 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}
Nd1	0.24478 (7)	0.41215 (6)	0.22477 (4)	0.0090 (2)
Nd2	0.67891 (6)	0.09060 (6)	0.22360 (4)	0.0081 (2)
Mo1	0.43929 (11)	0.25334 (11)	0.52879 (7)	0.0093 (3)
Mo2	0.72798 (11)	0.57014 (10)	0.12800 (7)	0.0081 (3)
Mo3	0.22775 (11)	0.92862 (10)	0.12894 (7)	0.0084 (3)
Mo4	0.09418 (11)	0.67225 (10)	0.52795 (7)	0.0090 (3)
O11	0.0842 (9)	0.4745 (9)	0.4032 (6)	0.0122 (14)
O2	0.6680 (10)	0.2917 (10)	0.5608 (6)	0.0180 (15)
O15	0.5756 (9)	0.4080 (9)	0.1806 (6)	0.0124 (14)
O8	0.3544 (9)	0.0941 (9)	0.1872 (6)	0.0116 (14)
O1	0.2954 (10)	0.4305 (9)	0.5914 (6)	0.0161 (15)
O5	0.8677 (10)	0.2270 (10)	0.3442 (7)	0.0183 (15)
O7	0.7035 (11)	0.0974 (11)	0.0170 (7)	0.0209 (17)
O12	−0.0335 (11)	0.8313 (10)	0.4610 (7)	0.0217 (16)
O4	0.4229 (10)	0.2652 (9)	0.3798 (6)	0.0153 (15)
O6	0.9983 (10)	0.0078 (11)	0.1526 (7)	0.0231 (17)
O9	0.2747 (11)	0.4070 (11)	0.0191 (7)	0.0227 (17)
O10	0.2620 (11)	0.7197 (10)	0.1964 (6)	0.0200 (16)
O13	0.6638 (11)	0.7841 (10)	0.1853 (7)	0.0207 (16)
O14	0.9451 (11)	0.4864 (12)	0.1581 (7)	0.0253 (18)
O3	0.3788 (10)	0.0521 (9)	0.5944 (6)	0.0150 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Nd1	0.0097 (3)	0.0080 (3)	0.0089 (3)	0.0000 (2)	0.0000 (2)	0.0003 (2)
Nd2	0.0096 (3)	0.0058 (3)	0.0091 (3)	−0.0022 (2)	−0.0012 (2)	−0.0003 (2)
Mo1	0.0098 (4)	0.0084 (4)	0.0101 (4)	−0.0026 (3)	−0.0019 (3)	0.0022 (3)
Mo2	0.0086 (4)	0.0075 (4)	0.0084 (4)	−0.0030 (3)	0.0001 (3)	0.0001 (3)
Mo3	0.0089 (4)	0.0080 (4)	0.0088 (4)	−0.0025 (3)	−0.0011 (3)	−0.0016 (3)
Mo4	0.0083 (4)	0.0079 (4)	0.0113 (4)	−0.0026 (3)	−0.0011 (3)	−0.0023 (3)
O11	0.013 (3)	0.013 (3)	0.011 (3)	−0.004 (3)	−0.001 (3)	0.000 (3)
O2	0.017 (4)	0.023 (4)	0.015 (4)	−0.006 (3)	0.000 (3)	0.002 (3)
O15	0.006 (3)	0.011 (3)	0.020 (4)	−0.001 (2)	0.003 (3)	−0.006 (3)
O8	0.010 (3)	0.008 (3)	0.017 (3)	−0.006 (3)	−0.002 (3)	−0.002 (3)
O1	0.019 (4)	0.009 (3)	0.019 (4)	−0.003 (3)	0.000 (3)	−0.001 (3)
O5	0.016 (4)	0.016 (4)	0.024 (4)	−0.004 (3)	−0.001 (3)	−0.004 (3)
O7	0.026 (4)	0.024 (4)	0.012 (4)	−0.001 (3)	0.000 (3)	−0.006 (3)
O12	0.023 (4)	0.016 (4)	0.027 (4)	0.003 (3)	−0.008 (3)	0.000 (3)
O4	0.015 (3)	0.009 (3)	0.019 (4)	0.005 (3)	0.001 (3)	−0.001 (3)
O6	0.012 (4)	0.030 (5)	0.026 (4)	0.002 (3)	0.001 (3)	−0.002 (3)
O9	0.026 (4)	0.027 (4)	0.016 (4)	−0.005 (3)	−0.001 (3)	−0.003 (3)
O10	0.032 (4)	0.015 (4)	0.016 (4)	−0.005 (3)	−0.013 (3)	0.004 (3)
O13	0.031 (4)	0.011 (4)	0.021 (4)	−0.006 (3)	−0.006 (3)	−0.001 (3)
O14	0.015 (4)	0.039 (5)	0.021 (4)	−0.007 (3)	0.003 (3)	0.007 (4)
O3	0.017 (3)	0.008 (3)	0.019 (4)	−0.002 (3)	0.000 (3)	0.008 (3)

Geometric parameters (Å, º)

Nd1—O11	2.326 (7)	Mo2—O15	1.798 (7)
Nd1—O10	2.339 (7)	Mo3—O6^{v}	1.737 (7)
Nd1—O9	2.400 (8)	Mo3—O7^{iv}	1.742 (8)
Nd1—O14^{i}	2.437 (8)	Mo3—O10	1.749 (7)
Nd1—O15	2.446 (6)	Mo3—O8^{vi}	1.814 (6)
Nd1—O8	2.472 (7)	Mo4—O12	1.680 (8)
Nd1—O4	2.528 (7)	Mo4—O5^{vii}	1.753 (7)
Nd1—Nd2	3.8158 (7)	Mo4—O11^{viii}	1.909 (7)
Nd2—O13^{ii}	2.366 (7)	Mo4—O2^{vii}	1.989 (7)
Nd2—O3^{iii}	2.387 (7)	Mo4—O11	2.115 (7)
Nd2—O5	2.399 (7)	Mo4—O1	2.386 (7)
Nd2—O7	2.411 (8)	Mo4—Mo4^{viii}	3.1674 (15)
Nd2—O6	2.444 (7)	O11—Mo4^{viii}	1.909 (7)
Nd2—O8	2.480 (7)	O2—Mo4^{vii}	1.989 (7)
Nd2—O15	2.484 (7)	O8—Mo3^{ii}	1.814 (6)
Nd2—O4	2.742 (7)	O5—Mo4^{vii}	1.753 (7)
Mo1—O1	1.739 (7)	O7—Mo3^{iv}	1.742 (8)
Mo1—O3	1.758 (7)	O6—Mo3^{ix}	1.737 (7)
Mo1—O4	1.764 (7)	O9—Mo2^{iv}	1.733 (8)
Mo1—O2	1.823 (7)	O13—Nd2^{vi}	2.366 (7)
Mo2—O9^{iv}	1.733 (8)	O14—Nd1^{x}	2.437 (8)

Mo2—O14	1.734 (8)	O3—Nd2^{iii}	2.387 (7)
Mo2—O13	1.753 (7)		

O11—Nd1—O10	88.8 (3)	O4—Nd2—Nd1	41.44 (15)
O11—Nd1—O9	153.3 (3)	O1—Mo1—O3	108.8 (3)
O10—Nd1—O9	83.4 (3)	O1—Mo1—O4	107.4 (3)
O11—Nd1—O14^{i}	82.8 (3)	O3—Mo1—O4	114.5 (3)
O10—Nd1—O14^{i}	82.1 (3)	O1—Mo1—O2	105.5 (3)
O9—Nd1—O14^{i}	70.9 (3)	O3—Mo1—O2	109.3 (3)
O11—Nd1—O15	125.3 (2)	O4—Mo1—O2	110.9 (3)
O10—Nd1—O15	81.3 (3)	O9^{iv}—Mo2—O14	109.3 (4)
O9—Nd1—O15	78.7 (3)	O9^{iv}—Mo2—O13	106.3 (4)
O14^{i}—Nd1—O15	146.7 (3)	O14—Mo2—O13	112.1 (4)
O11—Nd1—O8	116.2 (2)	O9^{iv}—Mo2—O15	109.1 (4)
O10—Nd1—O8	152.5 (3)	O14—Mo2—O15	107.0 (3)
O9—Nd1—O8	78.6 (3)	O13—Mo2—O15	113.0 (3)
O14^{i}—Nd1—O8	110.8 (3)	O6^{v}—Mo3—O7^{iv}	111.1 (4)
O15—Nd1—O8	75.0 (2)	O6^{v}—Mo3—O10	109.0 (4)
O11—Nd1—O4	70.6 (2)	O7^{iv}—Mo3—O10	108.4 (4)
O10—Nd1—O4	116.3 (2)	O6^{v}—Mo3—O8^{vi}	106.6 (4)
O9—Nd1—O4	135.5 (2)	O7^{iv}—Mo3—O8^{vi}	109.7 (3)
O14^{i}—Nd1—O4	146.5 (2)	O10—Mo3—O8^{vi}	112.0 (3)
O15—Nd1—O4	66.7 (2)	O12—Mo4—O5^{vii}	104.3 (4)
O8—Nd1—O4	66.3 (2)	O12—Mo4—O11^{viii}	102.4 (4)
O11—Nd1—Nd2	116.20 (17)	O5^{vii}—Mo4—O11^{viii}	95.7 (3)
O10—Nd1—Nd2	120.5 (2)	O12—Mo4—O2^{vii}	97.0 (4)
O9—Nd1—Nd2	89.6 (2)	O5^{vii}—Mo4—O2^{vii}	97.9 (3)
O14^{i}—Nd1—Nd2	148.9 (2)	O11^{viii}—Mo4—O2^{vii}	152.7 (3)
O15—Nd1—Nd2	39.66 (16)	O12—Mo4—O11	94.5 (3)
O8—Nd1—Nd2	39.68 (15)	O5^{vii}—Mo4—O11	160.8 (3)
O4—Nd1—Nd2	45.87 (15)	O11^{viii}—Mo4—O11	76.3 (3)
O13^{ii}—Nd2—O3^{iii}	73.9 (3)	O2^{vii}—Mo4—O11	83.4 (3)
O13^{ii}—Nd2—O5	129.8 (3)	O12—Mo4—O1	170.4 (3)
O3^{iii}—Nd2—O5	75.9 (3)	O5^{vii}—Mo4—O1	83.9 (3)
O13^{ii}—Nd2—O7	79.6 (3)	O11^{viii}—Mo4—O1	81.3 (3)
O3^{iii}—Nd2—O7	153.0 (3)	O2^{vii}—Mo4—O1	76.7 (3)
O5—Nd2—O7	126.9 (3)	O11—Mo4—O1	77.7 (3)
O13^{ii}—Nd2—O6	80.7 (3)	O12—Mo4—Mo4^{viii}	100.5 (3)
O3^{iii}—Nd2—O6	107.9 (3)	O5^{vii}—Mo4—Mo4^{viii}	133.6 (3)
O5—Nd2—O6	71.7 (3)	O11^{viii}—Mo4—Mo4^{viii}	40.4 (2)
O7—Nd2—O6	72.0 (3)	O2^{vii}—Mo4—Mo4^{viii}	117.3 (2)
O13^{ii}—Nd2—O8	79.2 (3)	O11—Mo4—Mo4^{viii}	35.83 (18)
O3^{iii}—Nd2—O8	91.6 (2)	O1—Mo4—Mo4^{viii}	76.52 (17)
O5—Nd2—O8	140.7 (2)	Mo4^{viii}—O11—Mo4	103.7 (3)
O7—Nd2—O8	78.5 (3)	Mo4^{viii}—O11—Nd1	122.5 (3)
O6—Nd2—O8	146.8 (3)	Mo4—O11—Nd1	133.7 (3)
O13^{ii}—Nd2—O15	147.6 (2)	Mo1—O2—Mo4^{vii}	136.8 (4)
O3^{iii}—Nd2—O15	124.5 (2)	Mo2—O15—Nd1	135.0 (4)

O5—Nd2—O15	82.5 (2)	Mo2—O15—Nd2	123.6 (3)
O7—Nd2—O15	77.3 (3)	Nd1—O15—Nd2	101.4 (2)
O6—Nd2—O15	112.6 (3)	Mo3^{ii}—O8—Nd1	126.2 (3)
O8—Nd2—O15	74.2 (2)	Mo3^{ii}—O8—Nd2	132.4 (3)
O13^{ii}—Nd2—O4	119.7 (3)	Nd1—O8—Nd2	100.8 (2)
O3^{iii}—Nd2—O4	63.0 (2)	Mo1—O1—Mo4	136.9 (4)
O5—Nd2—O4	78.3 (2)	Mo4^{vii}—O5—Nd2	152.6 (4)
O7—Nd2—O4	129.9 (2)	Mo3^{iv}—O7—Nd2	165.9 (5)
O6—Nd2—O4	150.0 (2)	Mo1—O4—Nd1	144.6 (4)
O8—Nd2—O4	63.0 (2)	Mo1—O4—Nd2	122.6 (3)
O15—Nd2—O4	62.9 (2)	Nd1—O4—Nd2	92.7 (2)
O13^{ii}—Nd2—Nd1	118.7 (2)	Mo3^{ix}—O6—Nd2	168.5 (5)
O3^{iii}—Nd2—Nd1	99.86 (17)	Mo2^{iv}—O9—Nd1	171.6 (5)
O5—Nd2—Nd1	105.23 (18)	Mo3—O10—Nd1	156.8 (4)
O7—Nd2—Nd1	88.49 (19)	Mo2—O13—Nd2^{vi}	159.0 (5)
O6—Nd2—Nd1	150.0 (2)	Mo2—O14—Nd1^{x}	169.8 (5)
O8—Nd2—Nd1	39.53 (15)	Mo1—O3—Nd2^{iii}	143.0 (4)
O15—Nd2—Nd1	38.92 (15)		

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