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[Oxybis(ethane-1,2-diyl)]bis(dimethylammonium) octamolybdate dihydrate

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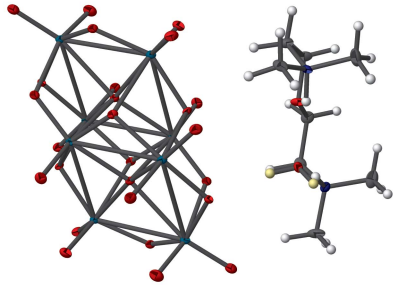
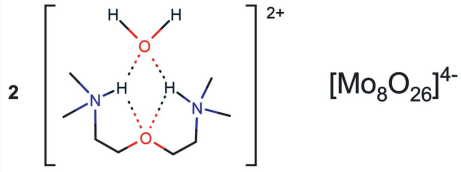
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $(C_8H_{22}N_2O)_2[Mo_8O_{26}]\cdot H_2O$, (*cis*-$H_2L)_2[\beta$-$Mo_8O_{26}]\cdot H_2O$, where L = (bis[2-*N*,*N*-dimethylamino)ethyl] ether), was synthesized from bis[2-(dimethylamino)ethyl] ether and MoO_3 under solvothermal conditions and characterized by multinuclear NMR and single-crystal X-ray diffraction techniques. The structure displays two [oxybis(ethane-1,2-diyl)]bis(dimethylammonium), or [*cis*-$H_2L]^{2+}$, cations, a central [β-$Mo_8O_{26}]^{4-}$ anionic cluster consisting of eight distorted MoO_6 octahedra, and two water molecules in their deuterated form. The central anion lies across an inversion center. The [*cis*-$H_2L]^{2+}$ cations are hydrogen bonded to the central [β-$Mo_8O_{26}]^{4-}$ cluster *via* bridging water molecules. In the crystal, O—H···O hydrogen bonds link the components into chains along [010]. Weak C—H···O hydrogen bonds link these chains into a three-dimensional network.

3D view



Chemical scheme



Structure description

Polyoxometalates (POMs) are self-assembled metal clusters finding broad application in coatings, the pulp and paper industry, catalysis, microelectronics, and medicine (Katsoulis, 1998; Chaidogiannos *et al.*, 2004; Long *et al.*, 2007; Rhule *et al.*, 1998). Generally, group 5 and 6 POMs are more common and can adopt a wide range of nuclearity (Pope, 1983; Pope & Müller, 1991). Within the context of molybdenum, seven isomers of the octamolybdate anion, $[Mo_8O_{26}]^{4-}$, have been reported (Bridgeman, 2002; Allis *et al.*, 2004). Here, we report the isolation of the title compound (**1**), which is characterized by a protonated bis(dialkyl)ammonium ether salt linked to an octamolybdate anion through hydrogen bonding (Table 1).

Compound (**1**) (Fig. 1) crystallizes as a salt containing two [bis(2-*N*,*N*-dimethylammonium)ethyl ether]$^{2+}$ cations and a [β-$Mo_8O_{26}]^{4-}$ anion hydrogen bonded through a single water molecule (deuterated)of hydration per $[H_2L]^{2+}$. The anion lies across a

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Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O14	1.00	2.26	2.737 (6)	108
N1—H1···O15	1.00	1.97	2.916 (7)	157
N2—H2···O14	1.00	2.46	2.808 (7)	100
N2—H2···O15	1.00	1.87	2.863 (6)	170
O15—D15*A*···O4	0.87	1.99	2.854 (5)	170
O15—D15*B*···O4^{ii}	0.87	1.99	2.863 (6)	177
C1—H1*A*···O5^{iii}	0.98	2.56	3.440 (6)	149
C1—H1*B*···O9	0.98	2.36	3.189 (7)	142
C1—H1*C*···O11^{ii}	0.98	2.39	3.355 (8)	168
C2—H2*A*···O3^{iv}	0.98	2.44	3.343 (7)	153
C2—H2*B*···O10^{ii}	0.98	2.34	3.300 (8)	167
C3—H3*A*···O13^{i}	0.99	2.52	3.407 (7)	150
C3—H3*B*···O11^{iii}	0.99	2.42	3.266 (7)	143
C4—H4*A*···O6^{v}	0.99	2.50	3.472 (8)	167
C6—H6*B*···O6^{i}	0.99	2.44	3.360 (8)	154
C7—H7*A*···O12^{v}	0.98	2.49	3.316 (8)	142
C7—H7*B*···O8^{ii}	0.98	2.26	3.227 (8)	170
C8—H8*A*···O9^{vi}	0.98	2.48	3.402 (7)	157
C8—H8*B*···O9^{ii}	0.98	2.48	3.461 (8)	176
C8—H8*C*···O11	0.98	2.40	3.155 (7)	134
C8—H8*C*···O7^{i}	0.98	2.52	3.270 (8)	133

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

center of inversion. The protonated amino arms of the ether groups are arranged in a *cis* orientation and create a hydrogen-bonding pocket for D_2O coordination between both N—H protons of a given ether group and a μ_2-O-atom of the $[Mo_8O_{26}]^{4-}$ anion. Overall, hydrogen-bond lengths range from 1.87–2.46 Å, with the close proximity of the ammonium protons to the oxygen atom of the ether group facilitating the longer (2.26–2.46 Å) hydrogen bonding. In the crystal, O—H···O hydrogen bonds link the components into chains (Fig. 2) along [010]. Furthermore, weak C—H···O hydrogen bonds link these chains into a three-dimensional network (Fig. 3). Although **1** was crystallized from D_2O, only the solvent molecules have been modeled with deuterium atoms; however, deuterium exchange with N—H protons is likely and supported by ^{1}H-NMR experiments (*see below*).

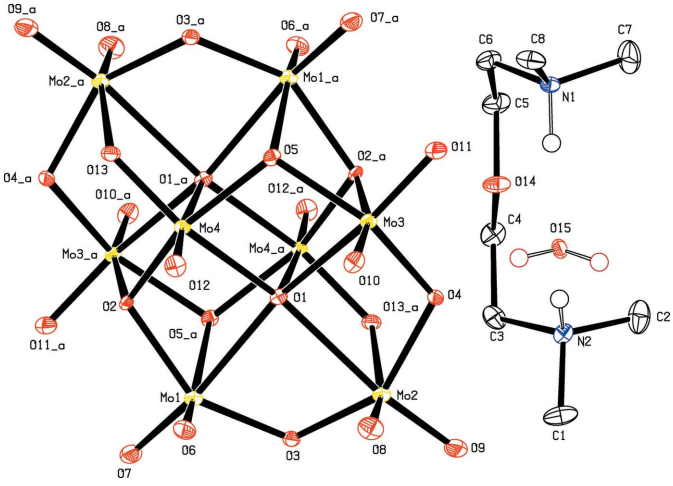

Figure 1
The molecular entities in the title compound with ellipsoids drawn at the 50% probability level. Only the symmetry-unique cation and solvent water molecules are shown. H atoms bonded to C atoms are omitted for the sake of clarity. Atoms labeled with the suffix 'a' are related by the symmetry operator $(-x+1, -y+1, -z+1)$.

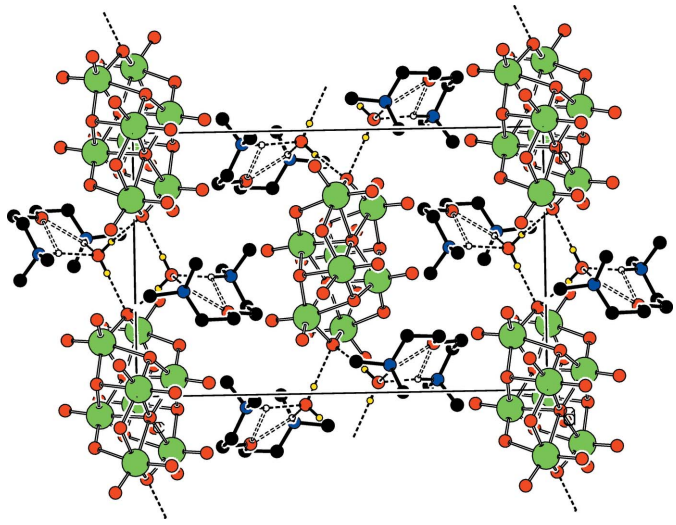

Figure 2
Part of the crystal structure with N—H···O and O—H···O hydrogen bonds shown as dashed lines viewed along the [100] direction of the unit cell.

In **1**, the octamolybate anion consists of six-coordinate Mo atoms in a distorted octahedral shape bound to oxygen through combinations of terminal, μ_2, μ_3, or μ_5 modes. Relevant bond metrics are reported in Table 2. Most notable, the μ_2-O13 bond lengths are closer to that of terminal [Mo4—O13: 1.759 (4) Å] and higher-coordination environment oxides [Mo2—O13: 2.270 (4) Å], respectively. The atypical bridging Mo—O bond lengths are a hallmark of the β-isomer and have been described as 'pseudoterminal' (Bridgeman, 2002). Taken together, these data are consistent with the structural trends present in reported $[\beta\text{-}Mo_8O_{26}]^{4-}$ motifs (Bridgeman, 2002).

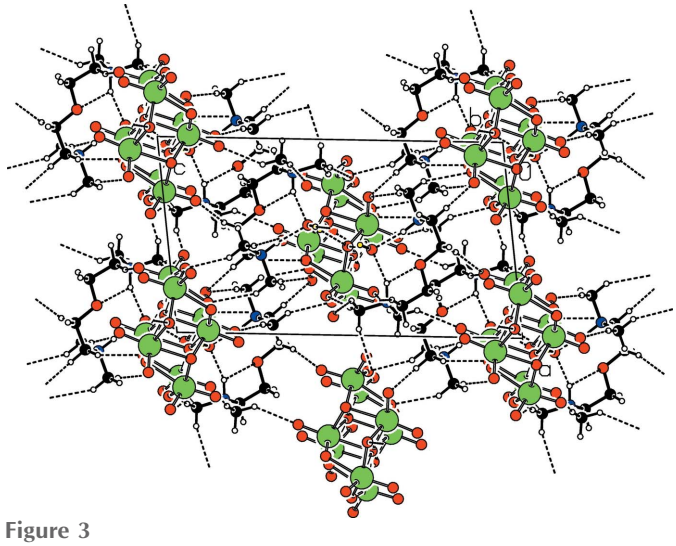

Figure 3
Part of the crystal structure with N—H···O, O—H···O and weak C—H···O hydrogen bonds shown as dashed lines viewed along the [010] direction of the unit cell.

Table 2
Selected bond lengths (Å).

Mo1—O1	2.340 (4)	Mo3—O1	2.277 (4)
Mo1—O2	2.015 (4)	Mo3—O2^{i}	2.334 (4)
Mo1—O3	1.893 (4)	Mo3—O4	1.933 (4)
Mo1—O5^{i}	2.357 (4)	Mo3—O5	1.984 (4)
Mo1—O6	1.696 (4)	Mo3—O10	1.705 (4)
Mo1—O7	1.700 (4)	Mo3—O11	1.699 (4)
Mo2—O1	2.450 (4)	Mo4—O1	2.172 (4)
Mo2—O3	1.901 (4)	Mo4—O1^{i}	2.368 (4)
Mo2—O4	1.968 (4)	Mo4—O2	1.942 (4)
Mo2—O8	1.699 (4)	Mo4—O5	1.963 (4)
Mo2—O9	1.696 (4)	Mo4—O12	1.692 (4)
Mo2—O13^{i}	2.270 (4)	Mo4—O13	1.759 (4)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

The octamolybdate anion, $[Mo_8O_{26}]^{4-}$, is common. A search of the Cambridge Structural Database (CSD version 5.40 up to May 2019; Groom *et al.*, 2016) listed 278 deposited structures. However, the *cis*-[bis(2-*N*,*N*-dimethylammonium)ethyl ether]$^{2+}$ cation reported here is the first crystallographic example in the literature.

Synthesis and crystallization

Synthesis of the title complex (**1**): All reagents were purchased from Sigma–Aldrich and used without further purification. MoO_3 (5.0 g, 34.7 mmol) and bis[2-(*N*,*N*-dimethylamino)ethyl] ether (13.1 ml, 69.4 mmol) were loaded into a 250 ml round-bottom flask equipped with a magnetic stir bar and diluted with 100 ml of H_2O. The resulting mint-green mixture was heated to 373 K. After 20 minutes the reaction presented as a colorless solution and was cooled to room temperature. The solution was transferred to a 500 ml beaker and diluted with 2-propanol (300 ml), resulting in the formation of a fine colorless precipitate. The solid was allowed to settle, the mother liquor decanted off, and the white solid collected and dried under reduced pressure at 333 K. ^{1}H NMR (400 MHz, D_2O) in p.p.m.: δ = 4.79 (*s*, 6H), 3.92 (*t*, 5.34 Hz, 4H), 3.42 (*t*, 5.34 Hz, 4H), 2.96 (*s*, 12H). ^{13}C NMR (100 MHz, D_2O) in p.p.m.: δ = 64.40, 56.78, 43.26.

The title complex (**1**) precipitated as colorless crystals from a D_2O solution stored inside of an NMR tube for three days.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

Table 3
Experimental details.

Crystal data	
Chemical formula	$(C_8H_{22}N_2O)_2[Mo_8O_{26}]\cdot 2D_2O$
M_r	1548.13
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	10.139 (3), 11.350 (3), 17.815 (5)
β (°)	96.773 (3)
V (Å^3)	2035.7 (9)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.48
Crystal size (mm)	0.28 × 0.23 × 0.02
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)
T_{min}, T_{max}	0.581, 0.646
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8296, 3652, 2992
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.603
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.074, 1.08
No. of reflections	3652
No. of parameters	269
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.75, −0.88

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2019). **4**, x191536 [https://doi.org/10.1107/S2414314619015360]

[Oxybis(ethane-1,2-diyl)]bis(dimethylammonium) octamolybdate dihydrate

David M. Ermert, Milan Gembicky and Arnold L. Rheingold

[Oxybis(ethane-1,2-diyl)]bis(dimethylammonium) octamolybdate dihydrate

Crystal data

$(C_8H_{22}N_2O)_2[Mo_8O_{26}]\cdot 2D_2O$
M_r = 1548.13
Monoclinic, $P2_1/n$
a = 10.139 (3) Å
b = 11.350 (3) Å
c = 17.815 (5) Å
β = 96.773 (3)°
V = 2035.7 (9) Å^3
Z = 2
$F(000)$ = 1496
D_x = 2.526 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4105 reflections
θ = 2.2–25.4°
μ = 2.48 mm^{-1}
T = 100 K
Block, colourless
0.28 × 0.23 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Detector resolution: 8.258 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
T_{min} = 0.581, T_{max} = 0.646
8296 measured reflections
3652 independent reflections
2992 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.4°, θ_{min} = 2.1°
h = −10→12
k = −9→13
l = −21→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.074
S = 1.08
3652 reflections
269 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0204P)^2 + 11.0569P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.88 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo4	0.51222 (5)	0.56859 (4)	0.59121 (2)	0.00664 (12)
Mo3	0.57308 (5)	0.29183 (4)	0.58295 (2)	0.00758 (12)
Mo2	0.27674 (5)	0.24855 (5)	0.49345 (3)	0.00926 (13)
Mo1	0.21926 (5)	0.52804 (5)	0.50414 (3)	0.00963 (13)
O5	0.6544 (4)	0.4504 (3)	0.59792 (19)	0.0083 (8)
O4	0.4575 (4)	0.1866 (3)	0.52022 (19)	0.0082 (8)
O1	0.4160 (4)	0.4202 (3)	0.53033 (19)	0.0081 (8)
O2	0.3639 (4)	0.6471 (3)	0.53276 (19)	0.0078 (8)
O3	0.1739 (4)	0.3814 (3)	0.45750 (19)	0.0090 (8)
O15	0.5610 (4)	0.0398 (4)	0.4110 (2)	0.0102 (9)
D15A	0.538031	0.090272	0.443795	0.015*
D15B	0.552187	−0.028589	0.431848	0.015*
O13	0.6163 (4)	0.6926 (3)	0.6051 (2)	0.0102 (8)
O7	0.1035 (4)	0.6188 (4)	0.4579 (2)	0.0138 (9)
O14	0.6350 (4)	0.1851 (4)	0.2749 (2)	0.0138 (9)
O11	0.7115 (4)	0.2063 (4)	0.5948 (2)	0.0127 (9)
O9	0.2025 (4)	0.1352 (4)	0.4428 (2)	0.0161 (9)
O10	0.5105 (4)	0.2820 (4)	0.6675 (2)	0.0116 (9)
O8	0.2277 (4)	0.2344 (4)	0.5810 (2)	0.0162 (9)
O12	0.4551 (4)	0.5481 (4)	0.6757 (2)	0.0117 (9)
O6	0.1744 (4)	0.5114 (4)	0.5923 (2)	0.0148 (9)
N1	0.8289 (5)	0.1047 (4)	0.3826 (2)	0.0108 (10)
H1	0.731353	0.088198	0.377628	0.013*
N2	0.4038 (5)	0.0472 (4)	0.2666 (3)	0.0120 (11)
H2	0.465967	0.050370	0.314483	0.014*
C4	0.5429 (6)	0.1940 (6)	0.2073 (3)	0.0163 (14)
H4A	0.567447	0.138138	0.168577	0.020*
H4B	0.543593	0.274796	0.186460	0.020*
C6	0.8473 (6)	0.2142 (6)	0.3394 (3)	0.0159 (13)
H6A	0.942746	0.224333	0.334054	0.019*
H6B	0.818449	0.282736	0.367715	0.019*
C2	0.4473 (7)	−0.0508 (6)	0.2199 (3)	0.0221 (15)
H2A	0.385925	−0.057648	0.173291	0.033*
H2B	0.447599	−0.124628	0.248378	0.033*
H2C	0.536992	−0.034577	0.207200	0.033*
C5	0.7685 (6)	0.2105 (6)	0.2617 (3)	0.0185 (14)
H5A	0.773187	0.287257	0.235703	0.022*
H5B	0.802965	0.148390	0.230206	0.022*
C8	0.8742 (6)	0.1196 (6)	0.4648 (3)	0.0172 (14)
H8A	0.969481	0.137400	0.471856	0.026*
H8B	0.857809	0.046710	0.491716	0.026*
H8C	0.825183	0.184516	0.484942	0.026*
C3	0.4072 (6)	0.1646 (6)	0.2288 (3)	0.0153 (13)
H3A	0.380344	0.226151	0.263347	0.018*
H3B	0.342107	0.164899	0.182807	0.018*

C7	0.8937 (7)	0.0006 (6)	0.3518 (4)	0.0244 (16)
H7A	0.866396	−0.005198	0.297311	0.037*
H7B	0.866965	−0.070964	0.376808	0.037*
H7C	0.990385	0.009487	0.361060	0.037*
C1	0.2683 (6)	0.0247 (6)	0.2869 (3)	0.0209 (15)
H1A	0.205718	0.018943	0.240680	0.031*
H1B	0.241766	0.089540	0.318142	0.031*
H1C	0.267874	−0.049343	0.315157	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo4	0.0060 (3)	0.0080 (3)	0.0061 (2)	−0.0005 (2)	0.00142 (17)	−0.00017 (18)
Mo3	0.0070 (3)	0.0078 (3)	0.0080 (2)	−0.0009 (2)	0.00073 (18)	0.00107 (19)
Mo2	0.0070 (3)	0.0099 (3)	0.0112 (2)	−0.0009 (2)	0.00251 (18)	0.0000 (2)
Mo1	0.0070 (3)	0.0103 (3)	0.0118 (2)	−0.0009 (2)	0.00221 (19)	−0.0002 (2)
O5	0.009 (2)	0.008 (2)	0.0078 (17)	−0.0009 (16)	0.0008 (15)	0.0025 (15)
O4	0.007 (2)	0.008 (2)	0.0096 (18)	0.0003 (16)	0.0029 (15)	−0.0001 (15)
O1	0.008 (2)	0.009 (2)	0.0073 (17)	−0.0017 (16)	0.0010 (15)	−0.0002 (15)
O2	0.007 (2)	0.005 (2)	0.0108 (18)	−0.0014 (16)	0.0003 (15)	0.0010 (15)
O3	0.007 (2)	0.009 (2)	0.0107 (18)	0.0000 (16)	0.0007 (15)	0.0011 (16)
O15	0.011 (2)	0.008 (2)	0.0122 (19)	0.0018 (18)	0.0033 (16)	−0.0002 (16)
O13	0.007 (2)	0.012 (2)	0.0113 (18)	−0.0006 (17)	0.0012 (15)	−0.0023 (16)
O7	0.011 (2)	0.013 (2)	0.018 (2)	−0.0020 (18)	0.0013 (16)	0.0032 (18)
O14	0.009 (2)	0.021 (3)	0.0116 (19)	−0.0033 (18)	0.0017 (16)	0.0065 (17)
O11	0.009 (2)	0.014 (2)	0.0151 (19)	−0.0011 (17)	−0.0002 (16)	−0.0001 (17)
O9	0.010 (2)	0.018 (3)	0.021 (2)	−0.0008 (18)	0.0049 (17)	−0.0042 (18)
O10	0.015 (2)	0.012 (2)	0.0082 (18)	−0.0027 (18)	0.0037 (16)	0.0010 (16)
O8	0.016 (2)	0.020 (3)	0.014 (2)	−0.0002 (19)	0.0061 (17)	0.0020 (18)
O12	0.015 (2)	0.013 (2)	0.0075 (18)	0.0002 (18)	0.0039 (16)	0.0027 (16)
O6	0.011 (2)	0.016 (2)	0.018 (2)	0.0006 (18)	0.0059 (16)	−0.0006 (18)
N1	0.008 (3)	0.012 (3)	0.013 (2)	−0.001 (2)	0.0044 (19)	0.000 (2)
N2	0.013 (3)	0.011 (3)	0.012 (2)	−0.002 (2)	0.0012 (19)	−0.001 (2)
C4	0.019 (3)	0.017 (4)	0.013 (3)	−0.002 (3)	0.001 (2)	0.003 (3)
C6	0.013 (3)	0.017 (4)	0.018 (3)	−0.004 (3)	0.003 (2)	0.002 (3)
C2	0.034 (4)	0.014 (4)	0.017 (3)	0.002 (3)	−0.002 (3)	0.001 (3)
C5	0.016 (3)	0.020 (4)	0.020 (3)	−0.005 (3)	0.004 (3)	0.008 (3)
C8	0.010 (3)	0.028 (4)	0.014 (3)	0.000 (3)	0.001 (2)	−0.002 (3)
C3	0.015 (3)	0.017 (3)	0.013 (3)	0.005 (3)	−0.002 (2)	0.003 (2)
C7	0.034 (4)	0.019 (4)	0.022 (3)	0.003 (3)	0.011 (3)	−0.006 (3)
C1	0.014 (3)	0.033 (4)	0.016 (3)	−0.009 (3)	0.003 (3)	0.004 (3)

Geometric parameters (Å, º)

Mo1—O1	2.340 (4)	N1—H1	1.0000
Mo1—O2	2.015 (4)	N1—C6	1.485 (7)
Mo1—O3	1.893 (4)	N1—C8	1.493 (7)
Mo1—O5^{i}	2.357 (4)	N1—C7	1.487 (8)

Mo1—O6	1.696 (4)	N2—H2	1.0000
Mo1—O7	1.700 (4)	N2—C2	1.486 (8)
Mo2—O1	2.450 (4)	N2—C3	1.494 (8)
Mo2—O3	1.901 (4)	N2—C1	1.484 (7)
Mo2—O4	1.968 (4)	C4—H4A	0.9900
Mo2—O8	1.699 (4)	C4—H4B	0.9900
Mo2—O9	1.696 (4)	C4—C3	1.509 (8)
Mo2—O13^{i}	2.270 (4)	C6—H6A	0.9900
Mo3—O1	2.277 (4)	C6—H6B	0.9900
Mo3—O2^{i}	2.334 (4)	C6—C5	1.516 (7)
Mo3—O4	1.933 (4)	C2—H2A	0.9800
Mo3—O5	1.984 (4)	C2—H2B	0.9800
Mo3—O10	1.705 (4)	C2—H2C	0.9800
Mo3—O11	1.699 (4)	C5—H5A	0.9900
Mo4—O1	2.172 (4)	C5—H5B	0.9900
Mo4—O1^{i}	2.368 (4)	C8—H8A	0.9800
Mo4—O2	1.942 (4)	C8—H8B	0.9800
Mo4—O5	1.963 (4)	C8—H8C	0.9800
Mo4—O12	1.692 (4)	C3—H3A	0.9900
Mo4—O13	1.759 (4)	C3—H3B	0.9900
Mo4—Mo3	3.2081 (11)	C7—H7A	0.9800
Mo4—Mo1	3.2177 (9)	C7—H7B	0.9800
O15—D15A	0.8701	C7—H7C	0.9800
O15—D15B	0.8698	C1—H1A	0.9800
O14—C4	1.437 (6)	C1—H1B	0.9800
O14—C5	1.430 (7)	C1—H1C	0.9800

Mo3—Mo4—Mo1	90.539 (19)	O6—Mo1—O3	103.34 (18)
O5—Mo4—Mo3	35.86 (11)	O6—Mo1—O7	105.74 (19)
O5—Mo4—Mo1	124.14 (11)	Mo4—O5—Mo3	108.74 (17)
O5—Mo4—O1	77.51 (15)	Mo4—O5—Mo1^{i}	110.13 (16)
O5—Mo4—O1^{i}	77.85 (14)	Mo3—O5—Mo1^{i}	104.21 (16)
O1—Mo4—Mo3	45.18 (10)	Mo3—O4—Mo2	113.77 (19)
O1^{i}—Mo4—Mo3	85.93 (9)	Mo4—O1—Mo4^{i}	104.28 (15)
O1—Mo4—Mo1	46.64 (10)	Mo4—O1—Mo3	92.26 (13)
O1^{i}—Mo4—Mo1	86.14 (9)	Mo4—O1—Mo2	164.06 (18)
O1—Mo4—O1^{i}	75.72 (15)	Mo4^{i}—O1—Mo2	91.55 (12)
O2—Mo4—Mo3	124.45 (11)	Mo4—O1—Mo1	90.93 (14)
O2—Mo4—Mo1	36.36 (11)	Mo3—O1—Mo4^{i}	97.77 (14)
O2—Mo4—O5	149.67 (15)	Mo3—O1—Mo2	87.39 (13)
O2—Mo4—O1^{i}	77.70 (14)	Mo3—O1—Mo1	162.78 (18)
O2—Mo4—O1	79.29 (15)	Mo1—O1—Mo4^{i}	97.84 (13)
O13—Mo4—Mo3	132.42 (13)	Mo1—O1—Mo2	84.95 (12)
O13—Mo4—Mo1	133.35 (12)	Mo4—O2—Mo3^{i}	109.56 (17)
O13—Mo4—O5	96.57 (17)	Mo4—O2—Mo1	108.81 (18)
O13—Mo4—O1	156.57 (16)	Mo1—O2—Mo3^{i}	104.03 (14)
O13—Mo4—O1^{i}	80.88 (15)	Mo1—O3—Mo2	117.03 (18)
O13—Mo4—O2	97.00 (16)	D15A—O15—D15B	104.5

O12—Mo4—Mo3	89.67 (14)	Mo4—O13—Mo2^{i}	117.40 (18)
O12—Mo4—Mo1	90.91 (13)	C5—O14—C4	112.4 (4)
O12—Mo4—O5	100.26 (17)	C6—N1—H1	107.1
O12—Mo4—O1	99.04 (17)	C6—N1—C8	111.7 (5)
O12—Mo4—O1^{i}	174.68 (17)	C6—N1—C7	112.7 (5)
O12—Mo4—O2	102.45 (17)	C8—N1—H1	107.1
O12—Mo4—O13	104.33 (18)	C7—N1—H1	107.1
O5—Mo3—Mo4	35.40 (11)	C7—N1—C8	110.7 (5)
O5—Mo3—O1	74.60 (14)	C2—N2—H2	107.8
O5—Mo3—O2^{i}	72.35 (14)	C2—N2—C3	113.0 (5)
O4—Mo3—Mo4	121.63 (11)	C3—N2—H2	107.8
O4—Mo3—O5	148.38 (15)	C1—N2—H2	107.8
O4—Mo3—O1	79.08 (14)	C1—N2—C2	110.6 (5)
O4—Mo3—O2^{i}	83.29 (14)	C1—N2—C3	109.5 (5)
O1—Mo3—Mo4	42.56 (9)	O14—C4—H4A	110.3
O1—Mo3—O2^{i}	72.39 (13)	O14—C4—H4B	110.3
O2^{i}—Mo3—Mo4	79.86 (9)	O14—C4—C3	107.0 (4)
O11—Mo3—Mo4	135.57 (14)	H4A—C4—H4B	108.6
O11—Mo3—O5	100.21 (17)	C3—C4—H4A	110.3
O11—Mo3—O4	98.89 (17)	C3—C4—H4B	110.3
O11—Mo3—O1	160.74 (16)	N1—C6—H6A	109.3
O11—Mo3—O2^{i}	88.35 (16)	N1—C6—H6B	109.3
O11—Mo3—O10	103.94 (18)	N1—C6—C5	111.4 (5)
O10—Mo3—Mo4	85.97 (14)	H6A—C6—H6B	108.0
O10—Mo3—O5	97.61 (17)	C5—C6—H6A	109.3
O10—Mo3—O4	101.94 (17)	C5—C6—H6B	109.3
O10—Mo3—O1	95.18 (16)	N2—C2—H2A	109.5
O10—Mo3—O2^{i}	165.54 (17)	N2—C2—H2B	109.5
O4—Mo2—O1	74.23 (14)	N2—C2—H2C	109.5
O4—Mo2—O13^{i}	77.07 (14)	H2A—C2—H2B	109.5
O3—Mo2—O4	145.37 (16)	H2A—C2—H2C	109.5
O3—Mo2—O1	74.65 (14)	H2B—C2—H2C	109.5
O3—Mo2—O13^{i}	78.41 (15)	O14—C5—C6	105.2 (5)
O13^{i}—Mo2—O1	70.16 (13)	O14—C5—H5A	110.7
O9—Mo2—O4	101.54 (17)	O14—C5—H5B	110.7
O9—Mo2—O1	162.10 (16)	C6—C5—H5A	110.7
O9—Mo2—O3	103.37 (18)	C6—C5—H5B	110.7
O9—Mo2—O13^{i}	91.97 (17)	H5A—C5—H5B	108.8
O9—Mo2—O8	104.9 (2)	N1—C8—H8A	109.5
O8—Mo2—O4	96.25 (17)	N1—C8—H8B	109.5
O8—Mo2—O1	92.90 (17)	N1—C8—H8C	109.5
O8—Mo2—O3	100.13 (18)	H8A—C8—H8B	109.5
O8—Mo2—O13^{i}	162.85 (17)	H8A—C8—H8C	109.5
O5^{i}—Mo1—Mo4	78.60 (9)	H8B—C8—H8C	109.5
O1—Mo1—Mo4	42.44 (9)	N2—C3—C4	112.4 (5)
O1—Mo1—O5^{i}	71.35 (13)	N2—C3—H3A	109.1
O2—Mo1—Mo4	34.83 (10)	N2—C3—H3B	109.1
O2—Mo1—O5^{i}	71.34 (14)	C4—C3—H3A	109.1

O2—Mo1—O1	73.89 (14)	C4—C3—H3B	109.1
O3—Mo1—Mo4	120.00 (11)	H3A—C3—H3B	107.9
O3—Mo1—O5^{i}	83.03 (15)	N1—C7—H7A	109.5
O3—Mo1—O1	77.57 (14)	N1—C7—H7B	109.5
O3—Mo1—O2	146.37 (16)	N1—C7—H7C	109.5
O7—Mo1—Mo4	133.80 (14)	H7A—C7—H7B	109.5
O7—Mo1—O5^{i}	88.28 (16)	H7A—C7—H7C	109.5
O7—Mo1—O1	159.59 (16)	H7B—C7—H7C	109.5
O7—Mo1—O2	99.00 (17)	N2—C1—H1A	109.5
O7—Mo1—O3	101.67 (17)	N2—C1—H1B	109.5
O6—Mo1—Mo4	84.40 (13)	N2—C1—H1C	109.5
O6—Mo1—O5^{i}	162.77 (16)	H1A—C1—H1B	109.5
O6—Mo1—O1	94.14 (16)	H1A—C1—H1C	109.5
O6—Mo1—O2	96.14 (17)	H1B—C1—H1C	109.5

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O14	1.00	2.26	2.737 (6)	108
N1—H1···O15	1.00	1.97	2.916 (7)	157
N2—H2···O14	1.00	2.46	2.808 (7)	100
N2—H2···O15	1.00	1.87	2.863 (6)	170
O15—D15*A*···O4	0.87	1.99	2.854 (5)	170
O15—D15*B*···O4^{ii}	0.87	1.99	2.863 (6)	177
C1—H1*A*···O5^{iii}	0.98	2.56	3.440 (6)	149
C1—H1*B*···O9	0.98	2.36	3.189 (7)	142
C1—H1*C*···O11^{ii}	0.98	2.39	3.355 (8)	168
C2—H2*A*···O3^{iv}	0.98	2.44	3.343 (7)	153
C2—H2*B*···O10^{ii}	0.98	2.34	3.300 (8)	167
C3—H3*A*···O13^{i}	0.99	2.52	3.407 (7)	150
C3—H3*B*···O11^{iii}	0.99	2.42	3.266 (7)	143
C4—H4*A*···O6^{v}	0.99	2.50	3.472 (8)	167
C6—H6*B*···O6^{i}	0.99	2.44	3.360 (8)	154
C7—H7*A*···O12^{v}	0.98	2.49	3.316 (8)	142
C7—H7*B*···O8^{ii}	0.98	2.26	3.227 (8)	170
C8—H8*A*···O9^{vi}	0.98	2.48	3.402 (7)	157
C8—H8*B*···O9^{ii}	0.98	2.48	3.461 (8)	176
C8—H8*C*···O11	0.98	2.40	3.155 (7)	134
C8—H8*C*···O7^{i}	0.98	2.52	3.270 (8)	133

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) −*x*+1/2, *y*−1/2, −*z*+1/2; (v) *x*+1/2, −*y*+1/2, *z*−1/2; (vi) *x*+1, *y*, *z*.

Selected bond lengths (Å)

Mo-O	(Å)	Mo-O	(Å)
Mo1-O1	2.340 (4)	Mo3-O1	2.277 (4)
Mo1-O2	2.015 (4)	Mo3-O2#1	2.334 (4)

Mo1-O3	1.893 (4)	Mo3-O4	1.933 (4)
Mo1-O5#1	2.357 (4)	Mo3-O5	1.984 (4)
Mo1-O6	1.696 (4)	Mo3-O10	1.705 (4)
Mo1-O7	1.700 (4)	Mo3-O11	1.699 (4)
Mo2-O1	2.450 (4)	Mo4-O1	2.172 (4)
Mo2-O3	1.901 (4)	Mo4-O1#1	2.368 (4)
Mo2-O4	1.968 (4)	Mo4-O2	1.942 (4)
Mo2-O8	1.699 (4)	Mo4-O5	1.963 (4)
Mo2-O9	1.696 (4)	Mo4-O12	1.692 (4)
Mo2-O13#1	2.270 (4)	Mo4-O13	1.759 (4)

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1