

Tetrakis(1-ethyl-3-methylimidazolium) β -hexacosaoxidooctamolybdate

Shiwei Lin,^{a,b} Weilin Chen,^a Zhiming Zhang,^a Wenli Liu^a
and Enbo Wang^{a*}

^aKey Laboratory of Polyoxometalate Science of the Ministry of Education, Department of Chemistry, Northeast Normal University, Changchun, Jilin 130024, People's Republic of China, and ^bDepartment of Chemistry, Changchun Normal University, Changchun, Jilin 130024, People's Republic of China
Correspondence e-mail: wangenbo@public.cc.jl.cn

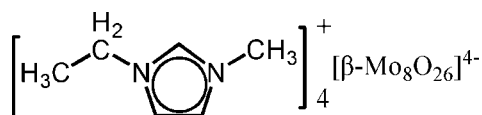
Received 9 March 2008; accepted 27 May 2008

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.026; wR factor = 0.071; data-to-parameter ratio = 19.1.

The title compound, $(\text{C}_6\text{H}_{11}\text{N}_2)_4[\text{Mo}_8\text{O}_{26}]$ or $(\text{emim})_4[\beta\text{-Mo}_8\text{O}_{26}]$ (emim is 1-ethyl-3-methylimidazolium), was obtained from the ionic liquid $[\text{emim}]\text{BF}_4$. The asymmetric unit contains two $[\text{emim}]^+$ cations and one-half of the $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ tetraanion, which occupies a special position on an inversion centre. The $\beta\text{-}[\text{Mo}_8\text{O}_{26}]^{4-}$ tetraanion features eight distorted MoO_6 coordination octahedra linked together through bridging O atoms.

Related literature

For related literature, see: Aguado *et al.* (2005).



Experimental

Crystal data

$(\text{C}_6\text{H}_{11}\text{N}_2)_4[\text{Mo}_8\text{O}_{26}]$
 $M_r = 1628.19$
Orthorhombic, $Pbca$
 $a = 15.6338$ (6) Å
 $b = 16.9231$ (6) Å
 $c = 17.9380$ (7) Å

$V = 4745.9$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 2.13$ mm⁻¹
 $T = 296$ (2) K
 $0.24 \times 0.22 \times 0.21$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.629$, $T_{\max} = 0.663$

27619 measured reflections
5677 independent reflections
4568 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.070$
 $S = 1.04$
5677 reflections

298 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.66$ e Å⁻³
 $\Delta\rho_{\min} = -0.61$ e Å⁻³

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Postdoctoral Station Foundation of the Ministry of Education of China (No. 20060200002) and the Testing Foundation of Northeast Normal University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2072).

References

- Aguado, R., Pedrosa, M. R. & Arnáiz, F. J. (2005). *Z. Anorg. Allg. Chem.* **631**, 1995–1999.
Bruker (1997). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2008). E64, m954 [doi:10.1107/S1600536808015936]

Tetrakis(1-ethyl-3-methylimidazolium) β -hexacosaoxidooctamolybdate

S. Lin, W. Chen, Z. Zhang, W. Liu and E. Wang

Comment

The asymmetric unit of the title compound, [emim]₄[β -Mo₈O₂₆] (emim = 1-ethyl-3-methylimidazolium), (1), contains two [emim]⁺ cations and one half of the [β -Mo₈O₂₆]⁴⁻ anion, which occupies a special position in the inversion centre (Fig. 1). The anion has eight molybdenum atoms with distorted octahedral coordinations and 26 oxygen atoms which fall into four categories, *i.e.* terminal, μ_2 -, μ_3 - and μ_5 -bridging atoms. The geometry of tetra-anion is characterized by a wide range of Mo—O distances varying from 1.683 (2) Å for one of the terminal bonds (Mo1—O5) to 2.510 (2) Å for one of the bonds involving 5-coordinated oxygen atom (Mo4—O1). The geometry of the anion is similar to that observed in previous structures, *e.g.* Aguado *et al.* (2005).

Experimental

A mixture of sodium molybdate, Na₂MoO₄·2H₂O (0.3 mmol), and ionic liquid 1-ethyl-3-methylimidazolium tetrafluoroborate, [emim]BF₄ (8 ml) was stirred at 170 °C for 24 h in air. The resulting clear solution was filtered and left at room temperature for 3 days. The colourless block crystals were filtered off, washed with cool distilled water and dried in a desiccator at room temperature.

Refinement

All H-atoms were included in the refinement in a riding model approximation with $U_{\text{iso}}=1.2U_{\text{eq}}$ (C) for aromatic (C—H 0.93 Å) and methylene (C—H 0.97 Å) H-atoms, $U_{\text{iso}} = 1.5U_{\text{eq}}$ (C) for methyl H-atoms (C—H 0.96 Å).

Figures

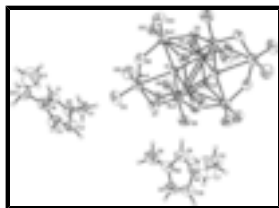


Fig. 1. The cations and anion in the structure of the title compound. Thermal displacement ellipsoids are drawn at 30% probability level; H atoms are shown as small circles of arbitrary radius. The unlabeled atoms are related to their symmetry related counterparts by the (2 - x, -y, 1 - z) transformation.

Tetrakis(1-ethyl-3-methylimidazolium) β -hexacosaoxidooctamolybdate

Crystal data

(C₆H₁₁N₂)₄[Mo₈O₂₆]

$M_r = 1628.19$

$F_{000} = 3152$

$D_x = 2.279 \text{ Mg m}^{-3}$

supplementary materials

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 15.6338$ (6) Å

$b = 16.9231$ (6) Å

$c = 17.9380$ (7) Å

$V = 4745.9$ (3) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 27619 reflections

$\theta = 2.1$ – 28.3°

$\mu = 2.13$ mm⁻¹

$T = 296$ (2) K

Block, colourless

$0.24 \times 0.22 \times 0.21$ mm

Data collection

Bruker APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0.01 pixels mm⁻¹

$T = 296$ (2) K

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.629$, $T_{\max} = 0.663$

27619 measured reflections

5677 independent reflections

4568 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\text{max}} = 28.3^\circ$

$\theta_{\text{min}} = 2.1^\circ$

$h = -20 \rightarrow 14$

$k = -22 \rightarrow 22$

$l = -20 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.070$

$S = 1.04$

5677 reflections

298 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 3.2572P]$

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

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.66$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.61$ e Å⁻³

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.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.919942 (16)	0.001136 (16)	0.429035 (13)	0.03331 (7)
Mo2	1.078576 (18)	-0.118127 (18)	0.408995 (16)	0.04219 (8)
Mo3	1.048047 (18)	0.149211 (16)	0.436802 (15)	0.03908 (8)
Mo4	1.212312 (18)	0.030332 (19)	0.417154 (16)	0.04370 (8)
O1	1.05538 (13)	0.01203 (12)	0.44123 (11)	0.0345 (4)
O2	0.95510 (13)	-0.10911 (12)	0.43767 (11)	0.0385 (5)
O3	0.93026 (12)	0.11030 (12)	0.46027 (12)	0.0372 (5)
O4	0.81442 (14)	-0.01285 (13)	0.45821 (12)	0.0436 (5)
O5	0.91133 (15)	0.01019 (14)	0.33592 (12)	0.0490 (6)
O6	1.19117 (14)	-0.08062 (14)	0.42354 (12)	0.0456 (5)
O7	1.16753 (14)	0.13133 (13)	0.44644 (13)	0.0470 (5)
O8	1.03691 (16)	0.15263 (15)	0.34304 (13)	0.0546 (6)
O9	1.04161 (16)	0.24423 (14)	0.46552 (15)	0.0579 (6)
O10	1.20274 (17)	0.04091 (16)	0.32313 (14)	0.0617 (7)
O11	1.31854 (16)	0.03784 (17)	0.43493 (15)	0.0641 (7)
O12	1.06442 (16)	-0.10397 (17)	0.31615 (14)	0.0631 (7)
O13	1.09196 (18)	-0.21648 (16)	0.41944 (16)	0.0644 (7)
N1	1.5874 (2)	0.0109 (2)	0.35763 (17)	0.0562 (8)
N2	1.2156 (2)	0.28339 (19)	0.6125 (2)	0.0619 (8)
N3	1.48859 (19)	0.09691 (18)	0.33891 (15)	0.0513 (7)
N4	1.1552 (2)	0.23736 (18)	0.71109 (19)	0.0599 (8)
C1	1.5344 (2)	0.0403 (2)	0.3079 (2)	0.0551 (9)
H1A	1.5299	0.0237	0.2587	0.066*
C2	1.5147 (3)	0.1051 (3)	0.4115 (2)	0.0647 (11)
H2A	1.4941	0.1413	0.4461	0.078*
C3	1.4202 (3)	0.1411 (3)	0.3036 (2)	0.0635 (11)
H3A	1.4151	0.1251	0.2525	0.095*
H3B	1.4330	0.1966	0.3058	0.095*
H3C	1.3674	0.1310	0.3292	0.095*
C4	1.5752 (3)	0.0513 (3)	0.4230 (2)	0.0702 (12)
H4A	1.6042	0.0427	0.4675	0.084*
C5	1.6476 (3)	-0.0559 (3)	0.3444 (2)	0.0754 (13)
H5A	1.6575	-0.0614	0.2913	0.091*
H5B	1.7020	-0.0443	0.3680	0.091*
C6	1.1661 (2)	0.2273 (2)	0.6388 (2)	0.0596 (10)
H6A	1.1423	0.1866	0.6107	0.072*
C7	1.1062 (3)	0.1867 (2)	0.7611 (2)	0.0708 (11)
H7A	1.0702	0.2194	0.7925	0.085*
H7B	1.0693	0.1528	0.7318	0.085*
C8	1.2360 (3)	0.3315 (3)	0.6708 (3)	0.0759 (13)
H8A	1.2698	0.3766	0.6681	0.091*
C9	1.2417 (3)	0.2920 (3)	0.5351 (3)	0.0872 (15)
H9A	1.2183	0.2494	0.5062	0.131*
H9B	1.3030	0.2909	0.5320	0.131*
H9C	1.2210	0.3414	0.5160	0.131*

supplementary materials

C10	1.1999 (3)	0.3033 (3)	0.7313 (3)	0.0806 (14)
H10A	1.2041	0.3243	0.7790	0.097*
C11	1.6139 (4)	-0.1312 (3)	0.3742 (4)	0.115 (2)
H11A	1.6539	-0.1729	0.3640	0.172*
H11B	1.5602	-0.1430	0.3508	0.172*
H11C	1.6059	-0.1265	0.4270	0.172*
C12	1.1603 (4)	0.1381 (4)	0.8080 (3)	0.118 (2)
H12A	1.1252	0.1054	0.8392	0.177*
H12B	1.1955	0.1714	0.8385	0.177*
H12C	1.1959	0.1054	0.7772	0.177*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.03345 (14)	0.03938 (14)	0.02710 (13)	-0.00539 (11)	-0.00506 (9)	-0.00023 (10)
Mo2	0.04347 (17)	0.04620 (16)	0.03690 (15)	-0.00116 (13)	0.00226 (11)	-0.00868 (12)
Mo3	0.04015 (16)	0.03777 (14)	0.03932 (15)	-0.00623 (12)	-0.00155 (11)	0.00535 (11)
Mo4	0.03562 (15)	0.05413 (18)	0.04136 (16)	-0.00311 (13)	0.00151 (11)	0.00798 (13)
O1	0.0351 (11)	0.0394 (11)	0.0289 (10)	-0.0030 (9)	-0.0017 (8)	0.0008 (8)
O2	0.0416 (12)	0.0382 (11)	0.0357 (11)	-0.0072 (9)	-0.0048 (9)	-0.0064 (9)
O3	0.0364 (11)	0.0367 (11)	0.0385 (12)	0.0003 (9)	-0.0014 (9)	0.0001 (9)
O4	0.0362 (12)	0.0553 (13)	0.0392 (12)	-0.0066 (10)	-0.0057 (9)	0.0017 (10)
O5	0.0548 (14)	0.0600 (15)	0.0322 (12)	-0.0031 (11)	-0.0100 (10)	0.0004 (10)
O6	0.0393 (12)	0.0519 (13)	0.0456 (13)	0.0050 (11)	0.0061 (9)	0.0019 (10)
O7	0.0383 (12)	0.0488 (13)	0.0540 (14)	-0.0123 (10)	-0.0017 (10)	0.0065 (11)
O8	0.0538 (15)	0.0651 (16)	0.0450 (14)	-0.0102 (12)	-0.0027 (11)	0.0139 (12)
O9	0.0633 (16)	0.0413 (13)	0.0689 (17)	-0.0040 (12)	-0.0009 (13)	-0.0003 (12)
O10	0.0706 (18)	0.0699 (17)	0.0446 (14)	0.0031 (14)	0.0051 (12)	0.0117 (12)
O11	0.0398 (14)	0.083 (2)	0.0689 (17)	-0.0065 (13)	0.0012 (12)	0.0155 (14)
O12	0.0624 (17)	0.089 (2)	0.0376 (13)	-0.0027 (14)	0.0031 (11)	-0.0157 (13)
O13	0.0703 (18)	0.0481 (15)	0.0748 (19)	0.0002 (13)	0.0033 (14)	-0.0174 (13)
N1	0.0514 (18)	0.072 (2)	0.0457 (17)	-0.0034 (15)	-0.0109 (14)	-0.0023 (15)
N2	0.0543 (19)	0.0583 (19)	0.073 (2)	-0.0168 (16)	-0.0121 (16)	0.0017 (17)
N3	0.0540 (18)	0.0637 (18)	0.0363 (15)	-0.0049 (15)	0.0002 (13)	-0.0049 (14)
N4	0.0528 (18)	0.0563 (18)	0.070 (2)	-0.0094 (15)	-0.0104 (16)	-0.0100 (16)
C1	0.055 (2)	0.072 (2)	0.0383 (18)	0.0020 (19)	-0.0073 (16)	-0.0061 (17)
C2	0.069 (3)	0.085 (3)	0.041 (2)	-0.002 (2)	0.0005 (18)	-0.013 (2)
C3	0.063 (3)	0.078 (3)	0.049 (2)	0.016 (2)	0.0034 (18)	-0.004 (2)
C4	0.077 (3)	0.098 (4)	0.036 (2)	-0.010 (3)	-0.0116 (18)	-0.004 (2)
C5	0.064 (3)	0.097 (3)	0.065 (3)	0.016 (3)	-0.024 (2)	-0.011 (2)
C6	0.061 (2)	0.055 (2)	0.064 (2)	-0.0145 (19)	-0.0155 (19)	-0.0003 (19)
C7	0.063 (3)	0.073 (3)	0.076 (3)	-0.004 (2)	-0.004 (2)	-0.004 (2)
C8	0.069 (3)	0.058 (2)	0.100 (4)	-0.023 (2)	-0.006 (3)	-0.017 (2)
C9	0.088 (3)	0.097 (4)	0.077 (3)	-0.038 (3)	-0.011 (3)	0.015 (3)
C10	0.076 (3)	0.079 (3)	0.087 (3)	-0.017 (2)	-0.003 (3)	-0.033 (3)
C11	0.124 (5)	0.087 (4)	0.134 (5)	0.016 (4)	-0.007 (4)	0.019 (4)
C12	0.099 (4)	0.143 (5)	0.112 (5)	0.022 (4)	-0.008 (4)	0.047 (4)

Geometric parameters (Å, °)

Mo1—O5	1.683 (2)	N2—C8	1.363 (5)
Mo1—O4	1.747 (2)	N2—C9	1.454 (6)
Mo1—O3	1.937 (2)	N3—C1	1.319 (5)
Mo1—O2	1.951 (2)	N3—C2	1.372 (4)
Mo1—O1	2.137 (2)	N3—C3	1.451 (5)
Mo1—O1 ⁱ	2.370 (2)	N4—C6	1.319 (5)
Mo1—Mo3	3.2109 (4)	N4—C10	1.366 (5)
Mo1—Mo2	3.2177 (4)	N4—C7	1.458 (5)
Mo2—O13	1.688 (3)	C1—H1A	0.9300
Mo2—O12	1.697 (3)	C2—C4	1.328 (6)
Mo2—O6	1.889 (2)	C2—H2A	0.9300
Mo2—O2	2.004 (2)	C3—H3A	0.9600
Mo2—O1	2.306 (2)	C3—H3B	0.9600
Mo2—O3 ⁱ	2.353 (2)	C3—H3C	0.9600
Mo3—O9	1.692 (2)	C4—H4A	0.9300
Mo3—O8	1.692 (2)	C5—C11	1.478 (7)
Mo3—O7	1.900 (2)	C5—H5A	0.9700
Mo3—O3	2.000 (2)	C5—H5B	0.9700
Mo3—O1	2.326 (2)	C6—H6A	0.9300
Mo3—O2 ⁱ	2.352 (2)	C7—C12	1.448 (6)
Mo4—O11	1.696 (3)	C7—H7A	0.9700
Mo4—O10	1.703 (2)	C7—H7B	0.9700
Mo4—O6	1.910 (2)	C8—C10	1.313 (6)
Mo4—O7	1.920 (2)	C8—H8A	0.9300
Mo4—O4 ⁱ	2.294 (2)	C9—H9A	0.9600
Mo4—O1	2.510 (2)	C9—H9B	0.9600
O1—Mo1 ⁱ	2.370 (2)	C9—H9C	0.9600
O2—Mo3 ⁱ	2.352 (2)	C10—H10A	0.9300
O3—Mo2 ⁱ	2.353 (2)	C11—H11A	0.9600
O4—Mo4 ⁱ	2.294 (2)	C11—H11B	0.9600
N1—C1	1.315 (4)	C11—H11C	0.9600
N1—C4	1.371 (5)	C12—H12A	0.9600
N1—C5	1.490 (5)	C12—H12B	0.9600
N2—C6	1.313 (5)	C12—H12C	0.9600
O5—Mo1—O4	103.54 (11)	Mo1—O1—Mo2	92.74 (7)
O5—Mo1—O3	101.94 (10)	Mo1—O1—Mo3	91.94 (8)
O4—Mo1—O3	96.96 (9)	Mo2—O1—Mo3	162.30 (10)
O5—Mo1—O2	100.86 (10)	Mo1—O1—Mo1 ⁱ	104.70 (8)
O4—Mo1—O2	96.47 (10)	Mo2—O1—Mo1 ⁱ	97.52 (7)
O3—Mo1—O2	149.93 (8)	Mo3—O1—Mo1 ⁱ	97.78 (7)
O5—Mo1—O1	99.96 (10)	Mo1—O1—Mo4	164.07 (10)
O4—Mo1—O1	156.48 (9)	Mo2—O1—Mo4	85.47 (7)
O3—Mo1—O1	78.79 (8)	Mo3—O1—Mo4	85.36 (6)

supplementary materials

O2—Mo1—O1	78.18 (8)	Mo1 ⁱ —O1—Mo4	91.23 (7)
O5—Mo1—O1 ⁱ	175.21 (10)	Mo1—O2—Mo2	108.90 (10)
O4—Mo1—O1 ⁱ	81.19 (8)	Mo1—O2—Mo3 ⁱ	110.24 (9)
O3—Mo1—O1 ⁱ	78.00 (8)	Mo2—O2—Mo3 ⁱ	104.12 (9)
O2—Mo1—O1 ⁱ	77.66 (8)	Mo1—O3—Mo3	109.25 (10)
O1—Mo1—O1 ⁱ	75.30 (8)	Mo1—O3—Mo2 ⁱ	109.70 (9)
O5—Mo1—Mo3	91.25 (8)	Mo3—O3—Mo2 ⁱ	104.20 (9)
O4—Mo1—Mo3	132.98 (7)	Mo1—O4—Mo4 ⁱ	118.79 (10)
O3—Mo1—Mo3	36.03 (6)	Mo2—O6—Mo4	118.90 (12)
O2—Mo1—Mo3	124.55 (6)	Mo3—O7—Mo4	118.38 (11)
O1—Mo1—Mo3	46.38 (5)	C1—N1—C4	107.6 (4)
O1 ⁱ —Mo1—Mo3	85.94 (5)	C1—N1—C5	125.2 (3)
O5—Mo1—Mo2	90.45 (8)	C4—N1—C5	127.1 (3)
O4—Mo1—Mo2	132.56 (7)	C6—N2—C8	107.1 (4)
O3—Mo1—Mo2	124.49 (6)	C6—N2—C9	125.5 (3)
O2—Mo1—Mo2	36.09 (6)	C8—N2—C9	127.4 (4)
O1—Mo1—Mo2	45.71 (5)	C1—N3—C2	108.1 (3)
O1 ⁱ —Mo1—Mo2	85.71 (5)	C1—N3—C3	126.2 (3)
Mo3—Mo1—Mo2	90.780 (10)	C2—N3—C3	125.6 (3)
O13—Mo2—O12	105.32 (14)	C6—N4—C10	107.5 (4)
O13—Mo2—O6	101.56 (12)	C6—N4—C7	126.6 (3)
O12—Mo2—O6	102.10 (11)	C10—N4—C7	125.9 (4)
O13—Mo2—O2	99.56 (11)	N1—C1—N3	109.4 (3)
O12—Mo2—O2	96.63 (10)	N1—C1—H1A	125.3
O6—Mo2—O2	146.78 (9)	N3—C1—H1A	125.3
O13—Mo2—O1	159.00 (11)	C4—C2—N3	106.9 (4)
O12—Mo2—O1	95.20 (11)	C4—C2—H2A	126.6
O6—Mo2—O1	77.94 (9)	N3—C2—H2A	126.6
O2—Mo2—O1	73.22 (8)	N3—C3—H3A	109.5
O13—Mo2—O3 ⁱ	87.26 (11)	N3—C3—H3B	109.5
O12—Mo2—O3 ⁱ	164.26 (11)	H3A—C3—H3B	109.5
O6—Mo2—O3 ⁱ	84.15 (8)	N3—C3—H3C	109.5
O2—Mo2—O3 ⁱ	71.54 (7)	H3A—C3—H3C	109.5
O1—Mo2—O3 ⁱ	71.77 (7)	H3B—C3—H3C	109.5
O13—Mo2—Mo1	134.56 (10)	C2—C4—N1	108.0 (3)
O12—Mo2—Mo1	85.45 (9)	C2—C4—H4A	126.0
O6—Mo2—Mo1	119.47 (7)	N1—C4—H4A	126.0
O2—Mo2—Mo1	35.01 (6)	C11—C5—N1	111.8 (4)
O1—Mo2—Mo1	41.55 (5)	C11—C5—H5A	109.3
O3 ⁱ —Mo2—Mo1	78.94 (5)	N1—C5—H5A	109.3
O9—Mo3—O8	105.31 (13)	C11—C5—H5B	109.3
O9—Mo3—O7	100.50 (11)	N1—C5—H5B	109.3
O8—Mo3—O7	101.36 (11)	H5A—C5—H5B	107.9
O9—Mo3—O3	101.19 (11)	N2—C6—N4	109.6 (3)
O8—Mo3—O3	97.23 (10)	N2—C6—H6A	125.2
O7—Mo3—O3	146.45 (9)	N4—C6—H6A	125.2

O9—Mo3—O1	160.30 (10)	C12—C7—N4	112.6 (4)
O8—Mo3—O1	94.20 (10)	C12—C7—H7A	109.1
O7—Mo3—O1	77.85 (8)	N4—C7—H7A	109.1
O3—Mo3—O1	73.12 (8)	C12—C7—H7B	109.1
O9—Mo3—O2 ⁱ	88.94 (10)	N4—C7—H7B	109.1
O8—Mo3—O2 ⁱ	163.60 (10)	H7A—C7—H7B	107.8
O7—Mo3—O2 ⁱ	83.54 (9)	C10—C8—N2	108.5 (4)
O3—Mo3—O2 ⁱ	71.60 (8)	C10—C8—H8A	125.8
O1—Mo3—O2 ⁱ	71.36 (7)	N2—C8—H8A	125.8
O9—Mo3—Mo1	135.89 (9)	N2—C9—H9A	109.5
O8—Mo3—Mo1	85.38 (8)	N2—C9—H9B	109.5
O7—Mo3—Mo1	119.53 (7)	H9A—C9—H9B	109.5
O3—Mo3—Mo1	34.72 (6)	N2—C9—H9C	109.5
O1—Mo3—Mo1	41.69 (5)	H9A—C9—H9C	109.5
O2 ⁱ —Mo3—Mo1	78.66 (5)	H9B—C9—H9C	109.5
O11—Mo4—O10	105.33 (13)	C8—C10—N4	107.3 (4)
O11—Mo4—O6	103.41 (12)	C8—C10—H10A	126.3
O10—Mo4—O6	98.49 (11)	N4—C10—H10A	126.3
O11—Mo4—O7	103.82 (12)	C5—C11—H11A	109.5
O10—Mo4—O7	98.36 (11)	C5—C11—H11B	109.5
O6—Mo4—O7	142.70 (9)	H11A—C11—H11B	109.5
O11—Mo4—O4 ⁱ	90.28 (10)	C5—C11—H11C	109.5
O10—Mo4—O4 ⁱ	164.39 (11)	H11A—C11—H11C	109.5
O6—Mo4—O4 ⁱ	77.48 (8)	H11B—C11—H11C	109.5
O7—Mo4—O4 ⁱ	77.39 (9)	C7—C12—H12A	109.5
O11—Mo4—O1	159.07 (10)	C7—C12—H12B	109.5
O10—Mo4—O1	95.59 (10)	H12A—C12—H12B	109.5
O6—Mo4—O1	72.50 (8)	C7—C12—H12C	109.5
O7—Mo4—O1	72.93 (8)	H12A—C12—H12C	109.5
O4 ⁱ —Mo4—O1	68.80 (7)	H12B—C12—H12C	109.5

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

Fig. 1

