

## Di- $\mu$ -methoxy-bis[aquadimethoxo-nitrosylmolybdenum(II)]

Jolanta Bucher, Olivier Blacque\* and Heinz Berke

Anorganisch-Chemisches Institut der Universität Zürich, Winterthurerstrasse 190, CH-8057 Zürich, Switzerland

Correspondence e-mail: oblacque@aci.uzh.ch

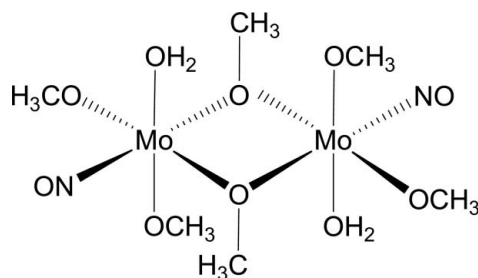
Received 24 October 2007; accepted 5 November 2007

Key indicators: single-crystal X-ray study;  $T = 183$  K; mean  $\sigma(\text{O}-\text{C}) = 0.006$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.128; data-to-parameter ratio = 18.7.

The title complex,  $[\text{Mo}_2(\text{CH}_3\text{O})_6(\text{NO})_2(\text{H}_2\text{O})_2]$ , is a pseudo-centrosymmetric dimer. The  $\text{Mo}^{\text{II}}$  centres are bridged by two methoxy groups and adopt distorted octahedral geometries. The crystal structure exhibits two intramolecular  $\text{O}-\text{H}\cdots\text{O}$  contacts ( $\text{O}\cdots\text{O} < 3.0$  Å) between coordinated water molecules and methoxy groups. One H atom of each water molecule is disordered equally over two sites.

### Related literature

For related di- $\mu$ -methoxy-bis(nitrosylmolybdenum) complexes, see: Hayton *et al.* (2002). For related di- $\mu$ -methoxy-bis(dimethoxomolybdenum) complexes, see: Kessler *et al.* (1993); Clegg *et al.* (1996); Bardina *et al.* (2006). For related di- $\mu$ -methoxy-bis(methoxomolybdenum) complexes, see: Hsieh & Zubieta (1987); Chilou *et al.* (1989). For reference bond lengths, see: Orpen *et al.* (1989).



### Experimental

#### Crystal data

$[\text{Mo}_2(\text{CH}_3\text{O})_6(\text{NO})_2(\text{H}_2\text{O})_2]$   
 $M_r = 474.14$

Monoclinic,  $P2_1/c$   
 $a = 8.7567$  (13) Å  
 $b = 15.6682$  (17) Å  
 $c = 12.5488$  (18) Å  
 $\beta = 106.750$  (16)°

$V = 1648.7$  (4) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 1.56$  mm<sup>-1</sup>  
 $T = 183$  (2) K  
 $0.5 \times 0.45 \times 0.43$  mm

#### Data collection

Stoe IPDS diffractometer  
Absorption correction: numerical  
(Coppens *et al.*, 1965)  
 $T_{\text{min}} = 0.522$ ,  $T_{\text{max}} = 0.614$

19216 measured reflections  
3874 independent reflections  
3240 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.085$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.128$   
 $S = 1.11$   
3874 reflections  
207 parameters  
4 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.75$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.21$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

Mo1—N1	1.754 (4)	Mo2—N2	1.760 (4)
Mo1—O3	1.900 (3)	Mo2—O6	2.049 (3)
Mo1—O4	1.919 (3)	Mo2—O7	2.170 (3)
Mo1—O5	2.269 (4)	Mo2—O8	1.939 (3)
Mo1—O6	2.170 (3)	Mo2—O9	1.891 (3)
Mo1—O7	2.046 (3)	Mo2—O10	2.274 (4)
N1—Mo1—O3	97.48 (15)	N2—Mo2—O6	97.39 (14)
N1—Mo1—O4	98.96 (15)	O9—Mo2—O6	155.87 (13)
O3—Mo1—O4	99.75 (14)	O8—Mo2—O6	93.05 (12)
N1—Mo1—O5	91.86 (16)	N2—Mo2—O7	169.55 (14)
O3—Mo1—O5	84.29 (15)	O9—Mo2—O7	88.62 (12)
O4—Mo1—O5	167.81 (13)	O8—Mo2—O7	85.31 (12)
O7—Mo1—O5	79.93 (13)	O6—Mo2—O7	72.85 (11)
O6—Mo1—O5	82.13 (13)	N2—Mo2—O8	99.19 (15)
N1—Mo1—O6	170.28 (14)	O9—Mo2—O8	100.86 (14)
O3—Mo1—O6	89.56 (12)	N2—Mo2—O9	99.69 (15)
O4—Mo1—O6	86.38 (12)	N2—Mo2—O10	90.14 (15)
O7—Mo1—O6	72.90 (11)	O9—Mo2—O10	84.10 (14)
N1—Mo1—O7	98.60 (14)	O8—Mo2—O10	168.46 (13)
O3—Mo1—O7	157.76 (13)	O6—Mo2—O10	78.93 (12)
O4—Mo1—O7	92.82 (12)	O7—Mo2—O10	84.40 (13)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O5—H5A <sup>i</sup> ⋯O8	0.89 (2)	2.24 (6)	2.991 (5)	142 (8)
O5—H5B <sup>i</sup> ⋯O2 <sup>ii</sup>	0.87 (2)	2.31 (6)	3.156 (5)	165 (20)
O5—H5C <sup>i</sup> ⋯O1 <sup>ii</sup>	0.87 (2)	2.29 (11)	3.081 (5)	150 (20)
O10—H10A <sup>i</sup> ⋯O4	0.89 (2)	2.06 (3)	2.929 (5)	163 (7)
O10—H10C <sup>i</sup> ⋯O8 <sup>iii</sup>	0.88 (2)	2.41 (5)	3.266 (5)	166 (15)
O10—H10B <sup>i</sup> ⋯O1 <sup>iv</sup>	0.87 (2)	2.46 (11)	3.212 (5)	145 (15)

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

Data collection: *IPDS Software* (Stoe & Cie, 1998); cell refinement: *IPDS Software*; data reduction: *X-RED32* (Stoe & Cie, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *SHELXL97*.

The authors thank the University of Zürich and the Swiss National Science Foundation for financial support.

---

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2260).

---

## References

- Bardina, N. V., Bazhenova, T. A., Lyssenko, K. A., Antipin, M. Yu., Shulga, Y. M., Filina, T. A. & Shestakov, A. F. (2006). *Mendeleev Commun.* **6**, 307–308.
- Chilou, V., Gouzerh, P., Jeannin, Y., Olivares, G., Robert, F., Hsieh, T.-C. & Zubieta, J. (1989). *Polyhedron*, **8**, 29–38.
- Clegg, W., Elsegood, M. R. J., Errington, R. J. & Bakri, R. (1996). *Acta Cryst.* **C52**, 2145–2148.
- Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Hayton, T. W., Legzdins, P. & Rettig, S. J. (2002). *Acta Cryst.* **C58**, m377–m378.
- Hsieh, T.-C. & Zubieta, J. (1987). *Inorg. Chim. Acta*, **127**, L31–L32.
- Kessler, V. G., Mironov, A. V., Turova, N. Ya., Yanovsky, A. I. & Struchkov, Y. T. (1993). *Polyhedron*, **12**, 1573–1576.
- Orpen, G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–83.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Stoe & Cie (1998). *IPDS Software* (Version 2.87 5/1998) and *X-RED32* (Version 1.01). Stoe & Cie, Darmstadt, Germany.

**supplementary materials**

*Acta Cryst.* (2007). E63, m2935-m2936 [ doi:10.1107/S1600536807055894 ]

## Di- $\mu$ -methoxy-bis[aquadimethoxonitrosylmolybdenum(II)]

J. Bucher, O. Blacque and H. Berke

### Comment

The molybdenum dimer (I) exhibits a planar central four-membered Mo<sub>2</sub>O<sub>2</sub> ring (torsional angle of 1.33 (11)°) with a Mo—Mo distance of 3.3933 (7) Å excluding any metal–metal bond. The nitrosyl ligands are nearly linear (176.7 (3) and 175.2 (3)°) and coordinated in *trans* positions to the methoxy bridges as observed in [Mo<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>(OMe)<sub>2</sub>(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>2</sub>(NO)<sub>2</sub>] (Hayton *et al.*, 2002). Two terminal methoxy ligands complete the nearly planar core [Mo<sub>2</sub>( $\mu$ -OMe)<sub>2</sub>(NO)<sub>2</sub>(OMe)<sub>2</sub>] whereas the two other ones are almost perpendicular to this plane and *trans* to coordinated water molecules. The Mo—O<sub>aq</sub> bond distances of 2.269 (4) Å and 2.274 (4) Å are longer than the reported unweighted mean of 2.201 Å (Orpen *et al.*, 1989).

In this conformation we observe two intramolecular O—H $\cdots$ H contacts (H10A $\cdots$ O4 = 2.06 (3) Å, O10 $\cdots$ O4 = 2.929 (5) Å; H5A $\cdots$ O8 = 2.24 (6) Å, O5 $\cdots$ O8 = 2.991 (5) Å) between the methoxy groups and the water molecules located on the same side of the Mo<sub>2</sub>O<sub>2</sub> plane (Fig. 1). The other water H atoms are positionally disordered and form intermolecular hydrogen bonds with oxygen atoms of the nitrosyl groups and of one terminal methoxy ligand (O $\cdots$ O < 3.3 Å).

### Experimental

Two equivalents of sodium hydroxide were added to a methanol solution of Mo(NO)<sub>2</sub>(Cl)<sub>2</sub> in order to produce Mo(NO)<sub>2</sub>(OH)<sub>2</sub>(MeOH)<sub>2</sub>. The green solution was slowly added to two equivalents of 1,3,5-triaza-7-phosphaadamantate (PTA) partly dissolved in methanol and the colour of the solution turned red within one hour. The IR spectrum of the reaction solution exhibited a strong NO band at 1638 cm<sup>-1</sup> indicating loss of one nitrosyl ligand from the molybdenum coordination sphere. Also the solid-state IR (ATR) spectrum of the soluble part revealed a  $\nu$ (NO) absorption at 1640 cm<sup>-1</sup> and a very weak one at 1620 cm<sup>-1</sup>, and additionally a strong  $\nu$ (CO) band at 1037 cm<sup>-1</sup> and a few other signals attributable to PTA and to the corresponding phosphine oxide (OPTA). The compounds soluble in methanol were redissolved in dichloromethane and red crystals of compound [Mo( $\mu$ -OMe)(OMe)<sub>2</sub>(NO)(H<sub>2</sub>O)]<sub>2</sub> (I) co-crystallized together with PTA and OPTA.

### Refinement

While the hydrogen atoms attached to carbon atoms were placed in geometrically calculated positions, those on water molecules were located in a difference Fourier map and then refined with the O—H distances restrained to 0.86 (2) Å. Three residual peaks could be attributed to H atoms for each coordinated water molecules. The positional disorders were refined with H5A and H10A with occupancy factors as unity and the second hydrogen atoms occupying two sites, H5B/H5C and H10B/H10C (occupancy factors of 1/2). Except H5A and H10A, all H atoms were included in the refinement with  $U_{iso}$  values of 1.3 $U_{eq}$  (parent atom).

## Figures

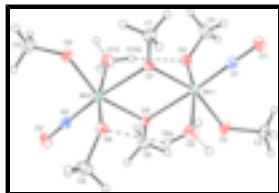


Fig. 1. The molecular structure of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

## Di- $\mu$ -methoxy-bis[aquadimethoxynitrosylmolybdenum(II)]

### Crystal data

[Mo<sub>2</sub>(CH<sub>3</sub>O)<sub>6</sub>(NO)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]

$M_r = 474.14$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.7567$  (13) Å

$b = 15.6682$  (17) Å

$c = 12.5488$  (18) Å

$\beta = 106.750$  (16)°

$V = 1648.7$  (4) Å<sup>3</sup>

$Z = 4$

$F_{000} = 944$

$D_x = 1.91$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 7998 reflections

$\theta = 2.8$ – $30.3$ °

$\mu = 1.56$  mm<sup>-1</sup>

$T = 183$  (2) K

Prism, red

$0.5 \times 0.45 \times 0.43$  mm

### Data collection

Stoe IPDS  
diffractometer

$\phi$  rotation scan

Absorption correction: numerical  
(Coppens *et al.*, 1965)

$T_{\min} = 0.522$ ,  $T_{\max} = 0.614$

19216 measured reflections

3874 independent reflections

3240 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.085$

$\theta_{\max} = 28$ °

$\theta_{\min} = 3.6$ °

$h = -11$ → $11$

$k = 0$ → $20$

$l = 0$ → $16$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.128$

$S = 1.11$

3874 reflections

4 restraints

H atoms treated by a mixture of  
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2]$

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

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

$\Delta\rho_{\max} = 0.75$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.21$  e Å<sup>-3</sup>

207 parameters

Extinction correction: none

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Mo1	0.14909 (4)	0.05522 (2)	0.25247 (3)	0.02109 (13)	
Mo2	0.35941 (4)	0.22983 (2)	0.21836 (3)	0.02114 (13)	
N1	-0.0325 (4)	0.0077 (2)	0.1801 (3)	0.0242 (7)	
O1	-0.1556 (4)	-0.0263 (2)	0.1254 (3)	0.0327 (7)	
N2	0.5425 (4)	0.2756 (2)	0.2931 (3)	0.0247 (7)	
O2	0.6664 (4)	0.3052 (2)	0.3518 (3)	0.0359 (8)	
O3	0.2412 (4)	-0.04131 (19)	0.3389 (3)	0.0299 (7)	
C3	0.1717 (6)	-0.1235 (3)	0.3376 (4)	0.0337 (10)	
H3A	0.0749	-0.1187	0.3586	0.044*	
H3B	0.2449	-0.1602	0.3893	0.044*	
H3C	0.1484	-0.1472	0.2641	0.044*	
O4	0.0788 (4)	0.11761 (19)	0.3613 (3)	0.0279 (6)	
C4	-0.0701 (6)	0.1164 (3)	0.3822 (5)	0.0391 (11)	
H4A	-0.1442	0.0841	0.3252	0.051*	
H4B	-0.1085	0.1738	0.3823	0.051*	
H4C	-0.0595	0.0905	0.4533	0.051*	
O5	0.2715 (5)	0.0038 (2)	0.1292 (3)	0.0407 (8)	
H5A	0.356 (7)	0.035 (5)	0.131 (8)	0.09 (3)*	
H5B	0.30 (2)	-0.048 (5)	0.145 (18)	0.114*	0.50
H5C	0.21 (2)	-0.004 (14)	0.061 (6)	0.114*	0.50
O6	0.3733 (3)	0.12295 (17)	0.3148 (2)	0.0212 (5)	
C6	0.4855 (6)	0.1103 (3)	0.4203 (4)	0.0313 (9)	
H6A	0.5912	0.1201	0.4148	0.041*	
H6B	0.4774	0.0529	0.4447	0.041*	
H6C	0.4639	0.1495	0.4730	0.041*	
O7	0.1374 (3)	0.16046 (17)	0.1539 (2)	0.0224 (6)	
C7	0.0167 (6)	0.1784 (3)	0.0530 (4)	0.0312 (9)	
H7A	0.0275	0.1402	-0.0042	0.041*	
H7B	0.0275	0.2362	0.0309	0.041*	
H7C	-0.0864	0.1709	0.0643	0.041*	
O8	0.4287 (4)	0.16934 (19)	0.1057 (3)	0.0274 (6)	
C8	0.5864 (6)	0.1568 (4)	0.1018 (5)	0.0395 (11)	
H8A	0.6580	0.1903	0.1588	0.051*	
H8B	0.5949	0.1739	0.0303	0.051*	
H8C	0.6139	0.0975	0.1138	0.051*	
O9	0.2601 (4)	0.32633 (19)	0.1369 (3)	0.0307 (7)	
C9	0.3085 (7)	0.4130 (3)	0.1455 (4)	0.0376 (11)	

## supplementary materials

---

H9A	0.2799	0.4395	0.2060	0.049*	
H9B	0.2564	0.4422	0.0774	0.049*	
H9C	0.4220	0.4161	0.1588	0.049*	
O10	0.2458 (5)	0.2776 (2)	0.3488 (4)	0.0392 (8)	
H10A	0.188 (7)	0.235 (3)	0.363 (6)	0.06 (2)*	
H10B	0.190 (18)	0.324 (6)	0.328 (14)	0.081*	0.50
H10C	0.305 (17)	0.297 (10)	0.413 (7)	0.081*	0.50

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Mo1	0.0247 (2)	0.02005 (19)	0.0179 (2)	-0.00202 (12)	0.00518 (15)	0.00039 (11)
Mo2	0.0235 (2)	0.01972 (19)	0.0211 (2)	-0.00026 (11)	0.00789 (15)	0.00202 (12)
N1	0.0294 (19)	0.0235 (16)	0.0190 (16)	-0.0002 (13)	0.0060 (15)	0.0005 (13)
O1	0.0332 (18)	0.0349 (17)	0.0261 (16)	-0.0083 (13)	0.0025 (14)	-0.0037 (13)
N2	0.0278 (19)	0.0248 (17)	0.0234 (18)	-0.0014 (13)	0.0106 (16)	0.0017 (13)
O2	0.0309 (17)	0.0423 (19)	0.0331 (18)	-0.0151 (15)	0.0071 (15)	-0.0078 (15)
O3	0.0353 (17)	0.0251 (14)	0.0269 (16)	-0.0017 (12)	0.0053 (14)	0.0059 (12)
C3	0.047 (3)	0.0226 (19)	0.031 (2)	-0.0029 (19)	0.010 (2)	0.0056 (17)
O4	0.0303 (16)	0.0289 (15)	0.0273 (16)	-0.0050 (12)	0.0126 (14)	-0.0034 (12)
C4	0.036 (3)	0.044 (3)	0.042 (3)	-0.011 (2)	0.019 (2)	-0.018 (2)
O5	0.048 (2)	0.0381 (19)	0.038 (2)	-0.0015 (17)	0.0153 (18)	-0.0040 (16)
O6	0.0217 (14)	0.0213 (12)	0.0170 (13)	-0.0015 (10)	-0.0003 (11)	0.0018 (10)
C6	0.031 (2)	0.034 (2)	0.025 (2)	-0.0029 (18)	0.0015 (19)	0.0077 (17)
O7	0.0224 (14)	0.0239 (13)	0.0185 (14)	0.0004 (11)	0.0020 (12)	0.0037 (11)
C7	0.029 (2)	0.035 (2)	0.024 (2)	0.0006 (17)	-0.0023 (18)	0.0072 (17)
O8	0.0312 (16)	0.0298 (15)	0.0243 (15)	0.0002 (12)	0.0128 (13)	-0.0014 (12)
C8	0.034 (3)	0.050 (3)	0.039 (3)	0.004 (2)	0.018 (2)	-0.008 (2)
O9	0.0354 (17)	0.0252 (15)	0.0349 (18)	0.0032 (12)	0.0153 (15)	0.0095 (12)
C9	0.057 (3)	0.022 (2)	0.039 (3)	0.004 (2)	0.023 (3)	0.0048 (19)
O10	0.043 (2)	0.0349 (18)	0.045 (2)	-0.0004 (15)	0.0203 (19)	-0.0024 (15)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Mo1—Mo2	3.3933 (7)	O5—H5A	0.89 (2)
Mo1—N1	1.754 (4)	O5—H5B	0.87 (2)
Mo1—O3	1.900 (3)	O5—H5C	0.87 (2)
Mo1—O4	1.919 (3)	O6—C6	1.417 (5)
Mo1—O5	2.269 (4)	C6—H6A	0.9600
Mo1—O6	2.170 (3)	C6—H6B	0.9600
Mo1—O7	2.046 (3)	C6—H6C	0.9600
Mo2—N2	1.760 (4)	O7—C7	1.425 (5)
Mo2—O6	2.049 (3)	C7—H7A	0.9600
Mo2—O7	2.170 (3)	C7—H7B	0.9600
Mo2—O8	1.939 (3)	C7—H7C	0.9600
Mo2—O9	1.891 (3)	O8—C8	1.410 (6)
Mo2—O10	2.274 (4)	C8—H8A	0.9600
N1—O1	1.220 (5)	C8—H8B	0.9600
N2—O2	1.214 (5)	C8—H8C	0.9600

O3—C3	1.423 (5)	O9—C9	1.418 (5)
C3—H3A	0.9600	C9—H9A	0.9600
C3—H3B	0.9600	C9—H9B	0.9600
C3—H3C	0.9600	C9—H9C	0.9600
O4—C4	1.402 (5)	O10—H10A	0.89 (2)
C4—H4A	0.9600	O10—H10B	0.87 (2)
C4—H4B	0.9600	O10—H10C	0.88 (2)
C4—H4C	0.9600		
N1—Mo1—O3	97.48 (15)	H4B—C4—H4C	109.5
N1—Mo1—O4	98.96 (15)	Mo1—O5—H5A	110 (6)
O3—Mo1—O4	99.75 (14)	Mo1—O5—H5B	111 (10)
N1—Mo1—O5	91.86 (16)	H5A—O5—H5B	108 (10)
O3—Mo1—O5	84.29 (15)	Mo1—O5—H5C	117 (10)
O4—Mo1—O5	167.81 (13)	H5A—O5—H5C	111 (10)
O7—Mo1—O5	79.93 (13)	H5B—O5—H5C	99 (10)
O6—Mo1—O5	82.13 (13)	C6—O6—Mo2	125.0 (2)
N1—Mo1—O6	170.28 (14)	C6—O6—Mo1	125.5 (2)
O3—Mo1—O6	89.56 (12)	Mo2—O6—Mo1	107.06 (12)
O4—Mo1—O6	86.38 (12)	O6—C6—H6A	109.5
O7—Mo1—O6	72.90 (11)	O6—C6—H6B	109.5
N1—Mo1—O7	98.60 (14)	H6A—C6—H6B	109.5
O3—Mo1—O7	157.76 (13)	O6—C6—H6C	109.5
O4—Mo1—O7	92.82 (12)	H6A—C6—H6C	109.5
N2—Mo2—O6	97.39 (14)	H6B—C6—H6C	109.5
O9—Mo2—O6	155.87 (13)	C7—O7—Mo1	126.3 (3)
O8—Mo2—O6	93.05 (12)	C7—O7—Mo2	126.0 (2)
N2—Mo2—O7	169.55 (14)	Mo1—O7—Mo2	107.17 (12)
O9—Mo2—O7	88.62 (12)	O7—C7—H7A	109.5
O8—Mo2—O7	85.31 (12)	O7—C7—H7B	109.5
O6—Mo2—O7	72.85 (11)	H7A—C7—H7B	109.5
N2—Mo2—O8	99.19 (15)	O7—C7—H7C	109.5
O9—Mo2—O8	100.86 (14)	H7A—C7—H7C	109.5
N2—Mo2—O9	99.69 (15)	H7B—C7—H7C	109.5
N2—Mo2—O10	90.14 (15)	C8—O8—Mo2	127.5 (3)
O9—Mo2—O10	84.10 (14)	O8—C8—H8A	109.5
O8—Mo2—O10	168.46 (13)	O8—C8—H8B	109.5
O6—Mo2—O10	78.93 (12)	H8A—C8—H8B	109.5
O7—Mo2—O10	84.40 (13)	O8—C8—H8C	109.5
O1—N1—Mo1	176.7 (3)	H8A—C8—H8C	109.5
O2—N2—Mo2	175.2 (3)	H8B—C8—H8C	109.5
C3—O3—Mo1	127.2 (3)	C9—O9—Mo2	130.1 (3)
O3—C3—H3A	109.5	O9—C9—H9A	109.5
O3—C3—H3B	109.5	O9—C9—H9B	109.5
H3A—C3—H3B	109.5	H9A—C9—H9B	109.5
O3—C3—H3C	109.5	O9—C9—H9C	109.5
H3A—C3—H3C	109.5	H9A—C9—H9C	109.5
H3B—C3—H3C	109.5	H9B—C9—H9C	109.5
C4—O4—Mo1	129.2 (3)	Mo2—O10—H10A	107 (5)
O4—C4—H4A	109.5	Mo2—O10—H10B	112 (10)



## supplementary materials

O4—C4—H4B	109.5	H10A—O10—H10B	113 (10)
H4A—C4—H4B	109.5	Mo2—O10—H10C	120 (10)
O4—C4—H4C	109.5	H10A—O10—H10C	108 (10)
H4A—C4—H4C	109.5	H10B—O10—H10C	97 (10)
N1—Mo1—O3—C3	0.0 (4)	O3—Mo1—O7—C7	131.3 (4)
O4—Mo1—O3—C3	100.5 (4)	O4—Mo1—O7—C7	-104.0 (3)
O7—Mo1—O3—C3	-136.0 (4)	O6—Mo1—O7—C7	170.7 (4)
O6—Mo1—O3—C3	-173.2 (4)	O5—Mo1—O7—C7	85.9 (3)
O5—Mo1—O3—C3	-91.1 (4)	N1—Mo1—O7—Mo2	-176.42 (15)
N1—Mo1—O4—C4	6.2 (4)	O3—Mo1—O7—Mo2	-40.6 (4)
O3—Mo1—O4—C4	-93.0 (4)	O4—Mo1—O7—Mo2	84.08 (14)
O7—Mo1—O4—C4	105.4 (4)	O6—Mo1—O7—Mo2	-1.26 (11)
O6—Mo1—O4—C4	178.1 (4)	O5—Mo1—O7—Mo2	-86.05 (15)
O5—Mo1—O4—C4	158.5 (6)	N2—Mo2—O7—C7	168.0 (7)
N2—Mo2—O6—C6	12.2 (3)	O9—Mo2—O7—C7	25.1 (3)
O9—Mo2—O6—C6	-122.6 (4)	O8—Mo2—O7—C7	-76.0 (3)
O8—Mo2—O6—C6	111.9 (3)	O6—Mo2—O7—C7	-170.6 (3)
O7—Mo2—O6—C6	-164.0 (3)	O10—Mo2—O7—C7	109.3 (3)
O10—Mo2—O6—C6	-76.5 (3)	N2—Mo2—O7—Mo1	-20.0 (8)
N2—Mo2—O6—Mo1	174.92 (15)	O9—Mo2—O7—Mo1	-162.99 (15)
O9—Mo2—O6—Mo1	40.1 (4)	O8—Mo2—O7—Mo1	95.99 (14)
O8—Mo2—O6—Mo1	-85.39 (14)	O6—Mo2—O7—Mo1	1.33 (11)
O7—Mo2—O6—Mo1	-1.26 (11)	O10—Mo2—O7—Mo1	-78.79 (15)
O10—Mo2—O6—Mo1	86.24 (15)	N2—Mo2—O8—C8	9.3 (4)
O3—Mo1—O6—C6	-29.9 (3)	O9—Mo2—O8—C8	111.1 (4)
O4—Mo1—O6—C6	69.9 (3)	O6—Mo2—O8—C8	-88.7 (4)
O7—Mo1—O6—C6	164.0 (3)	O7—Mo2—O8—C8	-161.2 (4)
O5—Mo1—O6—C6	-114.2 (3)	O10—Mo2—O8—C8	-134.2 (7)
O3—Mo1—O6—Mo2	167.46 (15)	N2—Mo2—O9—C9	-8.2 (4)
O4—Mo1—O6—Mo2	-92.74 (15)	O8—Mo2—O9—C9	-109.6 (4)
O7—Mo1—O6—Mo2	1.33 (11)	O6—Mo2—O9—C9	126.2 (4)
O5—Mo1—O6—Mo2	83.16 (15)	O7—Mo2—O9—C9	165.4 (4)
N1—Mo1—O7—C7	-4.5 (4)	O10—Mo2—O9—C9	80.9 (4)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A $\cdots$ O8	0.89 (2)	2.24 (6)	2.991 (5)	142 (8)
O5—H5B $\cdots$ O2 <sup>i</sup>	0.87 (2)	2.31 (6)	3.156 (5)	165 (20)
O5—H5C $\cdots$ O1 <sup>ii</sup>	0.87 (2)	2.29 (11)	3.081 (5)	150 (20)
O10—H10A $\cdots$ O4	0.89 (2)	2.06 (3)	2.929 (5)	163 (7)
O10—H10C $\cdots$ O8 <sup>iii</sup>	0.88 (2)	2.41 (5)	3.266 (5)	166 (15)
O10—H10B $\cdots$ O1 <sup>iv</sup>	0.87 (2)	2.46 (11)	3.212 (5)	145 (15)

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

Fig. 1

