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Tetrabenzimidazolium hexacosaoxidooctamolybdate(VI)

Wen Zhi, Zhan-Guo Zong, Jie Gong and Chuan-Bi Li*

College of Science, Changchun University, Changchun 130022, People's Republic of China
Correspondence e-mail: chuanbl@gmail.com

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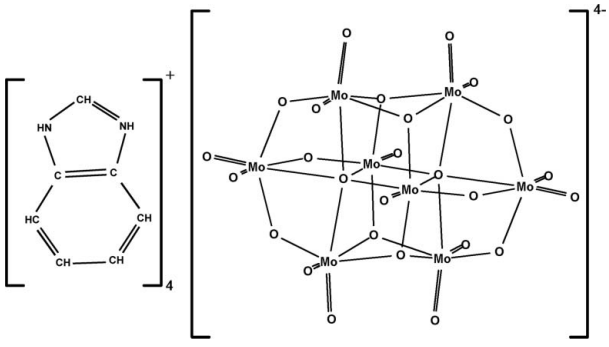
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.017; wR factor = 0.041; data-to-parameter ratio = 13.1.

The title compound, $(C_7H_7N_2)_4[Mo_8O_{26}]$, features centrosymmetric $[Mo_8O_{26}]^{4-}$ polyoxidomolybdate(VI) anions and benzimidazolium cations. The constituent MoO_6 octahedra show typical distortions. These components are connected to each other by way of N—H···O hydrogen bonds, thereby forming a three-dimensional framework.

Related literature

For related structures, see: Liu *et al.* (2006). For background literature, see: Brown & Altermatt (1985).



Experimental

Crystal data

$(C_7H_7N_2)_4[Mo_8O_{26}]$
M_r = 1660.10
Triclinic, $P\overline{1}$
a = 9.229 (1) Å
b = 10.225 (1) Å
c = 11.966 (2) Å
α = 84.510 (2)°
β = 83.261 (2)°
γ = 73.667 (1)°
V = 1073.8 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 2.36 mm^{-1}
T = 291 (2) K
0.24 × 0.17 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.630, T_{max} = 0.750
9089 measured reflections
4166 independent reflections
3739 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.041
S = 1.01
4166 reflections
317 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—O5	1.6890 (15)	Mo3—O1	1.6890 (15)
Mo1—O4	1.7113 (16)	Mo3—O2	1.7426 (14)
Mo1—O6	1.8964 (15)	Mo3—O3	1.9302 (14)
Mo1—O3	1.9896 (14)	Mo3—O12	1.9761 (14)
Mo1—O12^{i}	2.3304 (14)	Mo3—O9	2.1516 (14)
Mo1—O9	2.3744 (14)	Mo3—O9^{i}	2.3643 (14)
Mo2—O8	1.6961 (16)	Mo4—O11	1.7054 (16)
Mo2—O7	1.7115 (17)	Mo4—O13	1.7081 (16)
Mo2—O6	1.9164 (15)	Mo4—O10	1.8737 (14)
Mo2—O10	1.9270 (15)	Mo4—O12	2.0262 (14)
Mo2—O2^{i}	2.2735 (15)	Mo4—O9	2.2920 (14)
Mo2—O9	2.4668 (14)	Mo4—O3^{i}	2.3282 (14)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O12^{ii}	0.86	1.93	2.780 (2)	172
N2—H2···O7^{iii}	0.86	2.02	2.846 (3)	160
N3—H3···O13^{iv}	0.86	1.92	2.756 (2)	164
N4—H4···O4	0.86	1.95	2.781 (3)	162

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2458).

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Liu, B., Li, C.-B., Shi, Z. & Li, Y.-W. (2006). *Acta Cryst.* E**62**, m2764–m2766.

  doi:10.1107/S1600536807030735

supplementary materials

Acta Cryst. (2007). E**63**, m2002 [**doi:10.1107/S1600536807030735**]

Tetrabenzimidazolium hexacosaoxidooctamolybdate(VI)

W. Zhi, Z.-G. Zong, J. Gong and C.-B. Li

Comment

Recently, much attention has focused on the intriguing structure of polyoxometalate (POM) clusters. To extend our recent work, where $[Mo_8O_{26}]^{4-}$ units are hydrogen bonded to organic cations forming three-dimensional networks (Liu *et al.*, 2006), we obtained the title compound, (I), by using sodium molybadate and benzimidazole as the starting materials.

Compound (I) is composed of centrosymmetric $[Mo_8O_{26}]^{4-}$ anions and protonated benzimidazole cations as shown in Fig. 1. The $[Mo_8O_{26}]^{4-}$ anion is constructed from eight MoO_6 units. The four unique Mo atoms exhibit distorted octahedral geometries, with the Mo—O distances in the expected range (Table 1). Bond-valence sum calculations (Brown & Altermatt, 1985) indicate oxidation states of 5.93–5.98 for Mo, in agreement with the expected value of 6. By way of N—H···O hydrogen bonds (Table 2) between the organic cations and the anions, a three-dimensional network is formed (Fig. 2).

Experimental

$NaMo_4{\cdot}2H_2O$ (0.85 g 3.5 mmol) and benzimidazole (0.18 g 1.5 mmol) were dissolved in 10 ml water by vigorous stirring. 2 ml (1.5 *M*) HCl solution was added to the resulting suspension and stirred for 1 h. Then the suspension was sealed in a 20-ml Teflon-lined reactor with heating for 4 d at 438 K. After slowly cooling to room temperature over a period of 15 h, light yellow blocks of (I) were recovered.

Refinement

The H atoms were positioned geometrically (C—H = 0.93 Å, N—H = 0.86 Å) and refined as ridimg with U_{iso} = 1.2U_{eq}(carrier).

Figures

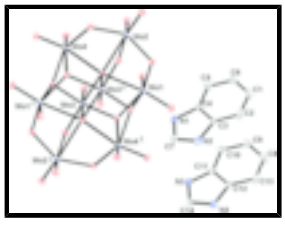


Fig. 1. he molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level (H atoms omitted for clarity). Symmetry code: i) 1 − *x*, 1 − *y*, 1 − *z*.].

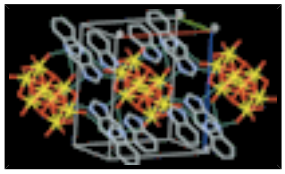


Fig. 2. The packing diagram of (I) with dashed lines indicating the N···O contacts for the hydrogen bonds.

Tetrabenzimidazolium hexacosaoxidooctamolybdate

Crystal data

$(C_7H_7N_2)_4[Mo_8O_{26}]$	$Z = 1$
$M_r = 1660.10$	$F_{000} = 796$
Triclinic, $P\overline{1}$	$D_x = 2.567$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.229$ (1) Å	Cell parameters from 3244 reflections
$b = 10.225$ (1) Å	$\theta = 1.7$–26.8º
$c = 11.966$ (2) Å	$\mu = 2.36$ mm^{-1}
$\alpha = 84.510$ (2)º	$T = 291$ (2) K
$\beta = 83.261$ (2)º	Block, light yellow
$\gamma = 73.667$ (1)º	0.24 × 0.17 × 0.12 mm
$V = 1073.8$ (2) Å^3	

Data collection

Bruker APEXII CCD diffractometer	4166 independent reflections
Radiation source: fine-focus sealed tube	3739 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 291(2)$ K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -11 \rightarrow 11$
$T_{min} = 0.630$, $T_{max} = 0.750$	$k = -12 \rightarrow 12$
9089 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.017$	$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2 + 0.4475P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.041$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
4166 reflections	$\Delta\rho_{min} = -0.39$ e Å^{-3}
317 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00508 (19)
Secondary atom site location: difference Fourier map	

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, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.59927 (2)	0.532347 (18)	0.263597 (15)	0.02152 (6)
Mo2	0.62950 (2)	0.795167 (18)	0.385504 (16)	0.02363 (6)
Mo3	0.323131 (19)	0.507990 (17)	0.449853 (15)	0.01839 (6)
Mo4	0.351949 (19)	0.767969 (17)	0.572658 (15)	0.02083 (6)
O1	0.19688 (17)	0.61523 (16)	0.36842 (13)	0.0302 (4)
O2	0.24277 (16)	0.37504 (15)	0.49679 (13)	0.0251 (3)
O3	0.47554 (16)	0.41241 (14)	0.33915 (12)	0.0210 (3)
O4	0.46161 (18)	0.63830 (16)	0.18737 (13)	0.0319 (4)
O5	0.71731 (18)	0.42950 (16)	0.16958 (13)	0.0335 (4)
O6	0.71645 (16)	0.65156 (15)	0.28576 (13)	0.0257 (3)
O7	0.49682 (19)	0.89710 (17)	0.30301 (14)	0.0369 (4)
O8	0.76798 (19)	0.87561 (17)	0.38041 (15)	0.0367 (4)
O9	0.48639 (15)	0.62428 (14)	0.43799 (12)	0.0205 (3)
O10	0.51772 (16)	0.83940 (14)	0.52966 (13)	0.0259 (3)
O11	0.21769 (18)	0.86259 (16)	0.48887 (15)	0.0331 (4)
O12	0.27027 (15)	0.60199 (14)	0.59189 (12)	0.0203 (3)
O13	0.29207 (18)	0.82691 (15)	0.70370 (13)	0.0305 (4)
N1	0.0343 (2)	0.2892 (2)	0.33685 (17)	0.0311 (4)
H1	−0.0577	0.3301	0.3599	0.037*
N2	0.2501 (2)	0.1370 (2)	0.32224 (17)	0.0361 (5)
H2	0.3204	0.0629	0.3343	0.043*
N3	−0.0228 (2)	0.9984 (2)	0.21433 (16)	0.0322 (5)
H3	−0.1129	1.0390	0.2419	0.039*
N4	0.1923 (2)	0.8467 (2)	0.19049 (17)	0.0361 (5)
H4	0.2642	0.7732	0.2001	0.043*
C1	0.3472 (4)	0.3758 (4)	0.0935 (2)	0.0571 (9)
H1A	0.4219	0.3912	0.0387	0.069*
C2	0.3790 (3)	0.2587 (3)	0.1625 (2)	0.0467 (7)
H2A	0.4734	0.1949	0.1568	0.056*
C3	0.2617 (3)	0.2403 (2)	0.2418 (2)	0.0301 (5)
C4	0.1232 (3)	0.3380 (2)	0.2503 (2)	0.0295 (5)
C5	0.0927 (3)	0.4568 (3)	0.1810 (2)	0.0459 (7)
H5	−0.0004	0.5225	0.1874	0.055*

C6	0.2088 (4)	0.4715 (3)	0.1025 (3)	0.0578 (8)
H6	0.1931	0.5493	0.0533	0.069*
C7	0.1140 (3)	0.1698 (3)	0.3779 (2)	0.0340 (5)
H7	0.0796	0.1168	0.4370	0.041*
C8	0.2702 (4)	1.0854 (3)	−0.0449 (2)	0.0535 (8)
H8	0.3405	1.1001	−0.1036	0.064*
C9	0.1329 (4)	1.1830 (3)	−0.0289 (2)	0.0513 (8)
H9	0.1144	1.2618	−0.0770	0.062*
C10	0.0226 (3)	1.1684 (3)	0.0553 (2)	0.0405 (6)
H10	−0.0696	1.2346	0.0657	0.049*
C11	0.0576 (3)	1.0483 (2)	0.12367 (19)	0.0281 (5)
C12	0.1963 (3)	0.9507 (2)	0.1081 (2)	0.0298 (5)
C13	0.3060 (3)	0.9675 (3)	0.0231 (2)	0.0449 (7)
H13	0.3990	0.9023	0.0127	0.054*
C14	0.0600 (3)	0.8788 (3)	0.2520 (2)	0.0368 (6)
H14	0.0301	0.8251	0.3125	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02068 (10)	0.01992 (10)	0.02009 (10)	−0.00139 (7)	0.00210 (7)	0.00032 (7)
Mo2	0.02158 (10)	0.01685 (10)	0.02935 (11)	−0.00353 (7)	0.00360 (8)	0.00124 (7)
Mo3	0.01566 (9)	0.01633 (10)	0.02133 (10)	−0.00196 (7)	−0.00036 (7)	−0.00102 (7)
Mo4	0.01792 (10)	0.01417 (10)	0.02733 (11)	−0.00122 (7)	0.00287 (7)	−0.00206 (7)
O1	0.0263 (8)	0.0277 (9)	0.0334 (9)	−0.0019 (7)	−0.0066 (7)	0.0022 (7)
O2	0.0219 (8)	0.0219 (8)	0.0313 (9)	−0.0065 (6)	0.0000 (6)	−0.0013 (6)
O3	0.0204 (7)	0.0180 (7)	0.0224 (8)	−0.0020 (6)	0.0008 (6)	−0.0038 (6)
O4	0.0282 (8)	0.0321 (9)	0.0299 (9)	−0.0013 (7)	−0.0023 (7)	0.0032 (7)
O5	0.0335 (9)	0.0305 (9)	0.0293 (9)	−0.0004 (7)	0.0067 (7)	−0.0027 (7)
O6	0.0225 (8)	0.0229 (8)	0.0282 (8)	−0.0048 (6)	0.0062 (6)	0.0001 (6)
O7	0.0344 (9)	0.0287 (9)	0.0376 (10)	0.0034 (7)	0.0013 (8)	0.0041 (7)
O8	0.0301 (9)	0.0274 (9)	0.0518 (11)	−0.0116 (7)	0.0069 (8)	−0.0008 (8)
O9	0.0195 (7)	0.0168 (7)	0.0230 (8)	−0.0026 (6)	0.0005 (6)	−0.0009 (6)
O10	0.0250 (8)	0.0197 (8)	0.0329 (9)	−0.0075 (6)	0.0035 (6)	−0.0053 (6)
O11	0.0285 (9)	0.0235 (8)	0.0448 (10)	−0.0034 (7)	−0.0065 (7)	0.0023 (7)
O12	0.0181 (7)	0.0167 (7)	0.0235 (8)	−0.0023 (6)	0.0026 (6)	−0.0008 (6)
O13	0.0304 (8)	0.0226 (8)	0.0334 (9)	−0.0007 (7)	0.0054 (7)	−0.0059 (7)
N1	0.0201 (9)	0.0316 (11)	0.0385 (12)	−0.0027 (8)	0.0014 (8)	−0.0048 (9)
N2	0.0284 (11)	0.0328 (11)	0.0419 (13)	0.0038 (9)	−0.0098 (9)	−0.0062 (9)
N3	0.0224 (10)	0.0354 (11)	0.0325 (11)	0.0012 (8)	0.0022 (8)	−0.0050 (9)
N4	0.0257 (10)	0.0314 (11)	0.0413 (12)	0.0059 (9)	−0.0022 (9)	0.0033 (9)
C1	0.063 (2)	0.087 (2)	0.0351 (16)	−0.047 (2)	0.0136 (14)	−0.0132 (16)
C2	0.0305 (14)	0.071 (2)	0.0411 (16)	−0.0146 (14)	0.0042 (12)	−0.0237 (15)
C3	0.0251 (12)	0.0352 (13)	0.0305 (13)	−0.0062 (10)	−0.0026 (10)	−0.0119 (10)
C4	0.0283 (12)	0.0278 (12)	0.0331 (13)	−0.0078 (10)	−0.0028 (10)	−0.0053 (10)
C5	0.0525 (17)	0.0352 (15)	0.0475 (17)	−0.0080 (13)	−0.0071 (14)	0.0014 (12)
C6	0.080 (2)	0.0532 (19)	0.0459 (18)	−0.0311 (18)	−0.0052 (17)	0.0080 (14)
C7	0.0297 (13)	0.0355 (14)	0.0367 (14)	−0.0080 (11)	−0.0056 (10)	−0.0012 (11)

C8	0.0538 (19)	0.078 (2)	0.0345 (16)	−0.0326 (17)	0.0006 (14)	0.0053 (15)
C9	0.071 (2)	0.0465 (17)	0.0429 (17)	−0.0272 (16)	−0.0195 (15)	0.0151 (13)
C10	0.0483 (16)	0.0296 (13)	0.0423 (16)	−0.0039 (12)	−0.0174 (13)	0.0002 (11)
C11	0.0267 (12)	0.0267 (12)	0.0288 (12)	−0.0019 (10)	−0.0053 (10)	−0.0044 (9)
C12	0.0253 (12)	0.0310 (13)	0.0308 (13)	−0.0035 (10)	−0.0019 (9)	−0.0042 (10)
C13	0.0311 (14)	0.0618 (19)	0.0375 (15)	−0.0079 (13)	0.0073 (12)	−0.0097 (13)
C14	0.0311 (13)	0.0394 (14)	0.0342 (14)	−0.0040 (11)	−0.0008 (11)	0.0051 (11)

Geometric parameters (Å, °)

Mo1—O5	1.6890 (15)	N2—C7	1.320 (3)
Mo1—O4	1.7113 (16)	N2—C3	1.378 (3)
Mo1—O6	1.8964 (15)	N2—H2	0.8600
Mo1—O3	1.9896 (14)	N3—C14	1.316 (3)
Mo1—O12^{i}	2.3304 (14)	N3—C11	1.381 (3)
Mo1—O9	2.3744 (14)	N3—H3	0.8600
Mo2—O8	1.6961 (16)	N4—C14	1.322 (3)
Mo2—O7	1.7115 (17)	N4—C12	1.384 (3)
Mo2—O6	1.9164 (15)	N4—H4	0.8600
Mo2—O10	1.9270 (15)	C1—C2	1.368 (4)
Mo2—O2^{i}	2.2735 (15)	C1—C6	1.374 (5)
Mo2—O9	2.4668 (14)	C1—H1A	0.9300
Mo3—O1	1.6890 (15)	C2—C3	1.395 (3)
Mo3—O2	1.7426 (14)	C2—H2A	0.9300
Mo3—O3	1.9302 (14)	C3—C4	1.383 (3)
Mo3—O12	1.9761 (14)	C4—C5	1.383 (3)
Mo3—O9	2.1516 (14)	C5—C6	1.371 (4)
Mo3—O9^{i}	2.3643 (14)	C5—H5	0.9300
Mo4—O11	1.7054 (16)	C6—H6	0.9300
Mo4—O13	1.7081 (16)	C7—H7	0.9300
Mo4—O10	1.8737 (14)	C8—C13	1.370 (4)
Mo4—O12	2.0262 (14)	C8—C9	1.381 (4)
Mo4—O9	2.2920 (14)	C8—H8	0.9300
Mo4—O3^{i}	2.3282 (14)	C9—C10	1.375 (4)
O2—Mo2^{i}	2.2735 (15)	C9—H9	0.9300
O3—Mo4^{i}	2.3282 (14)	C10—C11	1.388 (3)
O9—Mo3^{i}	2.3643 (14)	C10—H10	0.9300
O12—Mo1^{i}	2.3304 (14)	C11—C12	1.389 (3)
N1—C7	1.318 (3)	C12—C13	1.382 (3)
N1—C4	1.388 (3)	C13—H13	0.9300
N1—H1	0.8600	C14—H14	0.9300
O5—Mo1—O4	105.31 (8)	Mo4—O9—Mo3^{i}	97.32 (5)
O5—Mo1—O6	101.08 (7)	Mo3—O9—Mo1	90.98 (5)
O4—Mo1—O6	101.29 (7)	Mo4—O9—Mo1	163.40 (7)
O5—Mo1—O3	102.06 (7)	Mo3^{i}—O9—Mo1	96.91 (5)
O4—Mo1—O3	97.15 (7)	Mo3—O9—Mo2	164.34 (7)
O6—Mo1—O3	145.27 (6)	Mo4—O9—Mo2	86.20 (5)

O5—Mo1—O12^{i}	88.99 (7)	Mo3^{i}—O9—Mo2	91.36 (5)
O4—Mo1—O12^{i}	163.57 (6)	Mo1—O9—Mo2	84.96 (4)
O6—Mo1—O12^{i}	83.44 (6)	Mo4—O10—Mo2	117.78 (8)
O3—Mo1—O12^{i}	71.47 (5)	Mo3—O12—Mo4	108.37 (6)
O5—Mo1—O9	160.57 (7)	Mo3—O12—Mo1^{i}	110.62 (6)
O4—Mo1—O9	93.95 (6)	Mo4—O12—Mo1^{i}	103.56 (6)
O6—Mo1—O9	77.16 (5)	C7—N1—C4	108.8 (2)
O3—Mo1—O9	72.42 (5)	C7—N1—H1	125.6
O12^{i}—Mo1—O9	71.58 (5)	C4—N1—H1	125.6
O8—Mo2—O7	105.57 (8)	C7—N2—C3	109.2 (2)
O8—Mo2—O6	102.83 (7)	C7—N2—H2	125.4
O7—Mo2—O6	98.70 (8)	C3—N2—H2	125.4
O8—Mo2—O10	102.88 (8)	C14—N3—C11	109.3 (2)
O7—Mo2—O10	97.41 (7)	C14—N3—H3	125.4
O6—Mo2—O10	144.59 (6)	C11—N3—H3	125.4
O8—Mo2—O2^{i}	90.59 (7)	C14—N4—C12	109.0 (2)
O7—Mo2—O2^{i}	163.82 (7)	C14—N4—H4	125.5
O6—Mo2—O2^{i}	77.95 (6)	C12—N4—H4	125.5
O10—Mo2—O2^{i}	77.84 (6)	C2—C1—C6	122.2 (3)
O8—Mo2—O9	160.08 (7)	C2—C1—H1A	118.9
O7—Mo2—O9	94.33 (7)	C6—C1—H1A	118.9
O6—Mo2—O9	74.51 (5)	C1—C2—C3	116.3 (3)
O10—Mo2—O9	73.00 (5)	C1—C2—H2A	121.9
O2^{i}—Mo2—O9	69.50 (5)	C3—C2—H2A	121.9
O1—Mo3—O2	105.03 (7)	N2—C3—C4	106.3 (2)
O1—Mo3—O3	102.28 (7)	N2—C3—C2	132.9 (2)
O2—Mo3—O3	97.94 (6)	C4—C3—C2	120.9 (2)
O1—Mo3—O12	100.13 (7)	C5—C4—C3	122.5 (2)
O2—Mo3—O12	95.90 (6)	C5—C4—N1	131.4 (2)
O3—Mo3—O12	149.52 (6)	C3—C4—N1	106.2 (2)
O1—Mo3—O9	98.18 (7)	C6—C5—C4	115.6 (3)
O2—Mo3—O9	156.70 (6)	C6—C5—H5	122.2
O3—Mo3—O9	78.80 (6)	C4—C5—H5	122.2
O12—Mo3—O9	77.78 (5)	C5—C6—C1	122.6 (3)
O1—Mo3—O9^{i}	173.99 (6)	C5—C6—H6	118.7
O2—Mo3—O9^{i}	80.91 (6)	C1—C6—H6	118.7
O3—Mo3—O9^{i}	77.44 (6)	N1—C7—N2	109.5 (2)
O12—Mo3—O9^{i}	78.12 (5)	N1—C7—H7	125.2
O9—Mo3—O9^{i}	75.85 (6)	N2—C7—H7	125.2
O11—Mo4—O13	104.73 (8)	C13—C8—C9	122.0 (3)
O11—Mo4—O10	103.28 (7)	C13—C8—H8	119.0
O13—Mo4—O10	102.11 (7)	C9—C8—H8	119.0
O11—Mo4—O12	95.20 (7)	C10—C9—C8	122.5 (3)
O13—Mo4—O12	98.38 (7)	C10—C9—H9	118.7
O10—Mo4—O12	147.72 (6)	C8—C9—H9	118.7

O11—Mo4—O9	96.07 (7)	C9—C10—C11	115.7 (3)
O13—Mo4—O9	158.39 (6)	C9—C10—H10	122.2
O10—Mo4—O9	78.28 (6)	C11—C10—H10	122.2
O12—Mo4—O9	73.59 (5)	N3—C11—C10	132.2 (2)
O11—Mo4—O3^{i}	163.43 (7)	N3—C11—C12	106.1 (2)
O13—Mo4—O3^{i}	86.70 (6)	C10—C11—C12	121.7 (2)
O10—Mo4—O3^{i}	85.57 (6)	C13—C12—N4	132.2 (2)
O12—Mo4—O3^{i}	70.93 (5)	C13—C12—C11	121.7 (2)
O9—Mo4—O3^{i}	71.74 (5)	N4—C12—C11	106.1 (2)
Mo3—O2—Mo2^{i}	118.22 (7)	C8—C13—C12	116.3 (3)
Mo3—O3—Mo1	111.04 (7)	C8—C13—H13	121.8
Mo3—O3—Mo4^{i}	110.02 (6)	C12—C13—H13	121.8
Mo1—O3—Mo4^{i}	104.82 (6)	N3—C14—N4	109.6 (2)
Mo1—O6—Mo2	118.12 (7)	N3—C14—H14	125.2
Mo3—O9—Mo4	93.78 (5)	N4—C14—H14	125.2
Mo3—O9—Mo3^{i}	104.15 (6)		
O1—Mo3—O2—Mo2^{i}	−178.62 (8)	O5—Mo1—O9—Mo2	101.4 (2)
O3—Mo3—O2—Mo2^{i}	76.32 (8)	O4—Mo1—O9—Mo2	−86.24 (6)
O12—Mo3—O2—Mo2^{i}	−76.45 (8)	O6—Mo1—O9—Mo2	14.45 (5)
O9—Mo3—O2—Mo2^{i}	−3.8 (2)	O3—Mo1—O9—Mo2	177.50 (6)
O9^{i}—Mo3—O2—Mo2^{i}	0.49 (7)	O12^{i}—Mo1—O9—Mo2	101.73 (5)
O1—Mo3—O3—Mo1	73.20 (9)	O8—Mo2—O9—Mo3	−174.9 (2)
O2—Mo3—O3—Mo1	−179.44 (7)	O7—Mo2—O9—Mo3	7.9 (3)
O12—Mo3—O3—Mo1	−63.25 (14)	O6—Mo2—O9—Mo3	−89.9 (3)
O9—Mo3—O3—Mo1	−22.85 (7)	O10—Mo2—O9—Mo3	104.3 (3)
O9^{i}—Mo3—O3—Mo1	−100.65 (7)	O2^{i}—Mo2—O9—Mo3	−172.6 (3)
O1—Mo3—O3—Mo4^{i}	−171.21 (7)	O8—Mo2—O9—Mo4	94.7 (2)
O2—Mo3—O3—Mo4^{i}	−63.85 (8)	O7—Mo2—O9—Mo4	−82.57 (7)
O12—Mo3—O3—Mo4^{i}	52.35 (13)	O6—Mo2—O9—Mo4	179.59 (6)
O9—Mo3—O3—Mo4^{i}	92.74 (6)	O10—Mo2—O9—Mo4	13.86 (6)
O9^{i}—Mo3—O3—Mo4^{i}	14.94 (5)	O2^{i}—Mo2—O9—Mo4	96.92 (6)
O5—Mo1—O3—Mo3	−178.06 (7)	O8—Mo2—O9—Mo3^{i}	−2.6 (2)
O4—Mo1—O3—Mo3	−70.67 (9)	O7—Mo2—O9—Mo3^{i}	−179.81 (6)
O6—Mo1—O3—Mo3	51.17 (13)	O6—Mo2—O9—Mo3^{i}	82.35 (6)
O12^{i}—Mo1—O3—Mo3	97.14 (7)	O10—Mo2—O9—Mo3^{i}	−83.38 (6)
O9—Mo1—O3—Mo3	21.23 (6)	O2^{i}—Mo2—O9—Mo3^{i}	−0.32 (5)
O5—Mo1—O3—Mo4^{i}	63.17 (8)	O8—Mo2—O9—Mo1	−99.4 (2)
O4—Mo1—O3—Mo4^{i}	170.56 (7)	O7—Mo2—O9—Mo1	83.37 (7)
O6—Mo1—O3—Mo4^{i}	−67.60 (12)	O6—Mo2—O9—Mo1	−14.47 (5)
O12^{i}—Mo1—O3—Mo4^{i}	−21.63 (5)	O10—Mo2—O9—Mo1	179.80 (6)
O9—Mo1—O3—Mo4^{i}	−97.54 (6)	O2^{i}—Mo2—O9—Mo1	−97.14 (5)
O5—Mo1—O6—Mo2	178.51 (8)	O11—Mo4—O10—Mo2	−73.22 (10)
O4—Mo1—O6—Mo2	70.24 (10)	O13—Mo4—O10—Mo2	178.25 (8)

O3—Mo1—O6—Mo2	−50.49 (15)	O12—Mo4—O10—Mo2	50.01 (16)
O12^{i}—Mo1—O6—Mo2	−93.82 (8)	O9—Mo4—O10—Mo2	20.33 (8)
O9—Mo1—O6—Mo2	−21.28 (8)	O3^{i}—Mo4—O10—Mo2	92.58 (8)
O8—Mo2—O6—Mo1	−179.67 (9)	O8—Mo2—O10—Mo4	−179.12 (9)
O7—Mo2—O6—Mo1	−71.42 (10)	O7—Mo2—O10—Mo4	72.98 (10)
O10—Mo2—O6—Mo1	44.70 (16)	O6—Mo2—O10—Mo4	−43.50 (16)
O2^{i}—Mo2—O6—Mo1	92.49 (8)	O2^{i}—Mo2—O10—Mo4	−91.32 (9)
O9—Mo2—O6—Mo1	20.70 (7)	O9—Mo2—O10—Mo4	−19.30 (7)
O1—Mo3—O9—Mo4	80.78 (7)	O1—Mo3—O12—Mo4	−74.85 (8)
O2—Mo3—O9—Mo4	−94.17 (15)	O2—Mo3—O12—Mo4	178.69 (7)
O3—Mo3—O9—Mo4	−178.23 (6)	O3—Mo3—O12—Mo4	62.00 (13)
O12—Mo3—O9—Mo4	−17.88 (5)	O9—Mo3—O12—Mo4	21.42 (6)
O9^{i}—Mo3—O9—Mo4	−98.53 (6)	O9^{i}—Mo3—O12—Mo4	99.29 (7)
O1—Mo3—O9—Mo3^{i}	179.31 (7)	O1—Mo3—O12—Mo1^{i}	172.27 (7)
O2—Mo3—O9—Mo3^{i}	4.35 (18)	O2—Mo3—O12—Mo1^{i}	65.81 (7)
O3—Mo3—O9—Mo3^{i}	−79.70 (6)	O3—Mo3—O12—Mo1^{i}	−50.88 (13)
O12—Mo3—O9—Mo3^{i}	80.65 (6)	O9—Mo3—O12—Mo1^{i}	−91.46 (6)
O9^{i}—Mo3—O9—Mo3^{i}	0.0	O9^{i}—Mo3—O12—Mo1^{i}	−13.59 (5)
O1—Mo3—O9—Mo1	−83.31 (7)	O11—Mo4—O12—Mo3	74.37 (8)
O2—Mo3—O9—Mo1	101.73 (15)	O13—Mo4—O12—Mo3	−179.88 (7)
O3—Mo3—O9—Mo1	17.68 (5)	O10—Mo4—O12—Mo3	−50.81 (14)
O12—Mo3—O9—Mo1	178.03 (6)	O9—Mo4—O12—Mo3	−20.44 (6)
O9^{i}—Mo3—O9—Mo1	97.38 (6)	O3^{i}—Mo4—O12—Mo3	−96.34 (7)
O1—Mo3—O9—Mo2	−8.6 (3)	O11—Mo4—O12—Mo1^{i}	−168.13 (7)
O2—Mo3—O9—Mo2	176.4 (2)	O13—Mo4—O12—Mo1^{i}	−62.38 (7)
O3—Mo3—O9—Mo2	92.4 (3)	O10—Mo4—O12—Mo1^{i}	66.70 (13)
O12—Mo3—O9—Mo2	−107.3 (3)	O9—Mo4—O12—Mo1^{i}	97.06 (6)
O9^{i}—Mo3—O9—Mo2	172.1 (3)	O3^{i}—Mo4—O12—Mo1^{i}	21.17 (5)
O11—Mo4—O9—Mo3	−75.88 (7)	C6—C1—C2—C3	1.0 (4)
O13—Mo4—O9—Mo3	88.44 (18)	C7—N2—C3—C4	−0.9 (3)
O10—Mo4—O9—Mo3	−178.24 (6)	C7—N2—C3—C2	179.0 (3)
O12—Mo4—O9—Mo3	17.77 (5)	C1—C2—C3—N2	178.7 (3)
O3^{i}—Mo4—O9—Mo3	92.60 (6)	C1—C2—C3—C4	−1.4 (4)
O11—Mo4—O9—Mo3^{i}	179.31 (6)	N2—C3—C4—C5	−179.3 (2)
O13—Mo4—O9—Mo3^{i}	−16.37 (19)	C2—C3—C4—C5	0.7 (4)
O10—Mo4—O9—Mo3^{i}	76.96 (6)	N2—C3—C4—N1	0.5 (2)
O12—Mo4—O9—Mo3^{i}	−87.04 (6)	C2—C3—C4—N1	−179.4 (2)
O3^{i}—Mo4—O9—Mo3^{i}	−12.20 (4)	C7—N1—C4—C5	179.8 (3)
O11—Mo4—O9—Mo1	30.5 (2)	C7—N1—C4—C3	0.0 (3)
O13—Mo4—O9—Mo1	−165.2 (2)	C3—C4—C5—C6	0.4 (4)
O10—Mo4—O9—Mo1	−71.9 (2)	N1—C4—C5—C6	−179.4 (3)
O12—Mo4—O9—Mo1	124.1 (2)	C4—C5—C6—C1	−0.8 (5)
O3^{i}—Mo4—O9—Mo1	−161.0 (2)	C2—C1—C6—C5	0.1 (5)
O11—Mo4—O9—Mo2	88.42 (6)	C4—N1—C7—N2	−0.6 (3)

O13—Mo4—O9—Mo2	−107.26 (17)	C3—N2—C7—N1	0.9 (3)
O10—Mo4—O9—Mo2	−13.93 (6)	C13—C8—C9—C10	−0.6 (5)
O12—Mo4—O9—Mo2	−177.93 (6)	C8—C9—C10—C11	−0.2 (4)
O3^{i}—Mo4—O9—Mo2	−103.09 (5)	C14—N3—C11—C10	−179.7 (3)
O5—Mo1—O9—Mo3	−93.8 (2)	C14—N3—C11—C12	0.0 (3)
O4—Mo1—O9—Mo3	78.61 (7)	C9—C10—C11—N3	−179.7 (3)
O6—Mo1—O9—Mo3	179.31 (6)	C9—C10—C11—C12	0.7 (4)
O3—Mo1—O9—Mo3	−17.65 (5)	C14—N4—C12—C13	−179.6 (3)
O12^{i}—Mo1—O9—Mo3	−93.42 (5)	C14—N4—C12—C11	0.0 (3)
O5—Mo1—O9—Mo4	159.5 (2)	N3—C11—C12—C13	179.7 (2)
O4—Mo1—O9—Mo4	−28.1 (2)	C10—C11—C12—C13	−0.6 (4)
O6—Mo1—O9—Mo4	72.6 (2)	N3—C11—C12—N4	0.0 (3)
O3—Mo1—O9—Mo4	−124.4 (2)	C10—C11—C12—N4	179.7 (2)
O12^{i}—Mo1—O9—Mo4	159.8 (2)	C9—C8—C13—C12	0.7 (4)
O5—Mo1—O9—Mo3^{i}	10.6 (2)	N4—C12—C13—C8	179.5 (3)
O4—Mo1—O9—Mo3^{i}	−177.01 (6)	C11—C12—C13—C8	−0.1 (4)
O6—Mo1—O9—Mo3^{i}	−76.31 (6)	C11—N3—C14—N4	0.0 (3)
O3—Mo1—O9—Mo3^{i}	86.73 (6)	C12—N4—C14—N3	−0.1 (3)
O12^{i}—Mo1—O9—Mo3^{i}	10.96 (4)		

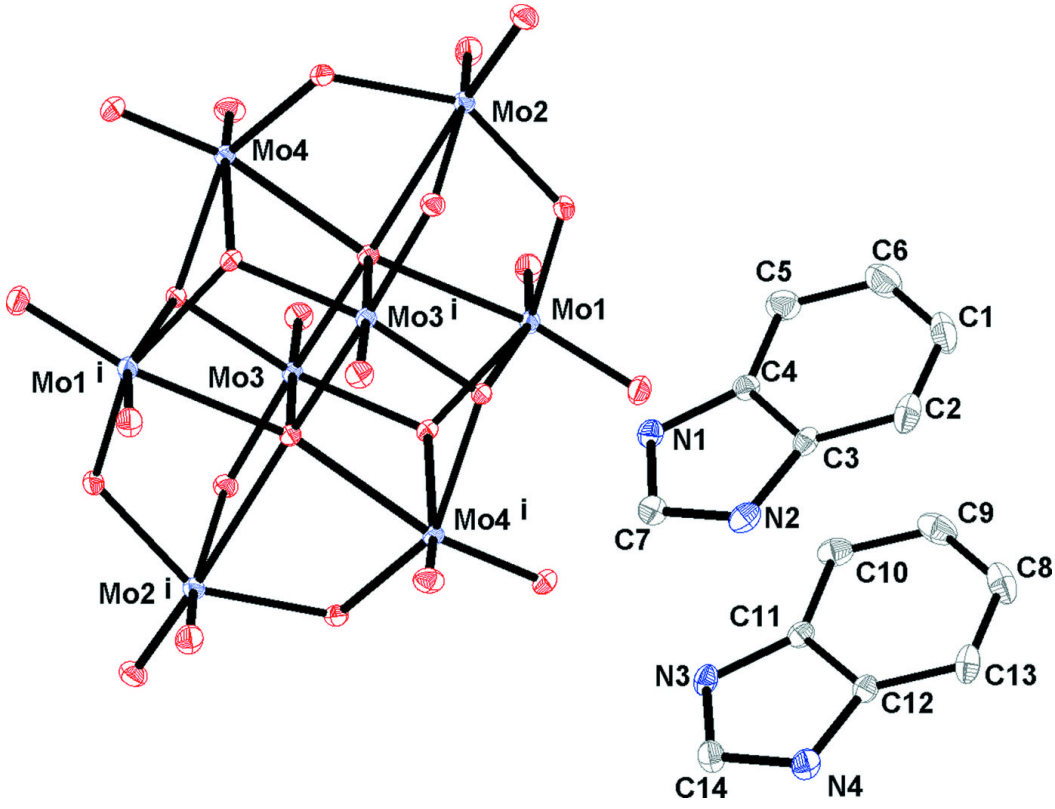
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O12^{ii}	0.86	1.93	2.780 (2)	172
N2—H2···O7^{iii}	0.86	2.02	2.846 (3)	160
N3—H3···O13^{iv}	0.86	1.92	2.756 (2)	164
N4—H4···O4	0.86	1.95	2.781 (3)	162

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

Fig. 1


Mo2
Mo4
Mo3 i
Mo1
Mo1 i
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Mo4 i
Mo2 i
C5
C6
C1
C4
C2
N1
C3
C7
N2
C9
C10
C8
C11
N3
C13
C12
C14
N4

Fig. 2