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Bis(2,2′-bipyridine *N*-oxide-κ^2O,N)-copper(II) β-octamolybdate

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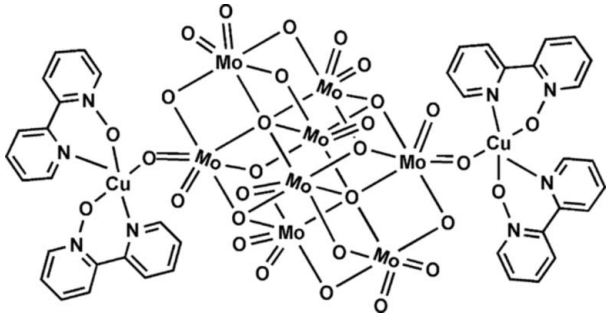
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.050; wR factor = 0.123; data-to-parameter ratio = 15.7.

The title compound, bis(2,2′-bipyridine *N*-oxide-κ^2N,O)hexacosaoxocopper(II)molybdenum(IV), $[Cu_2Mo_8O_{26}(C_{10}H_8N_2O)_4]$, was obtained by a hydrothermal reaction. The Cu atom is coordinated by two O atoms and two N atoms from two 2,2′-bipyridine *N*-oxide (*L*) ligands and one terminal O atom from one $[\beta\text{-}Mo_8O_{26}]^{4-}$ cluster with a square-pyramidal coordination geometry; the apical Cu—O bond distance is significantly longer than Cu—O bond distances in the basal plane. Two terminal O atoms of the $(\beta\text{-}Mo_8O_{26})^{4-}$ cluster link two $[Cu(L)_2]^{2+}$ units, forming a centrosymmetric dimer.

Related literature

For general background, see: Finn & Zubieta (2001); Hagrman *et al.* (1997); Kin & Pope (1999); Liu *et al.* (2001); Ramirez & von Ostwalden (1959); Sadakane *et al.* (1999); Yang *et al.* (2002). For related structures, see: Shen *et al.* (2004);



Experimental

Crystal data

$[Cu_2Mo_8O_{26}(C_{10}H_8N_2O)_4]$
M_r = 1999.34
Monoclinic, $P2_1/n$
a = 10.8210 (17) Å
b = 17.799 (2) Å
c = 14.187 (3) Å
β = 100.166 (5)°
V = 2689.6 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.68 mm^{-1}
T = 293 (2) K
0.40 × 0.38 × 0.20 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.357, T_{max} = 0.583
15777 measured reflections
6238 independent reflections
4242 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.123
S = 0.95
6238 reflections
397 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.99 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—O15	1.928 (4)	Cu1—N4	2.058 (6)
Cu1—O14	1.933 (4)	Cu1—O13	2.262 (4)
Cu1—N1	2.016 (5)		

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2239).

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supplementary materials

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Bis(2,2'-bipyridine *N*-oxide-κ^2O,N)copper(II) β-octamolybdate

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Comment

Recently, there has been an increasing interest in the assembly of polyoxometalate clusters into extended inorganic or hybrid inorganic-organic solids (Sadakane *et al.*, 1999; Kin & Pope, 1999), because of the remarkable features of metal oxide surfaces and their diversities in geometric topology by inclusion of transition-metal (TM) coordination complexes. Octamolybdate is an interesting cluster with a variety of structural isomers (Hagrman *et al.*, 1997; Yang *et al.*, 2002). As reported previously (Finn & Zubieta, 2001; Liu *et al.*, 2001), much of the work has been focused on the metal complexes with (β-Mo_8O_{26})$^{4-}$clusters. In order to enrich this system, the title compound, (I), was isolated by hydrothermal method.

As shown in Fig. 1, the title compound constains one Cu^{II} ion, two *L* ligands and one (β-Mo_8O_{26})$^{4-}$ cluster. The Cu^{II} ion exhibits a square pyramidal geometry and is coordinated by two oxygen atoms (Cu—O: 1.933 (4) and 1.928 (4) Å) and two nitrogen atoms (Cu—N: 2.016 (5) and 2.058 (6) Å) from two *L* ligands, and one terminal oxygen (Cu—O: 2.262 (4) Å) atom from the (β-Mo_8O_{26})$^{4-}$cluster. Two kinds of *L* ligands coordinate to one Cu atom as bidentate ligands. The dihedral angles between two rings from the same *L* ligand are 33.1° and 27.5°, respectively. Each (β-Mo_8O_{26})$^{4-}$ cluster acts as a bidentate bridging ligand and coordinates to two Cu^{II} ions (Fig. 2). So according to the way of the coordinated mode, five kinds of oxygen atoms exist in the cluster: terminal oxygen atoms Ot, terminal oxygen atoms Ot' coordinated to metal center, μ_2-O atoms, μ_3-O atoms and μ_4-O atoms. All of the distances of Mo—O are similar to the reported ones (Shen *et al.*, 2004).

Experimental

Ligand *L* was synthesized according to the method reported by Ramirez & Ostwalden (1959). A mixture of $Cu(OAc)_2{\cdot}H_2O$ (0.040 g, 0.2 mmol), $Na_2MoO_4{\cdot}2H_2O$ (0.242 g, 1.0 mmol), *L* (0.172 g, 1.0 mmol) and H_2O (10 ml) was stirred for 1 h and then transferred and sealed in a 25 ml Teflonlined stainless steel container. The container was heated to 150°C and held at that temperature for 72 h, then cooled to room temperature. Blue crystals of title compound were collected in 58.3% yield based on $Cu(OAc)_2$. Elemental analyses Calcd for $C_{40}H_{32}Cu_2Mo_8N_8O_{30}$: C, 24.03; H, 1.61; N, 5.60%. Found: C, 24.00; H, 1.63; N, 5.62%.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.93 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

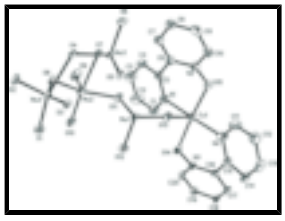

Fig. 1. A view of (I), showing the contents of the asymmetric unit and the atom numbering scheme.

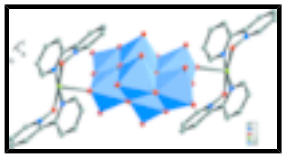

Fig. 2. Polyhedral and ball-stick representation of the dimer.

bis(2,2'-bipyridine N-oxide-κ^2*N*,*O*)hexacosaoxocopper(II)molybdenum(IV)

Crystal data

$[Cu_2Mo_8O_{26}(C_{10}H_8N_2O)_4]$	F_{000} = 1924
M_r = 1999.34	D_x = 2.469 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2yn	Cell parameters from 14895 reflections
a = 10.8210 (17) Å	θ = 1.8–28.6º
b = 17.799 (2) Å	μ = 2.68 mm^{-1}
c = 14.187 (3) Å	T = 293 (2) K
β = 100.166 (5)º	Block, blue
V = 2689.6 (8) Å^3	0.40 × 0.38 × 0.20 mm
Z = 2	

Data collection

Bruker APEX CCD area-detector diffractometer	6238 independent reflections
Radiation source: fine-focus sealed tube	4242 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.075
T = 293(2) K	θ_{max} = 28.6º
ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→14
T_{min} = 0.357, T_{max} = 0.583	k = −23→17
15777 measured reflections	l = −19→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} = 0.002$
6238 reflections	$\Delta\rho_{max} = 1.23$ e Å^{-3}
397 parameters	$\Delta\rho_{min} = -1.99$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.34259 (8)	0.98208 (4)	0.91471 (5)	0.0363 (2)
Mo1	0.11888 (5)	0.94081 (3)	0.67984 (3)	0.02653 (14)
Mo2	0.24132 (6)	0.85276 (3)	0.51635 (4)	0.03295 (16)
Mo3	0.14057 (5)	1.03734 (3)	0.46772 (3)	0.02377 (14)
Mo4	−0.02438 (5)	0.87730 (3)	0.36565 (4)	0.02872 (15)
C1	0.5002 (7)	0.8671 (4)	0.8542 (6)	0.055 (2)
H1	0.4731	0.8415	0.9039	0.066*
C2	0.5840 (8)	0.8310 (5)	0.8052 (7)	0.072 (3)
H2	0.6134	0.7830	0.8225	0.086*
C3	0.6217 (8)	0.8685 (4)	0.7309 (6)	0.063 (2)
H3	0.6742	0.8452	0.6946	0.075*
C4	0.5811 (7)	0.9415 (4)	0.7103 (5)	0.0501 (19)
H4	0.6096	0.9686	0.6623	0.060*
C5	0.4977 (6)	0.9736 (4)	0.7620 (5)	0.0375 (15)
C6	0.4494 (6)	1.0502 (3)	0.7347 (5)	0.0351 (15)
C7	0.4444 (6)	1.0780 (4)	0.6457 (5)	0.0409 (16)
H7	0.4702	1.0481	0.5991	0.049*
C8	0.4011 (6)	1.1510 (4)	0.6217 (5)	0.0452 (18)
H8	0.3967	1.1690	0.5597	0.054*
C9	0.3656 (7)	1.1953 (4)	0.6912 (6)	0.0519 (19)
H9	0.3358	1.2438	0.6770	0.062*
C10	0.3746 (7)	1.1670 (4)	0.7814 (6)	0.0476 (19)
H10	0.3521	1.1972	0.8292	0.057*
C11	0.1887 (7)	1.1000 (4)	0.9804 (6)	0.057 (2)

H11	0.2215	1.1265	0.9340	0.069*
C12	0.0967 (8)	1.1345 (4)	1.0210 (6)	0.055 (2)
H12	0.0699	1.1830	1.0033	0.066*
C13	0.0453 (7)	1.0948 (4)	1.0891 (5)	0.0517 (19)
H13	−0.0179	1.1161	1.1170	0.062*
C14	0.0897 (7)	1.0224 (4)	1.1152 (5)	0.0446 (18)
H14	0.0565	0.9950	1.1606	0.053*
C15	0.1853 (6)	0.9923 (4)	1.0714 (4)	0.0336 (14)
C16	0.2374 (6)	0.9187 (3)	1.1036 (4)	0.0312 (14)
C17	0.2456 (6)	0.8946 (4)	1.1981 (4)	0.0339 (14)
H17	0.2159	0.9255	1.2420	0.041*
C18	0.2969 (6)	0.8258 (3)	1.2277 (4)	0.0343 (15)
H18	0.3014	0.8105	1.2909	0.041*
C19	0.3418 (6)	0.7796 (4)	1.1625 (5)	0.0377 (15)
H19	0.3784	0.7335	1.1816	0.045*
C20	0.3313 (6)	0.8032 (4)	1.0699 (4)	0.0357 (15)
H20	0.3577	0.7717	1.0251	0.043*
N1	0.4574 (5)	0.9349 (3)	0.8340 (4)	0.0404 (13)
N2	0.4157 (5)	1.0958 (3)	0.8036 (4)	0.0416 (14)
N3	0.2833 (5)	0.8710 (3)	1.0421 (3)	0.0316 (12)
N4	0.2341 (5)	1.0310 (3)	1.0033 (4)	0.0457 (15)
O1	−0.0818 (4)	0.7978 (2)	0.4085 (3)	0.0405 (11)
O2	−0.0604 (5)	0.8703 (2)	0.2439 (3)	0.0430 (12)
O3	−0.1568 (4)	0.9471 (2)	0.3940 (3)	0.0267 (9)
O4	0.0424 (4)	1.0006 (2)	0.3476 (3)	0.0277 (9)
O5	−0.0477 (3)	1.0719 (2)	0.4804 (3)	0.0248 (9)
O6	0.1935 (4)	1.1205 (2)	0.4329 (3)	0.0350 (10)
O7	0.2674 (4)	0.9756 (2)	0.4708 (3)	0.0308 (9)
O8	0.1511 (4)	0.8562 (2)	0.3869 (3)	0.0320 (10)
O9	0.3890 (5)	0.8305 (3)	0.5016 (3)	0.0522 (13)
O10	0.1803 (5)	0.7707 (2)	0.5506 (3)	0.0485 (13)
O11	0.2636 (4)	0.9055 (2)	0.6369 (3)	0.0345 (10)
O12	0.0541 (4)	0.8596 (2)	0.7123 (3)	0.0375 (11)
O13	0.1864 (4)	0.9832 (2)	0.7857 (3)	0.0342 (10)
O14	0.2724 (4)	0.8878 (3)	0.9478 (3)	0.0393 (11)
O15	0.4261 (4)	1.0752 (2)	0.8964 (3)	0.0465 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0461 (5)	0.0390 (4)	0.0242 (4)	−0.0001 (4)	0.0075 (4)	−0.0028 (3)
Mo1	0.0344 (3)	0.0322 (3)	0.0136 (3)	0.0019 (2)	0.0060 (2)	0.0027 (2)
Mo2	0.0451 (4)	0.0343 (3)	0.0221 (3)	0.0076 (2)	0.0135 (3)	0.0031 (2)
Mo3	0.0284 (3)	0.0291 (3)	0.0154 (3)	−0.0022 (2)	0.0085 (2)	0.00181 (19)
Mo4	0.0387 (3)	0.0288 (3)	0.0208 (3)	−0.0006 (2)	0.0112 (2)	−0.0012 (2)
C1	0.060 (5)	0.052 (5)	0.063 (6)	0.002 (4)	0.035 (4)	0.007 (4)
C2	0.073 (6)	0.049 (5)	0.108 (8)	0.021 (4)	0.054 (6)	0.021 (5)
C3	0.060 (5)	0.059 (5)	0.081 (6)	0.015 (4)	0.043 (5)	0.000 (4)

C4	0.061 (5)	0.045 (4)	0.051 (5)	−0.002 (4)	0.030 (4)	0.003 (3)
C5	0.033 (4)	0.045 (4)	0.036 (4)	−0.004 (3)	0.012 (3)	−0.003 (3)
C6	0.034 (4)	0.036 (3)	0.036 (4)	−0.001 (3)	0.008 (3)	−0.002 (3)
C7	0.039 (4)	0.047 (4)	0.038 (4)	−0.010 (3)	0.010 (3)	−0.004 (3)
C8	0.039 (4)	0.053 (4)	0.043 (4)	−0.013 (3)	0.006 (3)	0.005 (3)
C9	0.049 (5)	0.038 (4)	0.069 (6)	−0.005 (3)	0.012 (4)	0.002 (4)
C10	0.050 (5)	0.037 (4)	0.060 (5)	0.000 (3)	0.022 (4)	−0.009 (4)
C11	0.055 (5)	0.052 (5)	0.073 (6)	−0.003 (4)	0.031 (4)	0.013 (4)
C12	0.069 (6)	0.044 (4)	0.051 (5)	0.010 (4)	0.012 (4)	0.007 (4)
C13	0.061 (5)	0.061 (5)	0.035 (4)	0.016 (4)	0.015 (4)	−0.006 (4)
C14	0.059 (5)	0.049 (4)	0.030 (4)	0.013 (4)	0.021 (3)	0.008 (3)
C15	0.034 (3)	0.043 (4)	0.025 (3)	0.000 (3)	0.009 (3)	0.000 (3)
C16	0.034 (3)	0.039 (3)	0.023 (3)	−0.006 (3)	0.011 (3)	−0.003 (3)
C17	0.041 (4)	0.041 (4)	0.024 (3)	0.001 (3)	0.015 (3)	−0.001 (3)
C18	0.046 (4)	0.043 (4)	0.015 (3)	0.002 (3)	0.008 (3)	0.000 (3)
C19	0.041 (4)	0.037 (4)	0.036 (4)	0.004 (3)	0.008 (3)	0.002 (3)
C20	0.041 (4)	0.040 (4)	0.028 (4)	0.006 (3)	0.011 (3)	−0.005 (3)
N1	0.035 (3)	0.041 (3)	0.046 (4)	0.000 (3)	0.011 (3)	0.002 (3)
N2	0.042 (3)	0.045 (3)	0.041 (4)	−0.011 (3)	0.015 (3)	−0.008 (3)
N3	0.037 (3)	0.045 (3)	0.013 (2)	−0.004 (2)	0.003 (2)	−0.001 (2)
N4	0.050 (4)	0.048 (4)	0.040 (4)	−0.002 (3)	0.011 (3)	0.014 (3)
O1	0.051 (3)	0.034 (2)	0.041 (3)	0.000 (2)	0.022 (2)	−0.001 (2)
O2	0.065 (3)	0.046 (3)	0.020 (2)	0.001 (2)	0.014 (2)	−0.0080 (19)
O3	0.033 (2)	0.033 (2)	0.014 (2)	−0.0038 (17)	0.0038 (17)	−0.0003 (16)
O4	0.035 (2)	0.037 (2)	0.013 (2)	−0.0007 (18)	0.0088 (17)	0.0021 (17)
O5	0.028 (2)	0.031 (2)	0.016 (2)	0.0010 (17)	0.0058 (17)	−0.0005 (16)
O6	0.040 (3)	0.039 (2)	0.029 (2)	−0.0037 (19)	0.013 (2)	0.0070 (19)
O7	0.035 (2)	0.039 (2)	0.020 (2)	−0.0016 (19)	0.0098 (18)	0.0010 (17)
O8	0.038 (2)	0.040 (2)	0.021 (2)	0.0049 (19)	0.0130 (19)	−0.0025 (18)
O9	0.054 (3)	0.063 (3)	0.044 (3)	0.024 (3)	0.020 (3)	0.005 (2)
O10	0.079 (4)	0.043 (3)	0.025 (3)	0.003 (3)	0.014 (2)	0.005 (2)
O11	0.036 (2)	0.047 (3)	0.021 (2)	0.003 (2)	0.0058 (19)	0.0016 (19)
O12	0.047 (3)	0.035 (2)	0.032 (3)	−0.002 (2)	0.012 (2)	0.0044 (19)
O13	0.038 (2)	0.047 (3)	0.016 (2)	0.003 (2)	−0.0007 (18)	0.0010 (18)
O14	0.051 (3)	0.055 (3)	0.013 (2)	−0.006 (2)	0.007 (2)	0.000 (2)
O15	0.060 (3)	0.048 (3)	0.036 (3)	−0.014 (2)	0.021 (2)	−0.008 (2)

Geometric parameters (Å, °)

Cu1—O15	1.928 (4)	C5—C6	1.487 (9)
Cu1—O14	1.933 (4)	C6—C7	1.349 (9)
Cu1—N1	2.016 (5)	C6—N2	1.368 (8)
Cu1—N4	2.058 (6)	C7—C8	1.402 (9)
Cu1—O13	2.262 (4)	C7—H7	0.9300
Mo1—O12	1.705 (4)	C8—C9	1.369 (9)
Mo1—O13	1.723 (4)	C8—H8	0.9300
Mo1—O11	1.885 (4)	C9—C10	1.363 (10)
Mo1—O4^{i}	2.011 (4)	C9—H9	0.9300
Mo1—O5^{i}	2.280 (4)	C10—N2	1.361 (8)

Mo1—O3^{i}	2.323 (4)	C10—H10	0.9300
Mo1—Mo3^{i}	3.2157 (8)	C11—N4	1.342 (9)
Mo2—O9	1.695 (5)	C11—C12	1.378 (10)
Mo2—O10	1.709 (4)	C11—H11	0.9300
Mo2—O8	1.922 (4)	C12—C13	1.389 (10)
Mo2—O11	1.929 (4)	C12—H12	0.9300
Mo2—O7	2.311 (4)	C13—C14	1.402 (9)
Mo2—O5^{i}	2.495 (4)	C13—H13	0.9300
Mo3—O6	1.693 (4)	C14—C15	1.405 (9)
Mo3—O7	1.753 (4)	C14—H14	0.9300
Mo3—O4	1.955 (4)	C15—N4	1.366 (8)
Mo3—O3^{i}	1.958 (4)	C15—C16	1.467 (8)
Mo3—O5	2.166 (4)	C16—N3	1.371 (7)
Mo3—O5^{i}	2.366 (4)	C16—C17	1.395 (8)
Mo3—Mo1^{i}	3.2157 (8)	C17—C18	1.378 (8)
Mo4—O1	1.700 (4)	C17—H17	0.9300
Mo4—O2	1.706 (4)	C18—C19	1.388 (8)
Mo4—O8	1.907 (4)	C18—H18	0.9300
Mo4—O3	1.991 (4)	C19—C20	1.364 (8)
Mo4—O4	2.339 (4)	C19—H19	0.9300
Mo4—O5^{i}	2.365 (4)	C20—N3	1.346 (8)
C1—N1	1.307 (9)	C20—H20	0.9300
C1—C2	1.392 (9)	N2—O15	1.352 (7)
C1—H1	0.9300	N3—O14	1.356 (6)
C2—C3	1.369 (10)	O3—Mo3^{i}	1.958 (4)
C2—H2	0.9300	O3—Mo1^{i}	2.323 (4)
C3—C4	1.386 (10)	O4—Mo1^{i}	2.011 (4)
C3—H3	0.9300	O5—Mo1^{i}	2.280 (4)
C4—C5	1.383 (9)	O5—Mo4^{i}	2.365 (4)
C4—H4	0.9300	O5—Mo3^{i}	2.366 (4)
C5—N1	1.366 (8)	O5—Mo2^{i}	2.495 (4)
O15—Cu1—O14	173.07 (19)	C2—C3—C4	119.5 (7)
O15—Cu1—N1	86.0 (2)	C2—C3—H3	120.2
O14—Cu1—N1	94.8 (2)	C4—C3—H3	120.2
O15—Cu1—N4	92.9 (2)	C5—C4—C3	119.2 (7)
O14—Cu1—N4	85.9 (2)	C5—C4—H4	120.4
N1—Cu1—N4	176.7 (2)	C3—C4—H4	120.4
O15—Cu1—O13	100.63 (18)	N1—C5—C4	120.8 (6)
O14—Cu1—O13	86.26 (17)	N1—C5—C6	120.9 (6)
N1—Cu1—O13	89.5 (2)	C4—C5—C6	118.3 (6)
N4—Cu1—O13	93.8 (2)	C7—C6—N2	118.7 (6)
O12—Mo1—O13	105.5 (2)	C7—C6—C5	122.3 (6)
O12—Mo1—O11	102.08 (19)	N2—C6—C5	118.9 (6)
O13—Mo1—O11	100.29 (19)	C6—C7—C8	121.5 (7)
O12—Mo1—O4^{i}	96.18 (19)	C6—C7—H7	119.2

O13—Mo1—O4^{i}	99.31 (18)	C8—C7—H7	119.2
O11—Mo1—O4^{i}	148.43 (16)	C9—C8—C7	118.9 (7)
O12—Mo1—O5^{i}	95.85 (17)	C9—C8—H8	120.5
O13—Mo1—O5^{i}	158.31 (17)	C7—C8—H8	120.5
O11—Mo1—O5^{i}	78.36 (15)	C10—C9—C8	118.7 (7)
O4^{i}—Mo1—O5^{i}	74.31 (14)	C10—C9—H9	120.6
O12—Mo1—O3^{i}	164.48 (19)	C8—C9—H9	120.6
O13—Mo1—O3^{i}	86.30 (17)	N2—C10—C9	121.8 (7)
O11—Mo1—O3^{i}	85.26 (16)	N2—C10—H10	119.1
O4^{i}—Mo1—O3^{i}	71.58 (14)	C9—C10—H10	119.1
O5^{i}—Mo1—O3^{i}	72.01 (13)	N4—C11—C12	124.6 (7)
O12—Mo1—Mo3^{i}	85.69 (16)	N4—C11—H11	117.7
O13—Mo1—Mo3^{i}	134.55 (14)	C12—C11—H11	117.7
O11—Mo1—Mo3^{i}	120.62 (12)	C11—C12—C13	118.1 (7)
O4^{i}—Mo1—Mo3^{i}	35.23 (11)	C11—C12—H12	121.0
O5^{i}—Mo1—Mo3^{i}	42.28 (9)	C13—C12—H12	121.0
O3^{i}—Mo1—Mo3^{i}	78.81 (10)	C12—C13—C14	119.4 (6)
O9—Mo2—O10	104.8 (2)	C12—C13—H13	120.3
O9—Mo2—O8	102.7 (2)	C14—C13—H13	120.3
O10—Mo2—O8	98.2 (2)	C13—C14—C15	118.6 (6)
O9—Mo2—O11	104.5 (2)	C13—C14—H14	120.7
O10—Mo2—O11	99.11 (19)	C15—C14—H14	120.7
O8—Mo2—O11	142.59 (17)	N4—C15—C14	121.8 (6)
O9—Mo2—O7	91.2 (2)	N4—C15—C16	119.7 (5)
O10—Mo2—O7	163.9 (2)	C14—C15—C16	118.4 (5)
O8—Mo2—O7	76.66 (16)	N3—C16—C17	116.9 (6)
O11—Mo2—O7	77.58 (16)	N3—C16—C15	121.1 (5)
O9—Mo2—O5^{i}	160.23 (19)	C17—C16—C15	122.0 (5)
O10—Mo2—O5^{i}	94.96 (19)	C18—C17—C16	121.3 (6)
O8—Mo2—O5^{i}	73.38 (14)	C18—C17—H17	119.3
O11—Mo2—O5^{i}	72.29 (15)	C16—C17—H17	119.3
O7—Mo2—O5^{i}	68.99 (13)	C17—C18—C19	119.5 (6)
O6—Mo3—O7	104.60 (19)	C17—C18—H18	120.2
O6—Mo3—O4	101.40 (18)	C19—C18—H18	120.2
O7—Mo3—O4	96.58 (18)	C20—C19—C18	118.7 (6)
O6—Mo3—O3^{i}	101.20 (18)	C20—C19—H19	120.7
O7—Mo3—O3^{i}	97.59 (17)	C18—C19—H19	120.7
O4—Mo3—O3^{i}	149.26 (16)	N3—C20—C19	121.3 (5)
O6—Mo3—O5	98.47 (17)	N3—C20—H20	119.3
O7—Mo3—O5	156.92 (16)	C19—C20—H20	119.3
O4—Mo3—O5	78.08 (15)	C1—N1—C5	118.9 (6)
O3^{i}—Mo3—O5	78.21 (15)	C1—N1—Cu1	119.4 (5)
O6—Mo3—O5^{i}	173.94 (17)	C5—N1—Cu1	121.7 (4)

O7—Mo3—O5^{i}	81.39 (15)	O15—N2—C10	116.4 (5)
O4—Mo3—O5^{i}	78.51 (14)	O15—N2—C6	123.2 (6)
O3^{i}—Mo3—O5^{i}	76.84 (14)	C10—N2—C6	120.3 (6)
O5—Mo3—O5^{i}	75.54 (15)	C20—N3—O14	116.7 (5)
O6—Mo3—Mo1^{i}	90.32 (15)	C20—N3—C16	122.2 (5)
O7—Mo3—Mo1^{i}	132.99 (13)	O14—N3—C16	120.9 (5)
O4—Mo3—Mo1^{i}	36.41 (11)	C11—N4—C15	117.5 (6)
O3^{i}—Mo3—Mo1^{i}	123.28 (12)	C11—N4—Cu1	117.6 (5)
O5—Mo3—Mo1^{i}	45.09 (10)	C15—N4—Cu1	123.5 (4)
O5^{i}—Mo3—Mo1^{i}	86.09 (9)	Mo3^{i}—O3—Mo4	110.49 (18)
O1—Mo4—O2	105.6 (2)	Mo3^{i}—O3—Mo1^{i}	109.81 (17)
O1—Mo4—O8	101.32 (19)	Mo4—O3—Mo1^{i}	104.73 (15)
O2—Mo4—O8	100.90 (19)	Mo3—O4—Mo1^{i}	108.35 (18)
O1—Mo4—O3	97.24 (18)	Mo3—O4—Mo4	110.44 (16)
O2—Mo4—O3	101.88 (19)	Mo1^{i}—O4—Mo4	103.48 (16)
O8—Mo4—O3	145.40 (16)	Mo3—O5—Mo1^{i}	92.63 (13)
O1—Mo4—O4	163.66 (17)	Mo3—O5—Mo4^{i}	91.37 (14)
O2—Mo4—O4	88.70 (18)	Mo1^{i}—O5—Mo4^{i}	163.22 (18)
O8—Mo4—O4	83.32 (16)	Mo3—O5—Mo3^{i}	104.46 (14)
O3—Mo4—O4	71.55 (14)	Mo1^{i}—O5—Mo3^{i}	98.10 (14)
O1—Mo4—O5^{i}	94.00 (17)	Mo4^{i}—O5—Mo3^{i}	96.66 (13)
O2—Mo4—O5^{i}	160.31 (17)	Mo3—O5—Mo2^{i}	163.68 (18)
O8—Mo4—O5^{i}	76.83 (15)	Mo1^{i}—O5—Mo2^{i}	86.25 (12)
O3—Mo4—O5^{i}	72.94 (14)	Mo4^{i}—O5—Mo2^{i}	85.35 (11)
O4—Mo4—O5^{i}	71.62 (13)	Mo3^{i}—O5—Mo2^{i}	91.81 (12)
N1—C1—C2	123.6 (7)	Mo3—O7—Mo2	117.80 (19)
N1—C1—H1	118.2	Mo4—O8—Mo2	118.75 (18)
C2—C1—H1	118.2	Mo1—O11—Mo2	117.9 (2)
C3—C2—C1	117.9 (7)	Mo1—O13—Cu1	147.4 (2)
C3—C2—H2	121.1	N3—O14—Cu1	117.5 (3)
C1—C2—H2	121.1	N2—O15—Cu1	113.8 (4)
N1—C1—C2—C3	−1.1 (14)	O1—Mo4—O4—Mo3	43.4 (7)
C1—C2—C3—C4	3.3 (14)	O2—Mo4—O4—Mo3	−165.4 (2)
C2—C3—C4—C5	−3.5 (13)	O8—Mo4—O4—Mo3	−64.26 (19)
C3—C4—C5—N1	1.5 (11)	O3—Mo4—O4—Mo3	91.63 (19)
C3—C4—C5—C6	−176.3 (7)	O5^{i}—Mo4—O4—Mo3	14.05 (15)
N1—C5—C6—C7	−152.9 (7)	O1—Mo4—O4—Mo1^{i}	−72.4 (7)
C4—C5—C6—C7	24.9 (10)	O2—Mo4—O4—Mo1^{i}	78.8 (2)
N1—C5—C6—N2	31.0 (9)	O8—Mo4—O4—Mo1^{i}	179.96 (18)
C4—C5—C6—N2	−151.2 (6)	O3—Mo4—O4—Mo1^{i}	−24.15 (15)
N2—C6—C7—C8	−2.5 (10)	O5^{i}—Mo4—O4—Mo1^{i}	−101.73 (16)
C5—C6—C7—C8	−178.6 (6)	O6—Mo3—O5—Mo1^{i}	81.92 (17)

C6—C7—C8—C9	1.3 (10)	O7—Mo3—O5—Mo1^{i}	−96.7 (4)
C7—C8—C9—C10	0.6 (10)	O4—Mo3—O5—Mo1^{i}	−18.03 (14)
C8—C9—C10—N2	−1.2 (11)	O3^{i}—Mo3—O5—Mo1^{i}	−178.32 (16)
N4—C11—C12—C13	−1.3 (13)	O5^{i}—Mo3—O5—Mo1^{i}	−99.06 (16)
C11—C12—C13—C14	1.1 (12)	O6—Mo3—O5—Mo4^{i}	−81.78 (17)
C12—C13—C14—C15	0.0 (11)	O7—Mo3—O5—Mo4^{i}	99.6 (4)
C13—C14—C15—N4	−1.2 (11)	O4—Mo3—O5—Mo4^{i}	178.27 (15)
C13—C14—C15—C16	175.9 (6)	O3^{i}—Mo3—O5—Mo4^{i}	17.98 (13)
N4—C15—C16—N3	−33.1 (9)	O5^{i}—Mo3—O5—Mo4^{i}	97.23 (15)
C14—C15—C16—N3	149.7 (6)	Mo1^{i}—Mo3—O5—Mo4^{i}	−163.70 (18)
N4—C15—C16—C17	145.2 (6)	O6—Mo3—O5—Mo3^{i}	−179.02 (18)
C14—C15—C16—C17	−32.0 (9)	O7—Mo3—O5—Mo3^{i}	2.4 (5)
N3—C16—C17—C18	−0.4 (9)	O4—Mo3—O5—Mo3^{i}	81.04 (16)
C15—C16—C17—C18	−178.8 (6)	O3^{i}—Mo3—O5—Mo3^{i}	−79.26 (16)
C16—C17—C18—C19	0.3 (10)	O5^{i}—Mo3—O5—Mo3^{i}	0.0
C17—C18—C19—C20	−1.4 (10)	Mo1^{i}—Mo3—O5—Mo3^{i}	99.06 (16)
C18—C19—C20—N3	2.8 (10)	O6—Mo3—O5—Mo2^{i}	−3.7 (7)
C2—C1—N1—C5	−0.9 (12)	O7—Mo3—O5—Mo2^{i}	177.7 (5)
C2—C1—N1—Cu1	−177.2 (7)	O4—Mo3—O5—Mo2^{i}	−103.7 (6)
C4—C5—N1—C1	0.7 (10)	O3^{i}—Mo3—O5—Mo2^{i}	96.0 (6)
C6—C5—N1—C1	178.5 (7)	O5^{i}—Mo3—O5—Mo2^{i}	175.3 (7)
C4—C5—N1—Cu1	176.9 (5)	Mo1^{i}—Mo3—O5—Mo2^{i}	−85.6 (6)
C6—C5—N1—Cu1	−5.3 (9)	O6—Mo3—O7—Mo2	179.6 (2)
O15—Cu1—N1—C1	143.8 (6)	O4—Mo3—O7—Mo2	−76.8 (2)
O14—Cu1—N1—C1	−29.3 (6)	O3^{i}—Mo3—O7—Mo2	75.8 (2)
O13—Cu1—N1—C1	−115.5 (6)	O5—Mo3—O7—Mo2	−1.9 (5)
O15—Cu1—N1—C5	−32.3 (5)	O5^{i}—Mo3—O7—Mo2	0.46 (18)
O14—Cu1—N1—C5	154.6 (5)	Mo1^{i}—Mo3—O7—Mo2	−75.9 (2)
O13—Cu1—N1—C5	68.3 (5)	O9—Mo2—O7—Mo3	179.4 (3)
C9—C10—N2—O15	177.1 (6)	O10—Mo2—O7—Mo3	3.7 (8)
C9—C10—N2—C6	0.0 (10)	O8—Mo2—O7—Mo3	76.6 (2)
C7—C6—N2—O15	−175.0 (6)	O11—Mo2—O7—Mo3	−76.0 (2)
C5—C6—N2—O15	1.2 (9)	O5^{i}—Mo2—O7—Mo3	−0.47 (18)
C7—C6—N2—C10	1.9 (9)	O1—Mo4—O8—Mo2	−69.1 (3)
C5—C6—N2—C10	178.1 (6)	O2—Mo4—O8—Mo2	−177.6 (2)
C19—C20—N3—O14	−177.5 (6)	O3—Mo4—O8—Mo2	52.0 (4)
C19—C20—N3—C16	−3.0 (10)	O4—Mo4—O8—Mo2	95.0 (2)
C17—C16—N3—C20	1.7 (9)	O5^{i}—Mo4—O8—Mo2	22.4 (2)
C15—C16—N3—C20	−179.8 (6)	O9—Mo2—O8—Mo4	178.5 (3)
C17—C16—N3—O14	176.0 (5)	O10—Mo2—O8—Mo4	71.2 (3)
C15—C16—N3—O14	−5.6 (8)	O11—Mo2—O8—Mo4	−45.6 (4)
C12—C11—N4—C15	0.2 (12)	O7—Mo2—O8—Mo4	−93.2 (2)
C12—C11—N4—Cu1	167.3 (7)	O5^{i}—Mo2—O8—Mo4	−21.51 (19)

C14—C15—N4—C11	1.1 (10)	O12—Mo1—O11—Mo2	72.2 (3)
C16—C15—N4—C11	−176.0 (6)	O13—Mo1—O11—Mo2	−179.4 (2)
C14—C15—N4—Cu1	−165.2 (5)	O4^{i}—Mo1—O11—Mo2	−51.8 (4)
C16—C15—N4—Cu1	17.7 (8)	O5^{i}—Mo1—O11—Mo2	−21.4 (2)
O15—Cu1—N4—C11	39.5 (6)	O3^{i}—Mo1—O11—Mo2	−94.0 (2)
O14—Cu1—N4—C11	−147.3 (6)	Mo3^{i}—Mo1—O11—Mo2	−20.0 (3)
O13—Cu1—N4—C11	−61.4 (6)	O9—Mo2—O11—Mo1	179.7 (2)
O15—Cu1—N4—C15	−154.2 (5)	O10—Mo2—O11—Mo1	−72.3 (3)
O14—Cu1—N4—C15	18.9 (5)	O8—Mo2—O11—Mo1	44.3 (4)
O13—Cu1—N4—C15	104.9 (5)	O7—Mo2—O11—Mo1	91.7 (2)
O1—Mo4—O3—Mo3^{i}	70.5 (2)	O5^{i}—Mo2—O11—Mo1	20.1 (2)
O2—Mo4—O3—Mo3^{i}	178.1 (2)	O12—Mo1—O13—Cu1	71.2 (4)
O8—Mo4—O3—Mo3^{i}	−51.7 (3)	O11—Mo1—O13—Cu1	−34.5 (5)
O4—Mo4—O3—Mo3^{i}	−97.32 (19)	O4^{i}—Mo1—O13—Cu1	170.3 (4)
O5^{i}—Mo4—O3—Mo3^{i}	−21.53 (16)	O5^{i}—Mo1—O13—Cu1	−118.9 (5)
O1—Mo4—O3—Mo1^{i}	−171.35 (19)	O3^{i}—Mo1—O13—Cu1	−119.0 (4)
O2—Mo4—O3—Mo1^{i}	−63.7 (2)	Mo3^{i}—Mo1—O13—Cu1	170.6 (3)
O8—Mo4—O3—Mo1^{i}	66.5 (3)	O15—Cu1—O13—Mo1	117.0 (4)
O4—Mo4—O3—Mo1^{i}	20.87 (13)	O14—Cu1—O13—Mo1	−63.7 (4)
O5^{i}—Mo4—O3—Mo1^{i}	96.67 (16)	N1—Cu1—O13—Mo1	31.2 (4)
O6—Mo3—O4—Mo1^{i}	−74.7 (2)	N4—Cu1—O13—Mo1	−149.3 (4)
O7—Mo3—O4—Mo1^{i}	178.90 (18)	C20—N3—O14—Cu1	−128.7 (5)
O3^{i}—Mo3—O4—Mo1^{i}	61.9 (4)	C16—N3—O14—Cu1	56.8 (6)
O5—Mo3—O4—Mo1^{i}	21.66 (16)	N1—Cu1—O14—N3	122.5 (4)
O5^{i}—Mo3—O4—Mo1^{i}	99.10 (17)	N4—Cu1—O14—N3	−54.3 (4)
O6—Mo3—O4—Mo4	172.59 (18)	O13—Cu1—O14—N3	−148.3 (4)
O7—Mo3—O4—Mo4	66.20 (19)	C10—N2—O15—Cu1	128.5 (5)
O3^{i}—Mo3—O4—Mo4	−50.8 (4)	C6—N2—O15—Cu1	−54.4 (7)
O5—Mo3—O4—Mo4	−91.03 (17)	N1—Cu1—O15—N2	59.8 (4)
O5^{i}—Mo3—O4—Mo4	−13.59 (15)	N4—Cu1—O15—N2	−123.3 (4)
Mo1^{i}—Mo3—O4—Mo4	−112.7 (2)	O13—Cu1—O15—N2	−28.9 (4)

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

Fig. 1

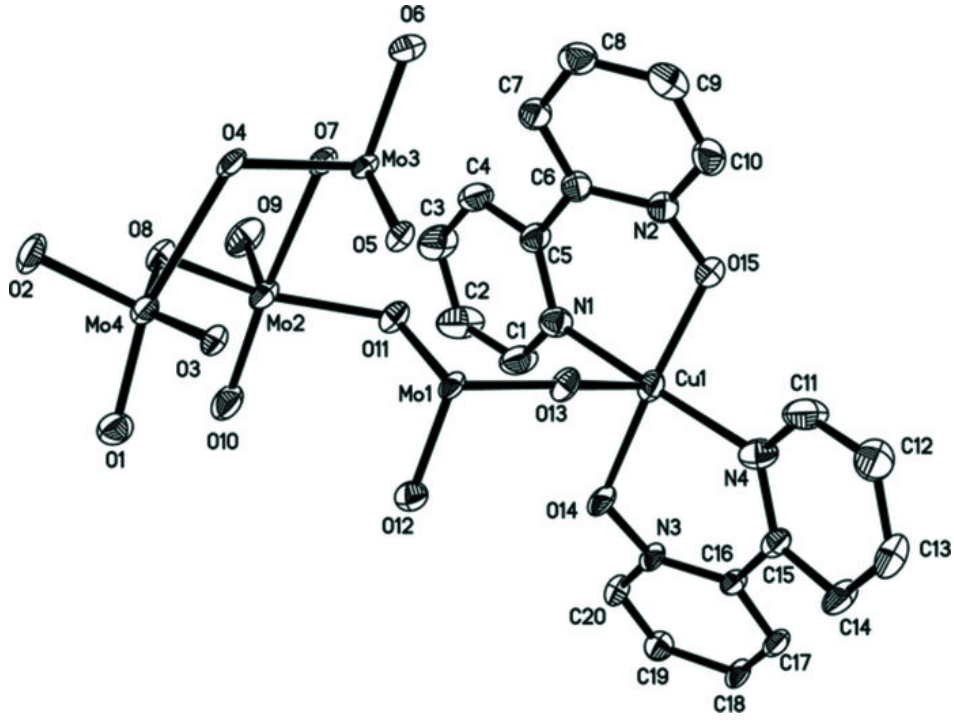
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Fig. 2