Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[1,1′-(butane-1,4-diyl)diimidazolium] δ-octamolybdate(VI)

Hai-Ge Hou* and Jing-Lin Yang

School of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China
Correspondence e-mail: huohaige@tom.com

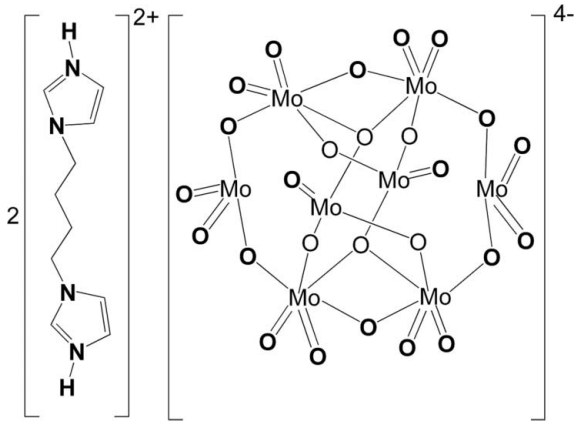
Received 3 June 2007; accepted 16 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.021; wR factor = 0.048; data-to-parameter ratio = 17.3.

The title compound, $(C_{10}H_{16}N_4)_2[\delta\text{-}Mo_8O_{26}]$, was synthesized by a hydrothermal reaction. The structure consists of a centrosymmetric δ-octamolybdate anion and two 1,1′-(butane-1,4-diyl)bis(imidazolium) cations for charge balance. The anions and cations are linked by N—H···O hydrogen bonds, generating a three-dimensional supramolecular structure.

Related literature

For related structures, see: Allis *et al.* (2004); Bridgeman (2002); Kalpana & Vidyasagar (2005). For the ligand synthesis, see: Ma *et al.* (2003).



Experimental

Crystal data

$(C_{10}H_{16}N_4)_2[Mo_8O_{26}]$
M_r = 1568.06
Monoclinic, $P2_1/n$
a = 12.252 (3) Å
b = 13.766 (3) Å
c = 12.405 (4) Å
β = 100.371 (4)°
V = 2058.1 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.45 mm^{-1}
T = 293 (2) K
0.40 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.405, T_{max} = 0.512
12476 measured reflections
4979 independent reflections
4400 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.048
S = 1.03
4979 reflections
288 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—O5	1.6843 (18)	Mo3—O12	1.6945 (19)
Mo1—O6	1.6983 (18)	Mo3—O11	1.6952 (17)
Mo1—O13^{i}	1.9249 (15)	Mo3—O13	1.9010 (16)
Mo1—O3	1.9738 (16)	Mo3—O4	1.9959 (17)
Mo1—O9^{i}	2.3020 (18)	Mo3—O10	2.3128 (17)
Mo1—O7	2.3377 (16)	Mo3—O7^{i}	2.3403 (15)
Mo2—O8	1.7055 (18)	Mo4—O2	1.7007 (18)
Mo2—O9	1.7611 (17)	Mo4—O1	1.7202 (16)
Mo2—O10	1.7788 (16)	Mo4—O3	1.8296 (17)
Mo2—O7	1.8367 (16)	Mo4—O4	1.8331 (17)

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{ii}	0.86 (3)	1.98 (3)	2.826 (3)	171 (3)
N4—H4N···O1^{iii}	0.86 (3)	2.27 (3)	3.009 (3)	143 (4)
N4—H4N···O6^{iv}	0.86 (3)	2.22 (4)	2.761 (3)	121 (3)

Symmetry codes: (ii) $-x+1, -y+1, -z+1$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, y, z-1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2067).

References

Allis, D. G., Rarig, R. S., Burkholder, E. & Zubieta, J. (2004). *J. Mol. Struct.* **688**, 11–31.
Bridgeman, A. J. (2002). *J. Phys. Chem. A*, **106**, 12151–12160.
Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kalpana, G. & Vidyasagar, K. (2005). *Acta Cryst.* E**61**, m1885–m1886.
Ma, J. F., Yang, J., Zheng, G. L., Li, L. & Liu, J. F. (2003). *Inorg. Chem.* **42**, 7531–7534.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807034605

supplementary materials

Acta Cryst. (2007). E**63**, m2194 [**doi:10.1107/S1600536807034605**]

Bis[1,1'-(butane-1,4-diyl)diimidazolium] δ-octamolybdate(VI)

H.-G. Hou and J.-L. Yang

Comment

Recently, much of work has been focused on the polyoxometalate with different neutral ligands. In this paper, the structure of the title compound, (I), is described (Fig. 1).

The compound (I) contains a centrosymmetric δ-octamolybdate anion (Allis *et al.*, 2004; Bridgeman, 2002) and two 1,1'-(butane-1,4-diyl)bis(imidazolium) cations. As shown in Fig. 2, each cation connects three anions through hydrogen bonds (Table 2), while each anion links six cations (Fig. 3). All anions and cations are linked each other in this mode to give a three-dimensional supramolecular structure. The Mo—O bond distances and angles are in normal range (Kalpana & Vidyasagar, 2005) (Table 1).

Experimental

1,1'-(Butane-1,4-diyl)bis(imidazole) was synthesized according to the method reported by Ma *et al.* (2003). A mixture of 1,1'-(butane-1,4-diyl)bis(imidazole) (0.190 g, 0.1 mmol), $Na_2MoO_4{\cdot}2H_2O$ (0.242 g, 1.0 mmol) and H_2O (10 ml) was adjusted to pH $\simeq$ 4 with HNO_3 (1 mol L^{-1}) and then transferred to and sealed in a 25 ml Teflon-lined stainless steel container. The container was heated to 423 K for 72 h, then cooled to room temperature. Colorless crystals of (I) were collected (yield 58.3% based on $Na_2MoO_4{\cdot}2H_2O$). Analysis calculated for $C_{20}H_{32}Mo_8N_8O_{26}$: C 15.32, H 2.06, N 7.15%; found: C 15.30, H 2.07, N 7.18%.

Refinement

All H atoms on C atoms were positioned geometrically and refined as riding, with C—H = 0.93 (CH), 0.97 Å (CH_2) and U_{iso}(H) = 1.2U_{eq}(C). The H atoms bonded to N atoms were located on a difference Fourier map and refined isotropically with a distance restraint of 0.86 (3) Å.

Figures

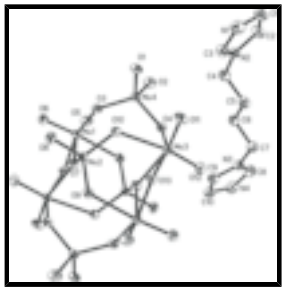


Fig. 1. The structures of the anion and cation of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Fig. 2. The hydrogen bonds around the 1,1'-(butane-1,4-diyl)bis(imidazole) ligand. Dashed lines denote hydrogen bonds. Only the H atoms involving hydrogen bonds have been shown. [Symmetry codes: (ii) −*x* + 1, −*y* + 1, −*z* + 1; (iii) −*x* + 3/2, *y* + 1/2, −*z* + 1/2; (iv) *x*, *y*, *z* − 1.]

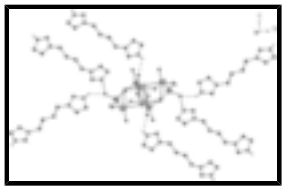


Fig. 3. The hydrogen bonds around the octamolybdate anion. Dashed lines denote hydrogen bonds. Only the H atoms involving hydrogen bonds have been shown.

Bis[1,1'-(butane-1,4-diyl)diimidazolium] δ-octamolybdate(VI)

Crystal data

$(C_{10}H_{16}N_4)_2[Mo_8O_{26}]$	F_{000} = 1504
M_r = 1568.06	D_x = 2.530 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 10235 reflections
a = 12.252 (3) Å	θ = 2.2–28.4º
b = 13.766 (3) Å	μ = 2.45 mm^{-1}
c = 12.405 (4) Å	T = 293 (2) K
β = 100.371 (4)º	Block, colourless
V = 2058.1 (9) Å^3	0.40 × 0.32 × 0.28 mm
Z = 2	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4979 independent reflections
Radiation source: fine-focus sealed tube	4400 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 293(2) K	θ_{max} = 28.4º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→12
T_{min} = 0.405, T_{max} = 0.512	k = −18→16
12476 measured reflections	l = −16→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.021	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.048	$w = 1/[\sigma^2(F_o^2) + (0.0222P)^2 + 0.1238P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.002

4979 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
288 parameters	$\Delta\rho_{min}$ = −0.63 e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.754292 (17)	1.019021 (14)	0.622942 (17)	0.02690 (6)
Mo2	0.455430 (16)	0.935845 (13)	0.613148 (16)	0.02382 (5)
Mo3	0.420322 (17)	0.803161 (14)	0.358359 (17)	0.02803 (6)
Mo4	0.687630 (18)	0.785795 (14)	0.499020 (17)	0.02708 (6)
O8	0.45559 (16)	0.90491 (13)	0.74631 (15)	0.0406 (4)
O10	0.48032 (14)	0.82703 (11)	0.54413 (14)	0.0303 (4)
O1	0.65759 (14)	0.67556 (12)	0.55209 (15)	0.0354 (4)
O7	0.56208 (13)	1.02931 (11)	0.61139 (14)	0.0284 (4)
O5	0.88155 (15)	1.01901 (14)	0.58946 (17)	0.0419 (5)
O11	0.41126 (16)	0.68162 (12)	0.37685 (17)	0.0452 (5)
O6	0.77946 (16)	1.01455 (12)	0.76203 (15)	0.0380 (4)
O12	0.39766 (17)	0.82020 (15)	0.22085 (16)	0.0461 (5)
O2	0.80695 (16)	0.76876 (14)	0.44952 (17)	0.0436 (5)
O13	0.28365 (13)	0.84549 (11)	0.39446 (14)	0.0302 (4)
O9	0.32230 (14)	0.98175 (12)	0.56067 (14)	0.0320 (4)
O4	0.58508 (14)	0.81338 (12)	0.37603 (14)	0.0306 (4)
N1	0.5128 (2)	0.38184 (18)	0.3352 (2)	0.0499 (6)
C7	0.5839 (2)	0.77486 (18)	0.0168 (2)	0.0416 (7)
H7A	0.5988	0.7377	−0.0454	0.050*
H7B	0.5043	0.7751	0.0143	0.050*
C1	0.5719 (3)	0.3377 (2)	0.2674 (3)	0.0499 (8)
H1	0.5733	0.2716	0.2523	0.060*
O3	0.71282 (15)	0.88071 (11)	0.60380 (14)	0.0352 (4)
N3	0.62268 (18)	0.87472 (15)	0.00892 (18)	0.0347 (5)
N2	0.60261 (18)	0.49305 (14)	0.27097 (18)	0.0322 (5)
C8	0.6744 (3)	0.9098 (2)	−0.0680 (3)	0.0476 (7)
H8	0.6948	0.8748	−0.1254	0.057*
C9	0.6086 (2)	0.94964 (19)	0.0782 (2)	0.0423 (7)
H9	0.5751	0.9452	0.1396	0.051*
C6	0.6404 (2)	0.72714 (18)	0.1215 (2)	0.0384 (6)
H6A	0.6394	0.7712	0.1823	0.046*
H6B	0.7172	0.7143	0.1169	0.046*
C3	0.5309 (2)	0.4750 (2)	0.3355 (2)	0.0420 (7)
H3	0.4985	0.5210	0.3748	0.050*
C2	0.6282 (3)	0.4077 (2)	0.2261 (3)	0.0466 (7)
H2	0.6757	0.3995	0.1763	0.056*
C4	0.6455 (2)	0.58912 (19)	0.2472 (2)	0.0421 (7)
H4A	0.7233	0.5830	0.2422	0.051*
H4B	0.6403	0.6327	0.3075	0.051*

C5	0.5841 (2)	0.63262 (19)	0.1429 (2)	0.0382 (6)
H5A	0.5076	0.6449	0.1493	0.046*
H5B	0.5846	0.5880	0.0825	0.046*
C10	0.6510 (3)	1.0294 (2)	0.0423 (3)	0.0487 (7)
H10	0.6524	1.0910	0.0731	0.058*
N4	0.6918 (2)	1.00341 (19)	−0.0483 (2)	0.0517 (7)
H1N	0.466 (2)	0.360 (2)	0.373 (2)	0.063 (10)*
H4N	0.724 (3)	1.047 (2)	−0.081 (3)	0.085 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02472 (11)	0.02370 (10)	0.03129 (12)	0.00105 (8)	0.00240 (8)	−0.00177 (8)
Mo2	0.02711 (11)	0.02013 (10)	0.02486 (11)	−0.00093 (8)	0.00640 (8)	0.00104 (7)
Mo3	0.03107 (12)	0.02043 (10)	0.03198 (12)	0.00099 (8)	0.00405 (9)	−0.00463 (8)
Mo4	0.03080 (12)	0.02067 (10)	0.03081 (12)	0.00111 (8)	0.00835 (9)	0.00170 (8)
O8	0.0557 (12)	0.0381 (10)	0.0283 (10)	−0.0024 (9)	0.0080 (9)	0.0029 (8)
O10	0.0346 (9)	0.0238 (8)	0.0323 (9)	0.0015 (7)	0.0055 (7)	0.0014 (7)
O1	0.0393 (10)	0.0226 (8)	0.0469 (11)	0.0027 (7)	0.0150 (9)	0.0044 (7)
O7	0.0279 (9)	0.0206 (7)	0.0374 (10)	−0.0001 (6)	0.0072 (7)	−0.0023 (7)
O5	0.0294 (10)	0.0454 (11)	0.0520 (12)	0.0045 (8)	0.0103 (9)	0.0049 (9)
O11	0.0467 (12)	0.0237 (9)	0.0649 (14)	0.0004 (8)	0.0087 (10)	−0.0057 (9)
O6	0.0442 (11)	0.0334 (9)	0.0346 (10)	−0.0002 (8)	0.0021 (8)	−0.0010 (8)
O12	0.0522 (12)	0.0514 (11)	0.0336 (11)	0.0079 (10)	0.0045 (9)	−0.0056 (9)
O2	0.0352 (11)	0.0460 (11)	0.0524 (13)	0.0027 (9)	0.0155 (9)	0.0076 (9)
O13	0.0270 (9)	0.0230 (8)	0.0398 (10)	−0.0032 (7)	0.0046 (7)	−0.0011 (7)
O9	0.0301 (9)	0.0344 (9)	0.0331 (10)	0.0027 (7)	0.0100 (7)	−0.0021 (7)
O4	0.0317 (9)	0.0304 (8)	0.0309 (9)	0.0050 (7)	0.0090 (7)	−0.0013 (7)
N1	0.0540 (16)	0.0433 (14)	0.0546 (17)	−0.0089 (12)	0.0161 (13)	0.0132 (12)
C7	0.0443 (17)	0.0320 (13)	0.0453 (17)	−0.0079 (12)	−0.0003 (13)	0.0026 (12)
C1	0.058 (2)	0.0252 (13)	0.064 (2)	−0.0010 (13)	0.0045 (16)	0.0032 (13)
O3	0.0397 (10)	0.0234 (8)	0.0395 (10)	0.0029 (7)	−0.0011 (8)	−0.0007 (7)
N3	0.0385 (12)	0.0307 (11)	0.0348 (12)	−0.0046 (9)	0.0069 (9)	0.0048 (9)
N2	0.0365 (12)	0.0255 (10)	0.0363 (12)	−0.0001 (9)	0.0107 (9)	0.0028 (9)
C8	0.0488 (18)	0.0539 (18)	0.0417 (17)	−0.0024 (14)	0.0127 (14)	0.0039 (14)
C9	0.0519 (18)	0.0346 (14)	0.0409 (17)	−0.0032 (13)	0.0097 (13)	0.0012 (12)
C6	0.0433 (16)	0.0315 (13)	0.0403 (16)	−0.0073 (11)	0.0071 (12)	0.0022 (11)
C3	0.0416 (16)	0.0440 (16)	0.0433 (17)	0.0020 (13)	0.0159 (13)	−0.0015 (13)
C2	0.0534 (18)	0.0331 (14)	0.058 (2)	0.0030 (13)	0.0236 (15)	−0.0082 (13)
C4	0.0506 (18)	0.0314 (13)	0.0437 (17)	−0.0121 (12)	0.0067 (13)	0.0014 (12)
C5	0.0433 (16)	0.0298 (13)	0.0426 (16)	−0.0082 (11)	0.0101 (12)	0.0005 (11)
C10	0.0542 (19)	0.0342 (14)	0.0522 (19)	−0.0058 (13)	−0.0057 (15)	0.0037 (13)
N4	0.0512 (16)	0.0470 (15)	0.0561 (17)	−0.0175 (13)	0.0076 (13)	0.0185 (13)

Geometric parameters (Å, °)

Mo1—O5	1.6843 (18)	C7—C6	1.508 (4)
Mo1—O6	1.6983 (18)	C7—H7A	0.9700

Mo1—O13^{i}	1.9249 (15)	C7—H7B	0.9700
Mo1—O3	1.9738 (16)	C1—C2	1.339 (4)
Mo1—O9^{i}	2.3020 (18)	C1—H1	0.9300
Mo1—O7	2.3377 (16)	N3—C8	1.328 (3)
Mo2—O8	1.7055 (18)	N3—C9	1.373 (3)
Mo2—O9	1.7611 (17)	N2—C3	1.315 (3)
Mo2—O10	1.7788 (16)	N2—C2	1.360 (3)
Mo2—O7	1.8367 (16)	N2—C4	1.473 (3)
Mo3—O12	1.6945 (19)	C8—N4	1.322 (4)
Mo3—O11	1.6952 (17)	C8—H8	0.9300
Mo3—O13	1.9010 (16)	C9—C10	1.325 (4)
Mo3—O4	1.9959 (17)	C9—H9	0.9300
Mo3—O10	2.3128 (17)	C6—C5	1.518 (3)
Mo3—O7^{i}	2.3403 (15)	C6—H6A	0.9700
Mo4—O2	1.7007 (18)	C6—H6B	0.9700
Mo4—O1	1.7202 (16)	C3—H3	0.9300
Mo4—O3	1.8296 (17)	C2—H2	0.9300
Mo4—O4	1.8331 (17)	C4—C5	1.500 (4)
O7—Mo3^{i}	2.3403 (15)	C4—H4A	0.9700
O13—Mo1^{i}	1.9249 (15)	C4—H4B	0.9700
O9—Mo1^{i}	2.3020 (18)	C5—H5A	0.9700
N1—C3	1.302 (4)	C5—H5B	0.9700
N1—C1	1.350 (4)	C10—N4	1.357 (4)
N1—H1N	0.86 (3)	C10—H10	0.9300
C7—N3	1.464 (3)	N4—H4N	0.86 (3)
O5—Mo1—O6	104.11 (10)	N3—C7—C6	111.2 (2)
O5—Mo1—O13^{i}	100.84 (8)	N3—C7—H7A	109.4
O6—Mo1—O13^{i}	98.34 (8)	C6—C7—H7A	109.4
O5—Mo1—O3	101.53 (8)	N3—C7—H7B	109.4
O6—Mo1—O3	94.76 (8)	C6—C7—H7B	109.4
O13^{i}—Mo1—O3	150.39 (7)	H7A—C7—H7B	108.0
O5—Mo1—O9^{i}	89.23 (8)	C2—C1—N1	106.6 (2)
O6—Mo1—O9^{i}	166.45 (8)	C2—C1—H1	126.7
O13^{i}—Mo1—O9^{i}	81.16 (7)	N1—C1—H1	126.7
O3—Mo1—O9^{i}	80.00 (7)	Mo4—O3—Mo1	141.37 (10)
O5—Mo1—O7	162.17 (8)	C8—N3—C9	107.9 (2)
O6—Mo1—O7	93.50 (8)	C8—N3—C7	126.3 (2)
O13^{i}—Mo1—O7	73.39 (6)	C9—N3—C7	125.8 (2)
O3—Mo1—O7	79.42 (6)	C3—N2—C2	108.3 (2)
O9^{i}—Mo1—O7	73.32 (6)	C3—N2—C4	126.4 (2)
O8—Mo2—O9	106.68 (9)	C2—N2—C4	125.3 (2)
O8—Mo2—O10	106.59 (8)	N4—C8—N3	107.6 (3)
O9—Mo2—O10	110.60 (8)	N4—C8—H8	126.2
O8—Mo2—O7	108.04 (9)	N3—C8—H8	126.2
O9—Mo2—O7	110.97 (7)	C10—C9—N3	108.1 (3)
O10—Mo2—O7	113.59 (7)	C10—C9—H9	125.9

O12—Mo3—O11	105.66 (10)	N3—C9—H9	125.9
O12—Mo3—O13	101.72 (9)	C7—C6—C5	111.8 (2)
O11—Mo3—O13	100.88 (8)	C7—C6—H6A	109.3
O12—Mo3—O4	94.59 (9)	C5—C6—H6A	109.3
O11—Mo3—O4	98.30 (8)	C7—C6—H6B	109.3
O13—Mo3—O4	150.33 (7)	C5—C6—H6B	109.3
O12—Mo3—O10	161.53 (8)	H6A—C6—H6B	107.9
O11—Mo3—O10	91.27 (8)	N1—C3—N2	108.5 (2)
O13—Mo3—O10	81.79 (7)	N1—C3—H3	125.8
O4—Mo3—O10	75.33 (6)	N2—C3—H3	125.8
O12—Mo3—O7^{i}	91.07 (8)	C1—C2—N2	107.1 (3)
O11—Mo3—O7^{i}	163.21 (8)	C1—C2—H2	126.5
O13—Mo3—O7^{i}	73.73 (6)	N2—C2—H2	126.5
O4—Mo3—O7^{i}	81.42 (6)	N2—C4—C5	112.9 (2)
O10—Mo3—O7^{i}	72.34 (6)	N2—C4—H4A	109.0
O2—Mo4—O1	105.88 (9)	C5—C4—H4A	109.0
O2—Mo4—O3	107.99 (9)	N2—C4—H4B	109.0
O1—Mo4—O3	112.24 (8)	C5—C4—H4B	109.0
O2—Mo4—O4	103.47 (9)	H4A—C4—H4B	107.8
O1—Mo4—O4	109.84 (8)	C4—C5—C6	108.7 (2)
O3—Mo4—O4	116.49 (7)	C4—C5—H5A	110.0
Mo2—O10—Mo3	123.22 (8)	C6—C5—H5A	110.0
Mo2—O7—Mo1	131.90 (8)	C4—C5—H5B	110.0
Mo2—O7—Mo3^{i}	137.19 (8)	C6—C5—H5B	110.0
Mo1—O7—Mo3^{i}	89.26 (5)	H5A—C5—H5B	108.3
Mo3—O13—Mo1^{i}	118.41 (8)	C9—C10—N4	106.6 (3)
Mo2—O9—Mo1^{i}	123.34 (8)	C9—C10—H10	126.7
Mo4—O4—Mo3	127.22 (9)	N4—C10—H10	126.7
C3—N1—C1	109.6 (2)	C8—N4—C10	109.8 (2)
C3—N1—H1N	118 (2)	C8—N4—H4N	132 (3)
C1—N1—H1N	132 (2)	C10—N4—H4N	118 (3)
O8—Mo2—O10—Mo3	153.57 (10)	O12—Mo3—O4—Mo4	−170.00 (11)
O9—Mo2—O10—Mo3	37.97 (12)	O11—Mo3—O4—Mo4	−63.41 (12)
O7—Mo2—O10—Mo3	−87.57 (11)	O13—Mo3—O4—Mo4	66.47 (18)
O12—Mo3—O10—Mo2	52.1 (3)	O10—Mo3—O4—Mo4	25.73 (10)
O11—Mo3—O10—Mo2	−151.20 (11)	O7^{i}—Mo3—O4—Mo4	99.61 (11)
O13—Mo3—O10—Mo2	−50.39 (10)	C3—N1—C1—C2	0.4 (4)
O4—Mo3—O10—Mo2	110.56 (10)	O2—Mo4—O3—Mo1	61.40 (17)
O7^{i}—Mo3—O10—Mo2	25.06 (9)	O1—Mo4—O3—Mo1	177.73 (14)
O8—Mo2—O7—Mo1	81.32 (13)	O4—Mo4—O3—Mo1	−54.42 (18)
O9—Mo2—O7—Mo1	−162.05 (10)	O5—Mo1—O3—Mo4	−62.11 (17)
O10—Mo2—O7—Mo1	−36.70 (14)	O6—Mo1—O3—Mo4	−167.57 (16)
O8—Mo2—O7—Mo3^{i}	−79.26 (14)	O13^{i}—Mo1—O3—Mo4	76.3 (2)
O9—Mo2—O7—Mo3^{i}	37.36 (15)	O9^{i}—Mo1—O3—Mo4	25.03 (15)
O10—Mo2—O7—Mo3^{i}	162.72 (11)	O7—Mo1—O3—Mo4	99.74 (16)
O5—Mo1—O7—Mo2	105.1 (2)	C6—C7—N3—C8	118.5 (3)

O6—Mo1—O7—Mo2	−83.91 (12)	C6—C7—N3—C9	−63.8 (4)
O13^{i}—Mo1—O7—Mo2	178.43 (13)	C9—N3—C8—N4	−0.3 (3)
O3—Mo1—O7—Mo2	10.29 (11)	C7—N3—C8—N4	177.8 (3)
O9^{i}—Mo1—O7—Mo2	92.90 (12)	C8—N3—C9—C10	0.5 (3)
O5—Mo1—O7—Mo3^{i}	−88.0 (2)	C7—N3—C9—C10	−177.6 (3)
O6—Mo1—O7—Mo3^{i}	83.03 (7)	N3—C7—C6—C5	167.6 (2)
O13^{i}—Mo1—O7—Mo3^{i}	−14.63 (6)	C1—N1—C3—N2	−1.3 (4)
O3—Mo1—O7—Mo3^{i}	177.23 (7)	C2—N2—C3—N1	1.7 (3)
O9^{i}—Mo1—O7—Mo3^{i}	−100.16 (6)	C4—N2—C3—N1	179.6 (3)
O12—Mo3—O13—Mo1^{i}	−67.03 (12)	N1—C1—C2—N2	0.6 (4)
O11—Mo3—O13—Mo1^{i}	−175.73 (10)	C3—N2—C2—C1	−1.4 (3)
O4—Mo3—O13—Mo1^{i}	54.92 (18)	C4—N2—C2—C1	−179.3 (3)
O10—Mo3—O13—Mo1^{i}	94.55 (9)	C3—N2—C4—C5	−96.4 (3)
O7^{i}—Mo3—O13—Mo1^{i}	20.64 (8)	C2—N2—C4—C5	81.1 (3)
O8—Mo2—O9—Mo1^{i}	−155.56 (10)	N2—C4—C5—C6	−175.5 (2)
O10—Mo2—O9—Mo1^{i}	−40.03 (12)	C7—C6—C5—C4	179.3 (2)
O7—Mo2—O9—Mo1^{i}	86.98 (11)	N3—C9—C10—N4	−0.5 (4)
O2—Mo4—O4—Mo3	160.04 (11)	N3—C8—N4—C10	0.0 (4)
O1—Mo4—O4—Mo3	47.35 (12)	C9—C10—N4—C8	0.3 (4)
O3—Mo4—O4—Mo3	−81.65 (12)		

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1^{ii}	0.86 (3)	1.98 (3)	2.826 (3)	171 (3)
N4—H4N···O1^{iii}	0.86 (3)	2.27 (3)	3.009 (3)	143 (4)
N4—H4N···O6^{iv}	0.86 (3)	2.22 (4)	2.761 (3)	121 (3)

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iv) *x*, *y*, *z*−1.

Fig. 1

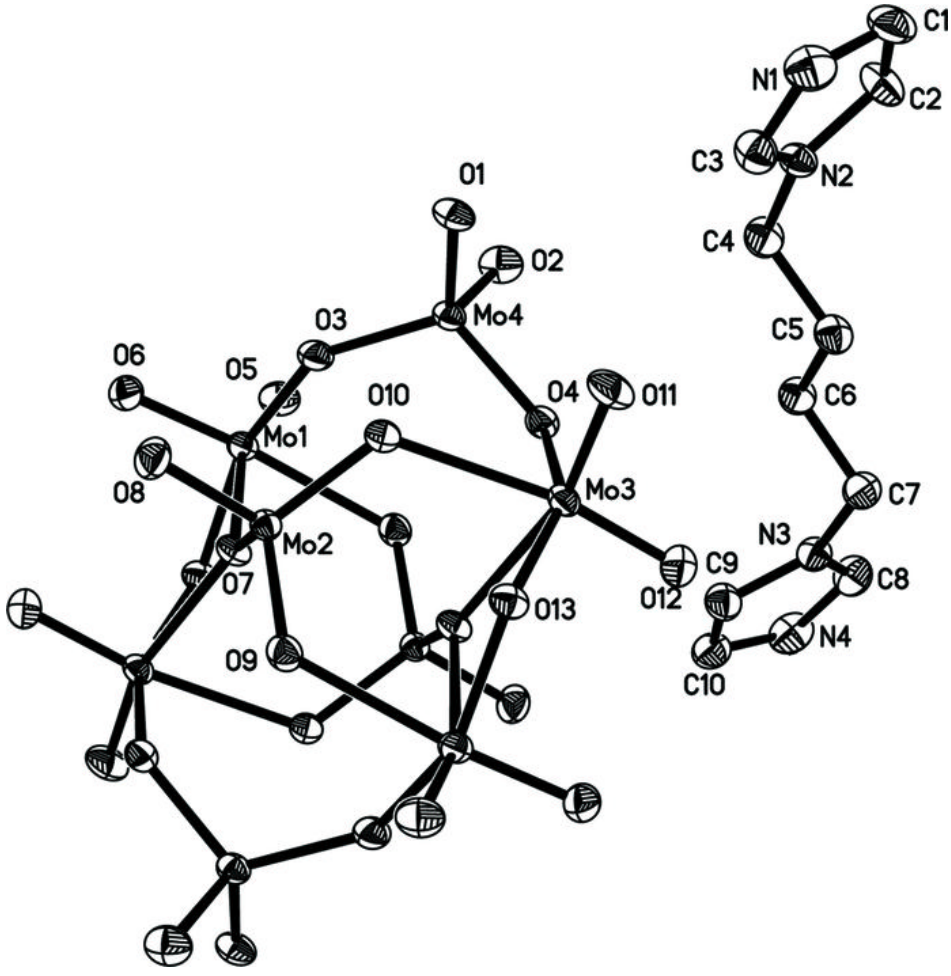

C1
N1
C2
C3
N2
O1
C4
O2
Mo4
O3
C5
O6
O5
O4
O11
C6
O10
Mo1
Mo3
O8
C7
N3
Mo2
C9
C8
O7
O12
O13
N4
O9
C10

Fig. 2

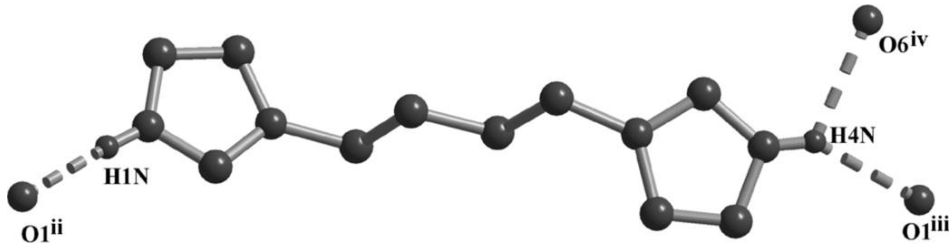


Fig. 3