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Tetraimidazolium bis(imidazole-κN)di-μ_4-oxido-tetra-μ_3-oxido-hexa-μ_2-oxido-tetradecaoxidooctamolybdate(VI) monohydrate

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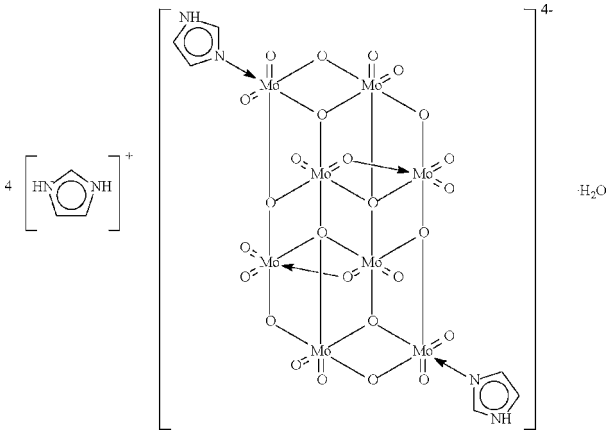
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.019; wR factor = 0.050; data-to-parameter ratio = 11.9.

The anion of the title salt, $(C_3H_5N_2)_4[Mo_8O_{26}(C_3H_4N_2)_2]\cdot H_2O$, is a centrosymmetric Mo_8O_{26} species in which two Mo atoms are each coordinated by an imidazole heterocycle. The anion is made up of two $MoNO_5$ and six MoO_6 edge-sharing octahedra. The cations interact with the tetraanion to give rise to a three-dimensional hydrogen-bonded network. The asymmetric unit features three cations, two of which lie on centers of inversion; the solvent water molecule is disordered with respect to one of these two cations.

Related literature

For tetrakis(dimethylammonium) bis(imidazole)octamolybdate trihydrate (which has the same anion), see Martían-Zarza *et al.* (1993).



Experimental

Crystal data

$(C_3H_5N_2)_4[Mo_8O_{26}(C_3H_4N_2)_2]\cdot H_2O$
M_r = 1614.06
Triclinic, $P\overline{1}$
a = 9.9723 (6) Å
b = 10.9891 (7) Å
c = 11.0273 (7) Å
α = 73.411 (1)°
β = 68.510 (1)°
γ = 66.702 (1)°
V = 1018.7 (1) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 2.49 mm^{-1}
T = 295 (2) K
0.32 × 0.20 × 0.18 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.509, T_{max} = 0.663
8712 measured reflections
4001 independent reflections
3818 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.050
S = 1.03
4001 reflections
335 parameters
84 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2n···O7^{i}	0.85	2.10	2.926 (3)	164
N3—H3n···O1^{ii}	0.85	2.24	2.889 (4)	134
N4—H4n···O8^{iii}	0.85	2.25	3.094 (4)	178
N5—H5n···O9^{iv}	0.85	2.25	2.866 (4)	130
N6—H6n···O1w^{v}	0.85	1.72	2.556 (7)	169
N7—H7n···O3	0.85	2.05	2.760 (9)	140
N8—H8n···O3^{vi}	0.85	2.08	2.766 (9)	138
O1w—H1w1···O5	0.85	2.22	3.010 (4)	154
O1w—H1w2···O9^{iv}	0.85	2.13	2.866 (4)	144

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2468).

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supplementary materials

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Tetraimidazolium bis(imidazole-κN)di-μ_4-oxido-tetra-μ_3-oxido-hexa-μ_2-oxido-tetradecaoxidooctamolybdate(VI) monohydrate

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

Comment

The tetraanion of the title salt, tetrakis(imidazolium) bis(imidazole)-octamolybdate monohydrate, was previously identified as tetrakis(dimethylammonium) bis(imidazole)-octamolybdate trihydrate (Martían-Zarza *et al.*, 1993). The anion is made up of two $MoNO_5$ and six MoO_6 edge-sharing octahedra as two of the molybdenum atoms are coordinated by the neutral *N*-heterocycles. In the present salt, two of the three cations of the asymmetric unit are disordered over different centers-of-inversion. The cations and tetraanion interact through hydrogen bonds to give rise to a three-dimensional network.

Experimental

Sodium molybdate dihydrate, $Na_2MoO_4{\cdot}2H_2O$ (0.65 g, 2.7 mmol) and ammonium acetate (0.15 g, 2 mmol) were dissolved in 30 ml water. Imidazole (0.10 g, 1.5 mmol) was added and the pH of the mixture adjusted to about 3.8 by the addition of acetic acid. The solution was heated at 333 K for about 12 h. It was then set aside for the growth of faint yellow crystals.

Refinement

Two imidazolium cations are disordered about centers-of-inversion. For these two five-membered rings, the 1,2-related distances were restrained to 1.35±0.01 Å and the 1,3-related ones to 2.18±0.01 Å. The rings were also restrained to be almost planar. The rings are necessarily of half-occupancy only; one of the rings is also disordered with respect to a water molecule. (The presence of water in the compound was confirmed by an infrared spectrum.) The O1w atom occupies the same position as the N5 atom of the ring; the temperature factors of the two atoms were restrained to be identical. Additionally, the anisotropic displacement parameters of all disordered atoms were restrained to be nearly isotropic.

The carbon- and nitrogen-boud hydrogen atoms were placed at calculated positions (C–H 0.95 and N–H 0.86 Å), and were included in the refinement in the riding model approximation, with U(H) set to 1.2 times U_{eq}(C,*N*). The hydrogen atoms of the half-occupancy water molecule were placed in chemically sensible positions on the basis of hydrogen bonds but were not refined; their temperature factors were set to 1.5 times U_{eq}(O).

Figures

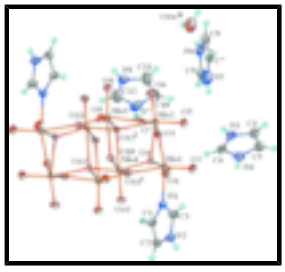


Fig. 1. **Figure 1**. Thermal ellipsoid plot of $4[C_3H_5N_2]^+ [(C_3H_4N_2)_2O_{26}Mo_8]^{4-}\cdot H_2O$. The tetraanion lies about a center-of-inversion, and the unlabeled atoms are related to the labeled ones by (i = 2 – *x*, 1 – *y*, 1 – *z*). The water molecule is disordered with respect to one of the disordered cations that lie on an inversion site; the symmetry-related equivalent is shown (and labeled with an ii superscript).

Tetraimidazolium bis(imidazole-κ*N*)di-μ$_4$-oxido-tetra-μ$_3$-oxido-hexa-μ$_2$-oxido-\ tetradecaoxido-octamolybdate(VI) monohydrate

Crystal data

$(C_3H_5N_2)_4[Mo_8O_{26}(C_3H_4N_2)_2]\cdot H_2O$	$Z = 1$
$M_r = 1614.06$	$F_{000} = 774$
Triclinic, $P\overline{1}$	$D_x = 2.631$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
$a = 9.9723$ (6) Å	Cell parameters from 6997 reflections
$b = 10.9891$ (7) Å	θ = 2.6–26.1°
$c = 11.0273$ (7) Å	μ = 2.49 mm^{-1}
α = 73.411 (1)°	$T = 295$ (2) K
β = 68.510 (1)°	Block, yellow
γ = 66.702 (1)°	0.32 × 0.20 × 0.18 mm
$V = 1018.7$ (1) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	4001 independent reflections
Radiation source: fine-focus sealed tube	3818 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 295(2)$ K	$\theta_{max} = 26.1°$
φ and ω scans	$\theta_{min} = 2.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12\rightarrow12$
$T_{min} = 0.509$, $T_{max} = 0.663$	$k = -13\rightarrow13$
8712 measured reflections	$l = -13\rightarrow13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.019$	H-atom parameters constrained

$wR(F^2) = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 1.3243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4001 reflections	$\Delta\rho_{max} = 0.56$ e Å^{-3}
335 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
84 restraints	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: 0.0115 (4)

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.66852 (3)	0.39691 (3)	0.78701 (2)	0.02253 (8)	
Mo2	0.68776 (3)	0.69805 (2)	0.70065 (2)	0.02160 (8)	
Mo3	0.90982 (3)	0.78651 (2)	0.41399 (2)	0.02006 (7)	
Mo4	0.85357 (2)	0.48189 (2)	0.46941 (2)	0.01675 (7)	
O1	0.7212 (3)	0.2601 (2)	0.9016 (2)	0.0378 (5)	
O2	0.4752 (2)	0.4615 (3)	0.8447 (2)	0.0391 (5)	
O3	0.7318 (2)	0.5242 (2)	0.81780 (19)	0.0260 (4)	
O4	0.6743 (2)	0.54871 (19)	0.60330 (18)	0.0202 (4)	
O5	0.4939 (2)	0.7582 (2)	0.7651 (2)	0.0347 (5)	
O6	0.7564 (3)	0.7744 (2)	0.7672 (2)	0.0344 (5)	
O7	0.7015 (2)	0.80454 (19)	0.52633 (19)	0.0226 (4)	
O8	0.9465 (2)	0.8797 (2)	0.4910 (2)	0.0306 (5)	
O9	0.8773 (3)	0.8888 (2)	0.2709 (2)	0.0339 (5)	
O10	0.8527 (2)	0.62994 (19)	0.35338 (19)	0.0229 (4)	
O11	1.1133 (2)	0.66457 (19)	0.35825 (18)	0.0213 (4)	
O12	1.0724 (2)	0.39639 (18)	0.43058 (18)	0.0195 (4)	
O13	0.8008 (2)	0.3943 (2)	0.4009 (2)	0.0262 (4)	
O1w	0.3555 (4)	1.0254 (4)	0.8580 (3)	0.0679 (9)	0.50
H1w1	0.3986	0.9668	0.8068	0.102*	0.50
H1w2	0.2719	1.0777	0.8417	0.102*	0.50
N1	0.6259 (3)	0.2831 (2)	0.6778 (2)	0.0260 (5)	
N2	0.5113 (3)	0.2301 (3)	0.5761 (3)	0.0368 (6)	
H2n	0.4468	0.2366	0.5392	0.044*	
N3	0.1740 (3)	0.7977 (3)	0.8697 (3)	0.0374 (6)	
H3n	0.2508	0.7723	0.8983	0.045*	
N4	0.0393 (3)	0.8412 (3)	0.7417 (3)	0.0415 (7)	
H4n	0.0137	0.8491	0.6736	0.050*	
C1	0.7039 (3)	0.1532 (3)	0.6544 (3)	0.0318 (7)	
H1	0.7924	0.0974	0.6771	0.038*	
C2	0.6319 (4)	0.1197 (3)	0.5934 (3)	0.0348 (7)	
H2	0.6593	0.0372	0.5682	0.042*	
C3	0.5111 (4)	0.3266 (3)	0.6266 (4)	0.0359 (7)	
H3	0.4401	0.4126	0.6260	0.043*	
C4	0.0271 (4)	0.8470 (4)	0.9408 (3)	0.0416 (8)	
H4	−0.0082	0.8596	1.0284	0.050*	

C5	−0.0579 (4)	0.8742 (4)	0.8598 (4)	0.0424 (8)	
H5	−0.1636	0.9091	0.8810	0.051*	
C6	0.1799 (4)	0.7951 (4)	0.7497 (4)	0.0424 (8)	
H6	0.2679	0.7657	0.6821	0.051*	
N5	0.3555 (4)	1.0254 (4)	0.8580 (3)	0.0679 (9)	0.50
H5n	0.3196	0.9993	0.8148	0.081*	0.50
N6	0.4811 (8)	1.0259 (7)	0.9896 (8)	0.041 (2)	0.50
H6n	0.5364	0.9982	1.0414	0.049*	0.50
C7	0.3365 (6)	1.1560 (6)	0.8651 (5)	0.0470 (18)	0.50
H7	0.2792	1.2331	0.8211	0.056*	0.50
C8	0.4124 (7)	1.1557 (6)	0.9444 (6)	0.0411 (16)	0.50
H8	0.4167	1.2306	0.9642	0.049*	0.50
C9	0.4474 (7)	0.9492 (6)	0.9394 (6)	0.0488 (18)	0.50
H9	0.4814	0.8558	0.9567	0.059*	0.50
N7	0.9672 (17)	0.4876 (9)	0.9137 (14)	0.050 (3)	0.50
H7n	0.9340	0.4812	0.8557	0.060*	0.50
N8	1.1196 (12)	0.4892 (9)	1.0082 (13)	0.039 (2)	0.50
H8n	1.2000	0.4842	1.0223	0.047*	0.50
C10	0.8828 (10)	0.5156 (13)	1.0355 (17)	0.056 (4)	0.50
H10	0.7784	0.5313	1.0716	0.067*	0.50
C11	0.978 (2)	0.5166 (13)	1.0951 (8)	0.056 (4)	0.50
H11	0.9520	0.5331	1.1804	0.067*	0.50
C12	1.1117 (12)	0.4716 (7)	0.8992 (9)	0.053 (2)	0.50
H12	1.1931	0.4514	0.8240	0.064*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01925 (13)	0.03160 (14)	0.02000 (13)	−0.01112 (10)	−0.00580 (9)	−0.00471 (10)
Mo2	0.01642 (12)	0.02691 (14)	0.02408 (13)	−0.00400 (9)	−0.00577 (9)	−0.01252 (10)
Mo3	0.01810 (12)	0.01817 (13)	0.02476 (13)	−0.00365 (9)	−0.00905 (9)	−0.00422 (9)
Mo4	0.01450 (12)	0.01969 (12)	0.01785 (12)	−0.00303 (9)	−0.00688 (9)	−0.00644 (9)
O1	0.0425 (13)	0.0477 (14)	0.0291 (11)	−0.0227 (11)	−0.0158 (10)	0.0037 (10)
O2	0.0244 (11)	0.0538 (15)	0.0419 (13)	−0.0139 (10)	−0.0021 (10)	−0.0201 (11)
O3	0.0255 (10)	0.0355 (11)	0.0226 (10)	−0.0108 (9)	−0.0097 (8)	−0.0082 (8)
O4	0.0154 (9)	0.0242 (10)	0.0222 (9)	−0.0034 (7)	−0.0066 (7)	−0.0083 (8)
O5	0.0207 (10)	0.0409 (13)	0.0418 (13)	−0.0046 (9)	−0.0047 (9)	−0.0190 (10)
O6	0.0366 (12)	0.0411 (13)	0.0371 (12)	−0.0132 (10)	−0.0143 (10)	−0.0175 (10)
O7	0.0172 (9)	0.0214 (10)	0.0291 (10)	−0.0010 (7)	−0.0102 (8)	−0.0069 (8)
O8	0.0271 (11)	0.0294 (11)	0.0406 (12)	−0.0084 (9)	−0.0106 (9)	−0.0133 (9)
O9	0.0367 (12)	0.0287 (11)	0.0356 (12)	−0.0091 (9)	−0.0180 (10)	0.0035 (9)
O10	0.0234 (10)	0.0236 (10)	0.0233 (10)	−0.0050 (8)	−0.0100 (8)	−0.0055 (8)
O11	0.0181 (9)	0.0243 (10)	0.0214 (9)	−0.0056 (8)	−0.0069 (7)	−0.0038 (8)
O12	0.0157 (9)	0.0218 (9)	0.0208 (9)	−0.0029 (7)	−0.0061 (7)	−0.0070 (7)
O13	0.0267 (10)	0.0281 (11)	0.0299 (11)	−0.0073 (8)	−0.0120 (8)	−0.0110 (8)
O1w	0.0488 (19)	0.081 (3)	0.068 (2)	−0.0181 (17)	−0.0152 (16)	−0.0111 (19)
N1	0.0218 (12)	0.0279 (13)	0.0307 (13)	−0.0075 (10)	−0.0113 (10)	−0.0047 (10)
N2	0.0321 (14)	0.0407 (16)	0.0495 (17)	−0.0065 (12)	−0.0253 (13)	−0.0146 (13)

N3	0.0322 (14)	0.0409 (16)	0.0471 (17)	−0.0132 (12)	−0.0201 (13)	−0.0052 (13)
N4	0.0448 (17)	0.0482 (17)	0.0431 (17)	−0.0137 (14)	−0.0205 (14)	−0.0161 (14)
C1	0.0243 (15)	0.0256 (15)	0.0468 (19)	−0.0036 (12)	−0.0169 (14)	−0.0055 (13)
C2	0.0321 (16)	0.0300 (16)	0.0441 (19)	−0.0080 (13)	−0.0101 (14)	−0.0132 (14)
C3	0.0298 (16)	0.0322 (17)	0.052 (2)	−0.0018 (13)	−0.0240 (15)	−0.0125 (15)
C4	0.045 (2)	0.053 (2)	0.0300 (17)	−0.0220 (17)	−0.0085 (15)	−0.0072 (15)
C5	0.0315 (17)	0.050 (2)	0.050 (2)	−0.0112 (15)	−0.0137 (15)	−0.0144 (17)
C6	0.0357 (18)	0.048 (2)	0.048 (2)	−0.0140 (16)	−0.0065 (16)	−0.0211 (17)
N5	0.0488 (19)	0.081 (3)	0.068 (2)	−0.0181 (17)	−0.0152 (16)	−0.0111 (19)
N6	0.031 (4)	0.061 (6)	0.031 (3)	−0.016 (4)	−0.014 (3)	0.000 (4)
C7	0.034 (3)	0.058 (4)	0.036 (4)	−0.013 (3)	−0.004 (3)	0.001 (3)
C8	0.042 (4)	0.043 (4)	0.037 (3)	−0.020 (3)	0.000 (3)	−0.009 (3)
C9	0.050 (4)	0.043 (4)	0.049 (4)	−0.023 (3)	0.005 (3)	−0.017 (3)
N7	0.044 (5)	0.071 (5)	0.055 (6)	−0.024 (5)	−0.033 (5)	−0.006 (4)
N8	0.046 (5)	0.050 (4)	0.037 (5)	−0.021 (3)	−0.025 (4)	−0.006 (4)
C10	0.047 (5)	0.073 (6)	0.050 (8)	−0.030 (5)	0.004 (5)	−0.021 (6)
C11	0.051 (8)	0.092 (7)	0.030 (4)	−0.025 (6)	−0.010 (5)	−0.016 (4)
C12	0.045 (5)	0.087 (6)	0.034 (5)	−0.025 (4)	−0.014 (4)	−0.012 (4)

Geometric parameters (Å, °)

Mo1—O1	1.706 (2)	N3—C4	1.352 (5)
Mo1—O2	1.704 (2)	N3—H3n	0.8500
Mo1—O3	1.896 (2)	N4—C6	1.317 (5)
Mo1—O4	2.225 (2)	N4—C5	1.355 (5)
Mo1—O11^{i}	2.158 (2)	N4—H4n	0.8500
Mo1—N1	2.203 (2)	C1—C2	1.344 (4)
Mo2—O3	1.964 (2)	C1—H1	0.9300
Mo2—O4	2.269 (2)	C2—H2	0.9300
Mo2—O5	1.711 (2)	C3—H3	0.9300
Mo2—O6	1.699 (2)	C4—C5	1.344 (5)
Mo2—O7	1.932 (2)	C4—H4	0.9300
Mo2—O12^{i}	2.292 (2)	C5—H5	0.9300
Mo3—O7	1.954 (2)	C6—H6	0.9300
Mo3—O8	1.710 (2)	N6—C9	1.326 (8)
Mo3—O9	1.713 (2)	N6—C8	1.350 (7)
Mo3—O10	2.333 (2)	N6—H6n	0.8500
Mo3—O11	1.923 (2)	C7—C8	1.348 (7)
Mo3—O12^{i}	2.232 (2)	C7—H1w2	1.383 (7)
Mo4—O4	1.887 (2)	C7—H7	0.9300
Mo4—O10	1.759 (2)	C8—H8	0.9300
Mo4—O13	1.697 (2)	C9—H9	0.9300
Mo4—O11^{i}	2.146 (2)	N7—C12	1.334 (8)
Mo4—O12	1.937 (2)	N7—C10	1.346 (8)
Mo4—O12^{i}	2.402 (2)	N7—H7n	0.8500
O1w—H1w1	0.85	N8—C12	1.304 (10)
O1w—H1w2	0.85	N8—C11	1.355 (7)
N1—C3	1.320 (4)	N8—H8n	0.8500

N1—C1	1.377 (4)	C10—C11	1.345 (8)
N2—C3	1.331 (4)	C10—H10	0.9300
N2—C2	1.355 (4)	C11—H11	0.9300
N2—H2n	0.8500	C12—H12	0.9300
N3—C6	1.311 (4)		
O2—Mo1—O1	105.35 (12)	Mo3—O11—Mo4^{i}	105.25 (8)
O2—Mo1—O3	101.92 (10)	Mo3—O11—Mo1^{i}	151.90 (10)
O1—Mo1—O3	102.68 (10)	Mo4^{i}—O11—Mo1^{i}	102.74 (8)
O2—Mo1—O11^{i}	156.67 (10)	Mo4—O12—Mo3^{i}	101.61 (8)
O1—Mo1—O11^{i}	92.44 (10)	Mo4—O12—Mo2^{i}	154.72 (10)
O3—Mo1—O11^{i}	88.40 (8)	Mo3^{i}—O12—Mo2^{i}	92.40 (7)
O2—Mo1—N1	85.71 (10)	Mo4—O12—Mo4^{i}	103.28 (8)
O1—Mo1—N1	93.59 (10)	Mo3^{i}—O12—Mo4^{i}	96.46 (7)
O3—Mo1—N1	159.31 (9)	Mo2^{i}—O12—Mo4^{i}	95.84 (6)
O11^{i}—Mo1—N1	78.10 (8)	H1w1—O1w—H1w2	108.5
O2—Mo1—O4	92.04 (10)	C3—N1—C1	105.7 (3)
O1—Mo1—O4	162.36 (9)	C3—N1—Mo1	125.4 (2)
O3—Mo1—O4	75.99 (8)	C1—N1—Mo1	128.82 (19)
O11^{i}—Mo1—O4	69.99 (7)	C3—N2—C2	108.3 (3)
N1—Mo1—O4	84.62 (8)	C3—N2—H2n	125.8
O6—Mo2—O5	104.82 (11)	C2—N2—H2n	125.8
O6—Mo2—O7	101.14 (10)	C6—N3—C4	109.6 (3)
O5—Mo2—O7	97.72 (10)	C6—N3—H3n	125.2
O6—Mo2—O3	96.44 (10)	C4—N3—H3n	125.2
O5—Mo2—O3	99.64 (10)	C6—N4—C5	108.7 (3)
O7—Mo2—O3	151.18 (8)	C6—N4—H4n	125.6
O6—Mo2—O4	161.32 (9)	C5—N4—H4n	125.6
O5—Mo2—O4	92.62 (9)	C2—C1—N1	109.3 (3)
O7—Mo2—O4	82.71 (7)	C2—C1—H1	125.4
O3—Mo2—O4	73.70 (7)	N1—C1—H1	125.4
O6—Mo2—O12^{i}	90.97 (9)	C1—C2—N2	106.2 (3)
O5—Mo2—O12^{i}	163.23 (9)	C1—C2—H2	126.9
O7—Mo2—O12^{i}	73.26 (7)	N2—C2—H2	126.9
O3—Mo2—O12^{i}	83.85 (8)	N1—C3—N2	110.4 (3)
O4—Mo2—O12^{i}	72.49 (6)	N1—C3—H3	124.8
O8—Mo3—O9	105.42 (11)	N2—C3—H3	124.8
O8—Mo3—O11	98.40 (9)	C5—C4—N3	106.4 (3)
O9—Mo3—O11	104.31 (10)	C5—C4—H4	126.8
O8—Mo3—O7	96.66 (9)	N3—C4—H4	126.8
O9—Mo3—O7	101.11 (10)	C4—C5—N4	107.2 (3)
O11—Mo3—O7	145.67 (8)	C4—C5—H5	126.4
O8—Mo3—O12^{i}	96.90 (9)	N4—C5—H5	126.4
O9—Mo3—O12^{i}	157.63 (9)	N3—C6—N4	108.0 (3)
O11—Mo3—O12^{i}	73.43 (7)	N3—C6—H6	126.0
O7—Mo3—O12^{i}	74.25 (7)	N4—C6—H6	126.0

O8—Mo3—O10	167.65 (9)	C9—N6—C8	108.3 (5)
O9—Mo3—O10	86.49 (9)	C9—N6—H6n	125.8
O11—Mo3—O10	81.41 (7)	C8—N6—H6n	125.8
O7—Mo3—O10	77.49 (7)	C8—C7—H1w2	141.5 (5)
O12^{i}—Mo3—O10	71.14 (7)	C8—C7—H7	124.6
O13—Mo4—O10	103.16 (9)	H1w2—C7—H7	91.4
O13—Mo4—O4	102.77 (9)	C7—C8—N6	107.0 (5)
O10—Mo4—O4	100.08 (9)	C7—C8—H8	126.5
O13—Mo4—O12	103.31 (9)	N6—C8—H8	126.5
O10—Mo4—O12	97.93 (8)	N6—C9—H9	124.3
O4—Mo4—O12	143.81 (8)	C12—N7—C10	108.6 (6)
O13—Mo4—O11^{i}	97.64 (9)	C12—N7—H7n	125.7
O10—Mo4—O11^{i}	159.11 (8)	C10—N7—H7n	125.7
O4—Mo4—O11^{i}	76.84 (8)	C12—N8—C11	108.6 (5)
O12—Mo4—O11^{i}	75.20 (8)	C12—N8—H8n	125.7
O13—Mo4—O12^{i}	179.16 (8)	C11—N8—H8n	125.7
O10—Mo4—O12^{i}	77.66 (7)	C11—C10—N7	106.9 (5)
O4—Mo4—O12^{i}	76.85 (7)	C11—C10—H10	126.6
O12—Mo4—O12^{i}	76.72 (8)	N7—C10—H10	126.6
O11^{i}—Mo4—O12^{i}	81.54 (7)	C10—C11—N8	107.5 (5)
Mo1—O3—Mo2	113.87 (9)	C10—C11—H11	126.3
Mo4—O4—Mo1	109.41 (8)	N8—C11—H11	126.3
Mo4—O4—Mo2	113.61 (8)	N8—C12—N7	108.5 (6)
Mo1—O4—Mo2	92.07 (7)	N8—C12—H12	125.8
Mo2—O7—Mo3	114.32 (9)	N7—C12—H12	125.8
Mo4—O10—Mo3	114.67 (9)		
O2—Mo1—O3—Mo2	−70.97 (13)	O11—Mo3—O10—Mo4	77.92 (10)
O1—Mo1—O3—Mo2	−179.92 (11)	O7—Mo3—O10—Mo4	−74.82 (10)
O11^{i}—Mo1—O3—Mo2	87.93 (10)	O12^{i}—Mo3—O10—Mo4	2.59 (9)
N1—Mo1—O3—Mo2	39.0 (3)	O8—Mo3—O11—Mo4^{i}	79.29 (10)
O4—Mo1—O3—Mo2	18.16 (9)	O9—Mo3—O11—Mo4^{i}	−172.35 (9)
O6—Mo2—O3—Mo1	178.21 (11)	O7—Mo3—O11—Mo4^{i}	−35.79 (17)
O5—Mo2—O3—Mo1	71.93 (12)	O12^{i}—Mo3—O11—Mo4^{i}	−15.45 (7)
O7—Mo2—O3—Mo1	−54.3 (2)	O10—Mo3—O11—Mo4^{i}	−88.22 (8)
O4—Mo2—O3—Mo1	−18.00 (9)	O8—Mo3—O11—Mo1^{i}	−95.6 (2)
O12^{i}—Mo2—O3—Mo1	−91.51 (10)	O9—Mo3—O11—Mo1^{i}	12.7 (2)
O13—Mo4—O4—Mo1	87.02 (10)	O7—Mo3—O11—Mo1^{i}	149.31 (18)
O10—Mo4—O4—Mo1	−166.87 (8)	O12^{i}—Mo3—O11—Mo1^{i}	169.6 (2)
O12—Mo4—O4—Mo1	−48.16 (17)	O10—Mo3—O11—Mo1^{i}	96.9 (2)
O11^{i}—Mo4—O4—Mo1	−7.97 (7)	O13—Mo4—O12—Mo3^{i}	−79.55 (10)
O12^{i}—Mo4—O4—Mo1	−92.20 (8)	O10—Mo4—O12—Mo3^{i}	174.83 (8)
O13—Mo4—O4—Mo2	−171.69 (9)	O4—Mo4—O12—Mo3^{i}	55.51 (16)
O10—Mo4—O4—Mo2	−65.58 (10)	O11^{i}—Mo4—O12—Mo3^{i}	14.97 (7)

O12—Mo4—O4—Mo2	53.13 (17)	O12^{i}—Mo4—O12—Mo3^{i}	99.58 (9)
O11^{i}—Mo4—O4—Mo2	93.32 (9)	O13—Mo4—O12—Mo2^{i}	42.8 (2)
O12^{i}—Mo4—O4—Mo2	9.08 (8)	O10—Mo4—O12—Mo2^{i}	−62.8 (2)
O2—Mo1—O4—Mo4	−156.55 (11)	O4—Mo4—O12—Mo2^{i}	177.84 (17)
O1—Mo1—O4—Mo4	13.9 (3)	O11^{i}—Mo4—O12—Mo2^{i}	137.3 (2)
O3—Mo1—O4—Mo4	101.67 (10)	O12^{i}—Mo4—O12—Mo2^{i}	−138.1 (3)
O11^{i}—Mo1—O4—Mo4	8.21 (8)	O13—Mo4—O12—Mo4^{i}	−179.13 (9)
N1—Mo1—O4—Mo4	−71.06 (10)	O10—Mo4—O12—Mo4^{i}	75.25 (9)
O2—Mo1—O4—Mo2	87.49 (9)	O4—Mo4—O12—Mo4^{i}	−44.07 (16)
O1—Mo1—O4—Mo2	−102.1 (3)	O11^{i}—Mo4—O12—Mo4^{i}	−84.61 (8)
O3—Mo1—O4—Mo2	−14.29 (7)	O12^{i}—Mo4—O12—Mo4^{i}	0.0
O11^{i}—Mo1—O4—Mo2	−107.74 (8)	O2—Mo1—N1—C3	34.4 (3)
N1—Mo1—O4—Mo2	172.98 (8)	O1—Mo1—N1—C3	139.6 (3)
O6—Mo2—O4—Mo4	−38.3 (3)	O3—Mo1—N1—C3	−78.3 (4)
O5—Mo2—O4—Mo4	162.41 (11)	O11^{i}—Mo1—N1—C3	−128.7 (3)
O7—Mo2—O4—Mo4	64.96 (10)	O4—Mo1—N1—C3	−58.0 (3)
O3—Mo2—O4—Mo4	−98.31 (10)	O2—Mo1—N1—C1	−141.6 (3)
O12^{i}—Mo2—O4—Mo4	−9.73 (8)	O1—Mo1—N1—C1	−36.5 (3)
O6—Mo2—O4—Mo1	73.9 (3)	O3—Mo1—N1—C1	105.6 (3)
O5—Mo2—O4—Mo1	−85.34 (9)	O11^{i}—Mo1—N1—C1	55.3 (3)
O7—Mo2—O4—Mo1	177.21 (7)	O4—Mo1—N1—C1	125.9 (3)
O3—Mo2—O4—Mo1	13.94 (7)	C3—N1—C1—C2	−1.9 (4)
O12^{i}—Mo2—O4—Mo1	102.52 (7)	Mo1—N1—C1—C2	174.7 (2)
O6—Mo2—O7—Mo3	67.07 (12)	N1—C1—C2—N2	1.5 (4)
O5—Mo2—O7—Mo3	173.91 (11)	C3—N2—C2—C1	−0.5 (4)
O3—Mo2—O7—Mo3	−59.4 (2)	C1—N1—C3—N2	1.6 (4)
O4—Mo2—O7—Mo3	−94.41 (10)	Mo1—N1—C3—N2	−175.2 (2)
O12^{i}—Mo2—O7—Mo3	−20.57 (9)	C2—N2—C3—N1	−0.7 (4)
O8—Mo3—O7—Mo2	−74.29 (12)	C6—N3—C4—C5	0.3 (4)
O9—Mo3—O7—Mo2	178.51 (11)	N3—C4—C5—N4	−0.2 (4)
O11—Mo3—O7—Mo2	41.28 (19)	C6—N4—C5—C4	0.1 (4)
O12^{i}—Mo3—O7—Mo2	21.03 (9)	C4—N3—C6—N4	−0.3 (4)
O10—Mo3—O7—Mo2	94.68 (10)	C5—N4—C6—N3	0.1 (4)
O13—Mo4—O10—Mo3	177.47 (9)	C9—N6—C8—C7	−0.2 (5)
O4—Mo4—O10—Mo3	71.68 (10)	C12—N7—C10—C11	0.1 (3)
O12—Mo4—O10—Mo3	−76.79 (10)	N7—C10—C11—N8	0.0 (3)
O11^{i}—Mo4—O10—Mo3	−7.8 (3)	C12—N8—C11—C10	−0.1 (5)
O12^{i}—Mo4—O10—Mo3	−2.33 (8)	C11—N8—C12—N7	0.2 (6)
O8—Mo3—O10—Mo4	−12.1 (4)	C10—N7—C12—N8	−0.2 (5)
O9—Mo3—O10—Mo4	−177.03 (12)		

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

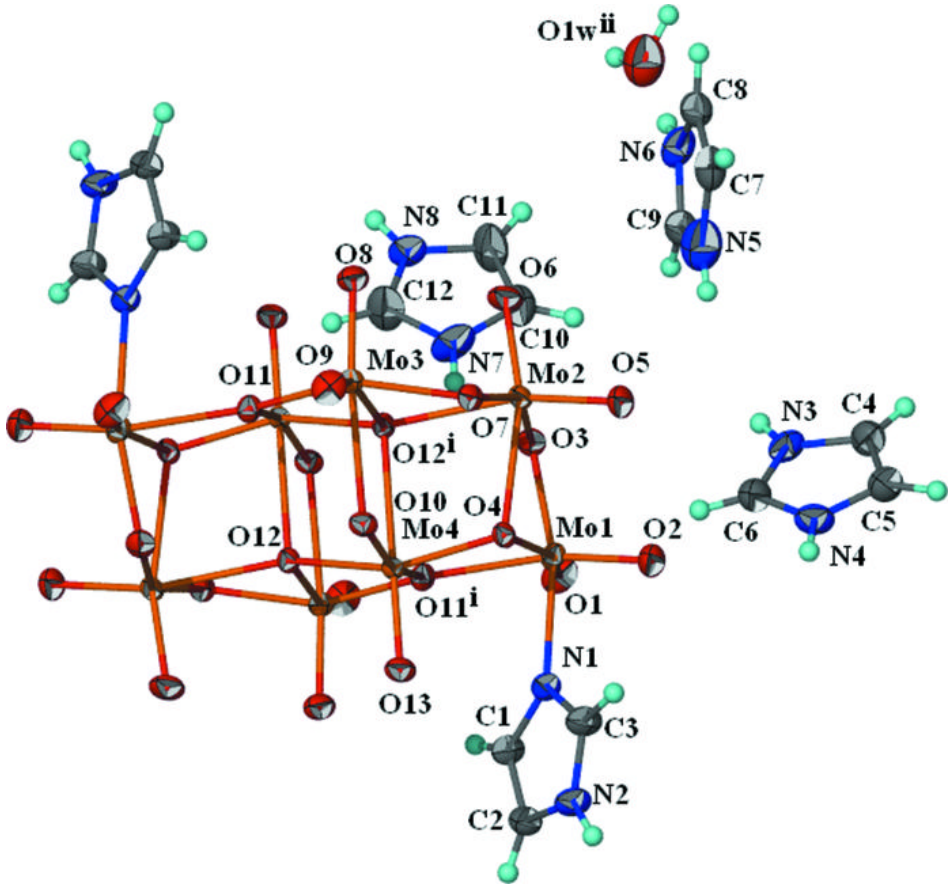
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N2—H2n···O7^{ii}	0.85	2.10	2.926 (3)	164
N3—H3n···O1^{iii}	0.85	2.24	2.889 (4)	134
N4—H4n···O8^{iv}	0.85	2.25	3.094 (4)	178
N5—H5n···O9^{v}	0.85	2.25	2.866 (4)	130
N6—H6n···O1w^{vi}	0.85	1.72	2.556 (7)	169
N7—H7n···O3	0.85	2.05	2.760 (9)	140
N8—H8n···O3^{vii}	0.85	2.08	2.766 (9)	138
O1w—H1w1···O5	0.85	2.22	3.010 (4)	154
O1w—H1w2···O9^{v}	0.85	2.13	2.866 (4)	144

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

Fig. 1


O1w^{ii}
C8
N6
C7
C9
N5
N8
C11
O8
O6
C12
C10
N7
O11
O9
Mo3
Mo2
O5
O7
O3
O12^{i}
O10
O4
Mo4
Mo1
O2
O12
O11^{i}
O1
N1
O13
C1
C3
N2
C2
N3
C4
C6
C5
N4