Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(4,4′-bipyridinium) bis(μ_5-hydrogen phosphate)pentakis(μ_2-oxido)decaoxidopentamolybdate dihydrate

Wu-Hua Chen, Yong Xiang, Zhan-Feng Chen, Qiu-Mu Wu and Qing-Xin Zeng*

Department of Chemistry, Fuzhou University, Fuzhou 350002, People's Republic of China
Correspondence e-mail: cwh_2125@163.com

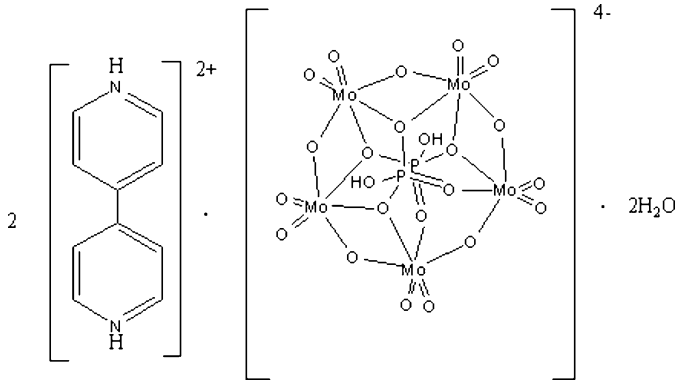
Received 10 May 2007; accepted 6 July 2007

Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.004 Å; R factor = 0.020; wR factor = 0.052; data-to-parameter ratio = 15.4.

The title compound, $(bipyH_2)_2[H_2Mo_5O_{23}P_2]\cdot 2H_2O$ or $(C_{10}H_{10}N_2)_2[Mo_5O_{15}(HPO_4)_2]\cdot 2H_2O$, contains the heteropolyanion $[P_2Mo_5O_{21}(OH)_2]^{4-}$, two diprotonated 4,4′-bipyridine (bipy) molecules as cations and two water molecules. The heteropolyanion is built up from five MoO_6 octahedra sharing four common edges and one common corner. The heteropolyanion has approximate noncrystallographic twofold rotation symmetry, the axis running through one Mo and one O atom. Five bridging O atoms between molybdenum centers connect the distorted MoO_6 octahedra to form a ring system, with ten Mo—O distances having an average value of 1.923 (2) Å. Six O atoms of the $PO_3(OH)$ tetrahedra above and below the ring stabilize the polyanionic framework. The ten Mo—OP bond lengths differ markedly, between 2.219 (2) and 2.461 (2) Å. The ten short terminal Mo—O distances average 1.706 (2) Å. The P—O bond lengths range between 1.513 (2) and 1.567 (2) Å. The crystal packing exhibits a great number of N—H···O and O—H···O intermolecular classical hydrogen bonds, which link the cations, water molecules and polyanions to form a three-dimensional framework.

Related literature

For related literature see: Haushalter & Mundi (1992); Lii *et al.* (1998); Luo *et al.* (2003); Wang *et al.* (1995); Wu *et al.* (2002).



Experimental

Crystal data

$(C_{10}H_{10}N_2)_2[Mo_5O_{15}(HPO_4)_2]\cdot 2H_2O$
M_r = 1264.09
Orthorhombic, *Pbca*
a = 11.823 (2) Å
b = 18.307 (4) Å
c = 31.517 (6) Å
V = 6821 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.99 mm^{-1}
T = 170 (2) K
0.62 × 0.10 × 0.06 mm

Data collection

Rigaku R-AXIS SPIDER diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.788, T_{max} = 0.888
58657 measured reflections
7810 independent reflections
6885 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
2 standard reflections
every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.052
S = 1.04
7810 reflections
507 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mo1—O6	1.7041 (19)	Mo1—O17	2.4114 (17)
Mo1—O8	1.7045 (19)	P1—O14	1.5134 (18)
Mo1—O10	1.9073 (17)	P1—O17	1.5365 (18)
Mo1—O15	1.9518 (17)	P1—O2	1.5369 (17)
Mo1—O18	2.2191 (17)	P1—O4	1.5606 (18)
O6—Mo1—O8	105.34 (10)	O8—Mo1—O17	164.57 (8)
O6—Mo1—O10	101.73 (8)	O10—Mo1—O17	72.50 (6)
O8—Mo1—O10	98.53 (8)	O15—Mo1—O17	83.87 (6)
O6—Mo1—O15	94.65 (8)	O18—Mo1—O17	72.85 (6)
O8—Mo1—O15	100.26 (8)	O14—P1—O17	110.84 (10)
O10—Mo1—O15	150.77 (7)	O14—P1—O2	109.63 (10)
O6—Mo1—O18	158.81 (8)	O17—P1—O2	111.07 (9)
O8—Mo1—O18	93.99 (8)	O14—P1—O4	110.65 (11)
O10—Mo1—O18	83.52 (7)	O17—P1—O4	109.33 (11)
O15—Mo1—O18	73.04 (6)	O2—P1—O4	105.19 (11)
O6—Mo1—O17	89.01 (8)		

 doi:10.1107/S1600536807033119

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H5A···O8^{i}	0.97	2.07	3.008 (3)	162
O1W—H5B···N4^{ii}	0.96	2.44	3.135 (4)	129
O2W—H6A···O1^{iii}	0.99	2.54	3.059 (3)	112
O2W—H6B···O11^{iii}	0.99	2.25	3.221 (3)	166
O2W—H6B···O16^{iii}	0.99	2.53	3.121 (4)	118
N1—H1···O22^{iv}	0.88	1.86	2.728 (3)	170
N3—H3···O9^{v}	0.88	1.89	2.761 (3)	169
N3—H3···O21^{v}	0.88	2.43	2.907 (3)	115
N4—H4···O19^{vi}	0.88	2.17	2.680 (3)	116
N4—H4···O1W^{iii}	0.88	2.47	3.135 (4)	132
O4—H21···O2W^{vii}	0.84	1.74	2.538 (3)	159
O7—H22···O1W^{viii}	0.84	1.98	2.805 (3)	169
N2—H2···O15	0.88	1.75	2.618 (3)	166
O1W—H5C···O23	0.97	2.05	3.009 (3)	173

Symmetry codes: (i) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, z$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$; (iv) $-x+2, -y+1, -z$; (v) $x+1, y, z$; (vi) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (vii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$; (viii) $x+\frac{1}{2}, y, -z+\frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

This work was supported by the Science and Technology Development Foundation of Fuzhou University (No. 2004-xq-05) and the Scientific Research Foundation of Fujian Education Department (No. JB04010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2017).

References

Haushalter, R. C. & Mundi, L. A. (1992). *Chem. Mater.* **4**, 31–48.
Lii, K.-H., Huang, Y.-F., Zima, V., Huang, C.-Y., Lin, H.-M., Jiang, Y.-C., Liao, F.-L. & Wang, S.-L. (1998). *Chem. Mater.* **10**, 2599–2609.
Luo, J.-H., Hong, M.-C., Wang, R. H., Shi, Q., Cao, R., Weng, J.-B., Sun, R.-Q. & Zhang, H.-H. (2003). *Inorg. Chem. Commun.* **6**, 702–705.
Rigaku (2004). *RAPID-AUTO*. Version 3.0. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1999). *SHELXTL/PC*. Version 2000. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2000). *SADABS*. University of Göttingen, Germany.
Wang, S., Mitzi, D. B., Field, C. A. & Guloy, A. (1995). *J. Am. Chem. Soc.* **117**, 5297–5302.
Wu, C.-D., Lu, C.-Z., Zhuang, H.-H. & Huang, J.-S. (2002). *Inorg. Chem.* **41**, 5636–5637.

supplementary materials

Acta Cryst. (2007). E**63**, m2245-m2246 [**doi:10.1107/S1600536807033119**]

Bis(4,4'-bipyridinium) bis(μ_5-hydrogen phosphate)pentakis(μ_2-oxido)decaoxidopentamolybdate dihydrate

W.-H. Chen, Y. Xiang, Z.-F. Chen, Q.-M. Wu and Q.-X. Zeng

Comment

Recently, many research activities have focused on the synthesis of open-framework metal phosphates with organic ligands because of their interesting structural chemistry and potential applications (Haushalter & Mundi, 1992; Lii *et al.*, 1998; Wang *et al.*, 1995). Many metal complexes containing polymolybdates have been synthesized and characterized (Luo *et al.*, 2003; Wu *et al.*, 2002). During our ongoing studies of related materials, we obtained the title compound (Fig.1). The unit cell contains two protonated 4,4'-bipyridine molecules, an $(H_2P_2Mo_5O_{23})^{4-}$ anion and two water molecules. The heteropolyanion of $(H_2P_2Mo_5O_{23})^{4-}$ is built up from five MoO_6 octahedra (Fig. 2) sharing four common edges and one common corner, capped by two $PO_3(OH)$ tetrahedra. All the Mo atoms exhibit a 6^+ oxidation state and possess distorted octahedral geometry. The Mo—O and P—O distances are in the range 1.693 (2)–2.461 (2) Å and 1.513 (2)–1.567 (2) Å. It is interesting to note that the protonated 4,4'-bipyridine cations and the water molecules play important roles for the crystal packing arrangement by linking $(H_2P_2Mo_5O_{23})^{4-}$ anions *via* numerous N—H···O and O—H···O hydrogen-bonds. There are two types of protonated 4,4'-bipyridine molecules and two kinds of lattice-waters in the crystal packing. For example, O1W links two $(H_2P_2Mo_5O_{23})^{4-}$ anions by three O—H···O(terminal oxygen) hydrogen bonds. However, O2W links two heteropolyanions *via* O—H···O(terminal oxygen) and O—H···O(double-bridging oxygen) hydrogen bonds. Similar to the lattice-water, the ligand 4,4-bpy with atoms N1 and N2 links two $(H_2P_2Mo_5O_{23})^{4-}$ anions by N—H···O(terminal oxygen) and an N—H···O(double-bridging oxygen) hydrogen-bonds, and the other 4,4-bpy ligand with atoms N3 and N4 have two N—H···O(double- bridging oxygen) hydrogen bonds and an N—H···O(terminal oxygen) hydrogen bond. The crystal packing exhibits a great number of N—H···O and O—H···O intra- and intermolecular classic hydrogen bonds, which link the cations, water molecules and the polyanions to form a three-dimensional framework (Fig. 3). The most important geometric parameters of the title compound are listed for one example Mo-complex in Table 1. The classic hydrogen bonding interactions are given in Table 2. Ten weak intermolecular C—H···O hydrogen bonding contacts are not considered for clarity.

Experimental

All reagents were used as purchased without further purification. The synthesis was carried out in a rational way from a mixture of H_2MoO_4 (2 mmol 0.324 g). 4,4'-bipyridine(4 mmol 0.674 g), H_3PO_4(0.2 mmol 85wt%) and H_2O(12 mmol). The mixture was loaded in a Teflon-lined autoclave (23 ml capacity) and was heated at 443k for 5 d under autogenous pressure. The solid product was collected by filtration, washed with water and dried at room temperature. Colorless crystals of the title compound were isolated.

Refinement

The structure was solved by direct methods and refined on F^2 using the *SHELXTL97* software package. The hydrogen atoms that are bonded to water oxygen, nitrogen and carbon atoms were placed geometrically, with O—H, N—H and C—H distances of 0.98 Å for water, 0.88 Å for N atoms and 0.95 Å for C atoms of the pyridinium moieties, and U_{iso}(H) = 1.2U_{eq} for (O), (N) and (C) atoms, respectively. The H atoms of O1W and O2W were located in a difference Fourier map and refined with O—H distances approximately equal to 0.98 Å. Atoms H5B and H5C of O1W were treated as disordered and their site occupation set 1/2 due to similar electron density peaks. Finally, the coordinates of H5A, H5B, and H5C of O1W were fixed to stabilize the refinement. The hydroxyl H atoms of the phosphate groups were from difference Fourier maps, they were finally refined with isotropic displacement parameters, using a rotation mode with the HFIX 147 instruction of *SHELXL97*, with O—H distances 0.84 Å and U_{iso}(H21, H22) = 1.5U_{eq} for O4 and O7, respectively. More details and interpretation of the hydrogen bonding situation are given in the _exptl_special_details. All non-hydrogen atoms were refined with anisotropic thermal parameters.

Figures

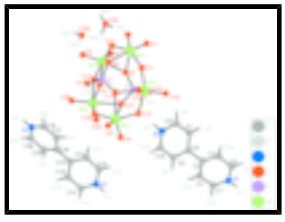


Fig. 1. : The molecular structure of the title compound, with atom labels and with displacement ellipsoids drawn at the 50% probability level, for non-H atoms.

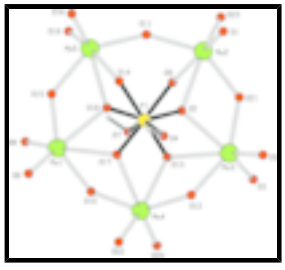


Fig. 2. : The polyanion of the title compound, viewed approximately upon the five-membered Mo skeleton to show the non-crystallographic twofold axis symmetry, the axis running through Mo4 and O11.

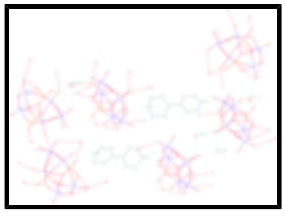


Fig. 3. : A packing diagram of the title compound, viewed along the *a* axis. Dashed lines indicate hydrogen bonds of the type N—H···O and O—H···O.

bis(4,4'-bipyridinium) bis(μ$_5$-hydrogen phosphate)pentakis(μ$_2$-oxido)decaoxidopentamolybdate dihydrate

Crystal data

$(C_{10}H_{10}N_2)_2[Mo_5O_{15}(HPO_4)_2]\cdot 2H_2O$	F_{000} = 4912
M_r = 1264.09	D_x = 2.462 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation

	λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 58657 reflections
a = 11.823 (2) Å	θ = 3.1–27.5º
b = 18.307 (4) Å	μ = 1.99 mm^{-1}
c = 31.517 (6) Å	T = 170 (2) K
V = 6821 (2) $Å^3$	Needle, colorless
Z = 8	0.62 × 0.10 × 0.06 mm

Data collection

Rigaku R-AXIS SPIDER diffractometer	R_{int} = 0.032
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 3.1º
T = 170(2) K	h = −15→13
ω oscillation scans	k = −23→23
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	l = −40→40
T_{min} = 0.788, T_{max} = 0.888	2 standard reflections
58657 measured reflections	every 150 reflections
7810 independent reflections	intensity decay: none
6885 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.020	H-atom parameters constrained
$wR(F^2)$ = 0.052	$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 8.9692P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
7810 reflections	$\Delta\rho_{max}$ = 0.55 e $Å^{-3}$
507 parameters	$\Delta\rho_{min}$ = −0.53 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Due to short H···H contacts detected with *PLATON*, atoms H4, H5B and H22 were omitted in a test-refinement and could be located at approximate positions as before. Additionally, a third H-atom at O1W could be found. Atom H22 at O7 of the second HPO_4 moiety could be rotated away from a short H5B···H22 contact of 1.52 Å by using the BUMP instruction of *SHELXL97*. But then H22 lost its good donor property for the hydrogen bonding contact O7—H22···O1W. Atom H5B is a bifurcated donor for the contacts O1W—H5B···O7 and O1W—H5B···N4. The third H-atom H5C is a donor for the contact O1W—H5C···O23. It is thinkable that there is a kind of flip-flop disorder of H-atoms between H5b, H22 and also H4 of the bipyridine. We decided to reduce the problem by leaving atoms H5B and H5C at their observed positions with site occupation 1/2 each. The possibility of H-atom migration and tautomeric situations can lead to the observed H···H collisions in the measured subcell. The structure can be seen as a superstructure with different hydrogen bonds in adjacent subcells or as a statistical distribution of alternately protonated (O1W) and deprotonated

(N4, O7) atoms. Some weak C—H···O hydrogen bonding contacts with Hbipy as donor molecules and terminal oxygen atoms of the polyanion as acceptors were also observed with *PLATON*. The classic hydrogen bonds including alternative bonds with H5B and H5C as donors are shown in the Table of hydrogen bonding geometry.

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}	Occ. (<1)
Mo1	0.673842 (17)	0.395894 (11)	0.164003 (6)	0.01949 (5)	
Mo2	0.222060 (17)	0.435677 (11)	0.110922 (6)	0.01973 (5)	
Mo3	0.300879 (18)	0.259156 (12)	0.092842 (7)	0.02377 (5)	
Mo4	0.568983 (17)	0.235361 (11)	0.129289 (6)	0.01836 (5)	
Mo5	0.488850 (17)	0.531167 (11)	0.136063 (7)	0.01990 (5)	
P1	0.49905 (5)	0.39040 (3)	0.071272 (19)	0.01954 (12)	
P2	0.39585 (5)	0.35973 (3)	0.182165 (18)	0.01672 (11)	
O1	0.19088 (17)	0.46367 (11)	0.06076 (6)	0.0326 (4)	
O2	0.37835 (14)	0.36919 (9)	0.08454 (5)	0.0219 (3)	
O3	0.24558 (18)	0.18901 (11)	0.12105 (8)	0.0445 (5)	
O4	0.51165 (18)	0.36254 (11)	0.02472 (6)	0.0348 (5)	
H21	0.5561	0.3900	0.0114	0.052*	
O5	0.26267 (18)	0.24150 (13)	0.04182 (7)	0.0436 (5)	
O6	0.80289 (15)	0.38793 (11)	0.14037 (6)	0.0323 (4)	
O7	0.40830 (16)	0.34044 (10)	0.23039 (5)	0.0273 (4)	
H22	0.4292	0.3777	0.2438	0.041*	
O8	0.70150 (17)	0.41885 (11)	0.21538 (6)	0.0329 (4)	
O9	0.28124 (14)	0.39508 (9)	0.17575 (5)	0.0211 (3)	
O10	0.63370 (14)	0.29609 (9)	0.17264 (5)	0.0220 (3)	
O11	0.33624 (14)	0.50730 (9)	0.12380 (5)	0.0224 (4)	
O12	0.45737 (15)	0.22831 (9)	0.08715 (5)	0.0236 (4)	
O13	0.40575 (14)	0.28766 (9)	0.15824 (5)	0.0212 (3)	
O14	0.51224 (15)	0.47256 (9)	0.07319 (5)	0.0236 (4)	
O15	0.64225 (14)	0.49222 (9)	0.14026 (5)	0.0213 (3)	
O16	0.51736 (17)	0.60912 (10)	0.10865 (6)	0.0336 (4)	
O17	0.58737 (14)	0.35249 (9)	0.09946 (5)	0.0217 (3)	
O18	0.48900 (14)	0.41418 (9)	0.17009 (5)	0.0202 (3)	
O19	0.47497 (15)	0.56183 (10)	0.18699 (6)	0.0281 (4)	
O20	0.54467 (15)	0.15868 (10)	0.15847 (6)	0.0264 (4)	
O21	0.19002 (14)	0.33318 (10)	0.10747 (6)	0.0246 (4)	
O22	0.69131 (15)	0.21350 (10)	0.10217 (6)	0.0263 (4)	
O23	0.10681 (15)	0.46518 (10)	0.13920 (6)	0.0293 (4)	

O1W	0.0081 (2)	0.45867 (14)	0.22711 (7)	0.0484 (6)	
H5A	0.0746	0.4565	0.2450	0.058*	
H5B	−0.0590	0.4580	0.2444	0.058*	0.50
H5C	0.0365	0.4571	0.1983	0.058*	0.50
O2W	0.1367 (2)	0.07679 (19)	0.03045 (8)	0.0781 (10)	
H6A	0.2105	0.0628	0.0180	0.094*	
H6B	0.1396	0.0637	0.0609	0.094*	
N1	1.1584 (2)	0.70730 (15)	−0.05533 (7)	0.0388 (6)	
H1	1.2067	0.7286	−0.0726	0.047*	
N2	0.7791 (2)	0.53924 (13)	0.08094 (7)	0.0304 (5)	
H2	0.7310	0.5176	0.0982	0.036*	
N3	1.10428 (19)	0.30064 (13)	0.19184 (8)	0.0331 (5)	
H3	1.1565	0.3317	0.1835	0.040*	
N4	0.6958 (2)	0.05518 (14)	0.25719 (9)	0.0415 (6)	
H4	0.6442	0.0236	0.2655	0.050*	
C1	1.1859 (3)	0.64345 (19)	−0.03824 (9)	0.0388 (7)	
H1A	1.2567	0.6216	−0.0448	0.047*	
C2	1.1126 (2)	0.60884 (16)	−0.01112 (9)	0.0315 (6)	
H2A	1.1324	0.5633	0.0013	0.038*	
C3	1.0093 (2)	0.64131 (14)	−0.00206 (7)	0.0225 (5)	
C4	0.9837 (2)	0.70774 (16)	−0.02057 (9)	0.0339 (6)	
H4A	0.9134	0.7308	−0.0148	0.041*	
C5	1.0605 (3)	0.74020 (18)	−0.04738 (10)	0.0417 (7)	
H5	1.0437	0.7859	−0.0601	0.050*	
C6	0.7391 (2)	0.57677 (16)	0.04779 (9)	0.0311 (6)	
H6	0.6598	0.5798	0.0432	0.037*	
C7	0.8120 (2)	0.61103 (15)	0.02029 (8)	0.0262 (5)	
H7	0.7837	0.6382	−0.0031	0.031*	
C8	0.9276 (2)	0.60545 (13)	0.02716 (7)	0.0213 (5)	
C9	0.9667 (2)	0.56597 (14)	0.06186 (8)	0.0270 (5)	
H9	1.0456	0.5614	0.0670	0.032*	
C10	0.8895 (2)	0.53352 (15)	0.08870 (9)	0.0306 (6)	
H10	0.9152	0.5070	0.1128	0.037*	
C11	1.1214 (2)	0.26356 (15)	0.22744 (9)	0.0291 (6)	
H11	1.1880	0.2714	0.2437	0.035*	
C12	1.0420 (2)	0.21368 (14)	0.24067 (8)	0.0253 (5)	
H12	1.0539	0.1865	0.2660	0.030*	
C13	0.9441 (2)	0.20307 (13)	0.21692 (8)	0.0219 (5)	
C14	0.9291 (2)	0.24402 (16)	0.17998 (9)	0.0313 (6)	
H14	0.8629	0.2382	0.1633	0.038*	
C15	1.0119 (2)	0.29308 (18)	0.16812 (10)	0.0375 (7)	
H15	1.0030	0.3214	0.1431	0.045*	
C16	0.7041 (2)	0.07013 (16)	0.21599 (10)	0.0356 (7)	
H16	0.6547	0.0473	0.1962	0.043*	
C17	0.7839 (2)	0.11843 (15)	0.20234 (9)	0.0294 (6)	
H17	0.7901	0.1295	0.1730	0.035*	
C18	0.8559 (2)	0.15142 (14)	0.23123 (8)	0.0240 (5)	
C19	0.8440 (2)	0.13527 (17)	0.27439 (9)	0.0337 (6)	
H19	0.8914	0.1579	0.2949	0.040*	

C20	0.7622 (3)	0.08594 (19)	0.28668 (10)	0.0422 (7)
H20	0.7530	0.0738	0.3158	0.051*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01621 (10)	0.02008 (10)	0.02219 (10)	−0.00188 (7)	0.00135 (8)	0.00065 (8)
Mo2	0.01688 (10)	0.02155 (10)	0.02075 (10)	0.00192 (7)	0.00012 (8)	0.00032 (8)
Mo3	0.01946 (10)	0.02123 (11)	0.03062 (11)	−0.00034 (8)	−0.00211 (9)	−0.00514 (8)
Mo4	0.01718 (10)	0.01714 (10)	0.02077 (10)	0.00070 (7)	0.00317 (8)	−0.00003 (7)
Mo5	0.01883 (10)	0.01682 (10)	0.02407 (10)	−0.00144 (7)	0.00478 (8)	−0.00049 (8)
P1	0.0208 (3)	0.0208 (3)	0.0170 (3)	0.0005 (2)	0.0042 (2)	0.0008 (2)
P2	0.0161 (3)	0.0168 (3)	0.0172 (3)	−0.0008 (2)	0.0034 (2)	0.0014 (2)
O1	0.0368 (11)	0.0358 (11)	0.0253 (9)	0.0069 (9)	−0.0058 (8)	0.0010 (8)
O2	0.0181 (8)	0.0215 (8)	0.0259 (8)	0.0009 (7)	0.0031 (7)	0.0000 (7)
O3	0.0312 (11)	0.0299 (11)	0.0724 (15)	−0.0077 (9)	0.0030 (11)	0.0052 (10)
O4	0.0471 (12)	0.0362 (11)	0.0212 (9)	−0.0060 (9)	0.0104 (9)	−0.0043 (8)
O5	0.0348 (11)	0.0538 (14)	0.0422 (12)	0.0135 (10)	−0.0133 (10)	−0.0234 (10)
O6	0.0196 (9)	0.0342 (10)	0.0433 (11)	0.0016 (8)	0.0054 (8)	0.0061 (9)
O7	0.0356 (10)	0.0265 (9)	0.0199 (8)	0.0025 (8)	0.0009 (8)	0.0034 (7)
O8	0.0393 (11)	0.0321 (10)	0.0271 (9)	−0.0067 (9)	−0.0053 (8)	−0.0007 (8)
O9	0.0176 (8)	0.0234 (9)	0.0222 (8)	0.0003 (6)	0.0037 (7)	0.0029 (7)
O10	0.0220 (8)	0.0210 (8)	0.0229 (8)	0.0004 (7)	−0.0002 (7)	0.0016 (7)
O11	0.0199 (8)	0.0203 (8)	0.0272 (9)	0.0020 (7)	0.0025 (7)	0.0006 (7)
O12	0.0234 (9)	0.0233 (9)	0.0242 (8)	0.0023 (7)	0.0001 (7)	−0.0056 (7)
O13	0.0216 (8)	0.0186 (8)	0.0233 (8)	−0.0008 (7)	0.0049 (7)	−0.0018 (7)
O14	0.0275 (9)	0.0212 (9)	0.0219 (8)	−0.0006 (7)	0.0044 (7)	0.0024 (7)
O15	0.0172 (8)	0.0201 (8)	0.0264 (8)	−0.0031 (6)	0.0053 (7)	0.0014 (7)
O16	0.0355 (11)	0.0233 (10)	0.0421 (11)	−0.0030 (8)	0.0092 (9)	0.0046 (8)
O17	0.0183 (8)	0.0231 (8)	0.0237 (8)	0.0009 (7)	0.0027 (7)	0.0014 (7)
O18	0.0175 (8)	0.0173 (8)	0.0259 (8)	−0.0022 (6)	0.0020 (7)	0.0010 (7)
O19	0.0245 (9)	0.0283 (10)	0.0315 (9)	−0.0022 (7)	0.0050 (8)	−0.0094 (8)
O20	0.0278 (9)	0.0223 (9)	0.0293 (9)	−0.0012 (7)	0.0033 (8)	0.0035 (7)
O21	0.0160 (8)	0.0255 (9)	0.0323 (9)	−0.0018 (7)	0.0009 (7)	−0.0022 (7)
O22	0.0243 (9)	0.0246 (9)	0.0300 (9)	0.0025 (7)	0.0095 (8)	0.0010 (7)
O23	0.0206 (9)	0.0343 (10)	0.0331 (10)	0.0052 (8)	0.0008 (8)	−0.0014 (8)
O1W	0.0473 (14)	0.0580 (15)	0.0398 (12)	0.0031 (11)	0.0049 (10)	0.0056 (11)
O2W	0.0599 (17)	0.127 (3)	0.0471 (15)	0.0313 (18)	0.0066 (13)	0.0466 (17)
N1	0.0390 (14)	0.0514 (16)	0.0259 (12)	−0.0203 (12)	0.0101 (11)	0.0036 (11)
N2	0.0302 (12)	0.0294 (12)	0.0314 (12)	−0.0051 (9)	0.0138 (10)	0.0022 (10)
N3	0.0209 (11)	0.0327 (12)	0.0458 (14)	−0.0022 (9)	0.0081 (10)	0.0101 (11)
N4	0.0327 (13)	0.0318 (13)	0.0598 (17)	−0.0071 (11)	0.0152 (13)	0.0056 (12)
C1	0.0270 (14)	0.0536 (19)	0.0358 (15)	−0.0034 (13)	0.0124 (13)	−0.0033 (14)
C2	0.0282 (14)	0.0348 (15)	0.0316 (14)	−0.0001 (11)	0.0080 (12)	0.0024 (11)
C3	0.0210 (12)	0.0274 (13)	0.0191 (11)	−0.0056 (10)	0.0020 (10)	−0.0020 (9)
C4	0.0302 (14)	0.0318 (14)	0.0397 (15)	−0.0005 (12)	0.0057 (12)	0.0054 (12)
C5	0.0482 (19)	0.0388 (17)	0.0382 (16)	−0.0099 (14)	0.0037 (15)	0.0121 (13)
C6	0.0222 (13)	0.0364 (15)	0.0347 (14)	−0.0035 (11)	0.0053 (12)	−0.0026 (12)

C7	0.0230 (13)	0.0321 (14)	0.0234 (12)	−0.0020 (10)	0.0009 (10)	−0.0005 (10)
C8	0.0220 (12)	0.0207 (12)	0.0211 (11)	−0.0022 (9)	0.0049 (10)	−0.0029 (9)
C9	0.0231 (13)	0.0279 (13)	0.0299 (13)	0.0016 (10)	0.0038 (11)	0.0025 (11)
C10	0.0327 (15)	0.0310 (14)	0.0281 (13)	0.0003 (11)	0.0049 (12)	0.0062 (11)
C11	0.0218 (12)	0.0298 (14)	0.0357 (14)	0.0006 (10)	0.0018 (11)	−0.0015 (11)
C12	0.0244 (12)	0.0239 (12)	0.0276 (12)	0.0021 (10)	0.0021 (10)	0.0001 (10)
C13	0.0221 (12)	0.0196 (11)	0.0239 (11)	0.0022 (9)	0.0052 (10)	−0.0018 (9)
C14	0.0236 (13)	0.0400 (16)	0.0303 (14)	−0.0012 (11)	−0.0012 (11)	0.0065 (12)
C15	0.0301 (15)	0.0444 (17)	0.0380 (15)	−0.0010 (13)	0.0040 (12)	0.0174 (13)
C16	0.0285 (14)	0.0295 (14)	0.0489 (17)	−0.0025 (11)	0.0062 (13)	−0.0090 (13)
C17	0.0244 (13)	0.0289 (13)	0.0348 (14)	0.0008 (11)	0.0044 (11)	−0.0053 (11)
C18	0.0196 (12)	0.0215 (12)	0.0308 (12)	0.0033 (9)	0.0042 (10)	0.0001 (10)
C19	0.0265 (14)	0.0435 (17)	0.0313 (14)	−0.0006 (12)	0.0039 (12)	0.0077 (12)
C20	0.0342 (16)	0.0523 (19)	0.0401 (16)	0.0019 (14)	0.0115 (14)	0.0157 (15)

Geometric parameters (Å, °)

Mo1—O6	1.7041 (19)	N1—C5	1.328 (4)
Mo1—O8	1.7045 (19)	N1—H1	0.88
Mo1—O10	1.9073 (17)	N2—C10	1.332 (4)
Mo1—O15	1.9518 (17)	N2—C6	1.337 (4)
Mo1—O18	2.2191 (17)	N2—H2	0.88
Mo1—O17	2.4114 (17)	N3—C11	1.327 (4)
Mo2—O1	1.7024 (19)	N3—C15	1.330 (4)
Mo2—O23	1.7155 (18)	N3—H3	0.88
Mo2—O21	1.9174 (18)	N4—C16	1.331 (4)
Mo2—O11	1.9251 (18)	N4—C20	1.341 (4)
Mo2—O9	2.2839 (17)	N4—H4	0.88
Mo2—O2	2.3637 (17)	C1—C2	1.372 (4)
Mo3—O3	1.693 (2)	C1—H1A	0.95
Mo3—O5	1.701 (2)	C2—C3	1.389 (4)
Mo3—O21	1.9409 (18)	C2—H2A	0.95
Mo3—O12	1.9427 (18)	C3—C4	1.382 (4)
Mo3—O2	2.2283 (17)	C3—C8	1.487 (3)
Mo3—O13	2.4613 (17)	C4—C5	1.376 (4)
Mo4—O20	1.7026 (17)	C4—H4A	0.95
Mo4—O22	1.7270 (17)	C5—H5	0.95
Mo4—O12	1.8766 (18)	C6—C7	1.374 (4)
Mo4—O10	1.9204 (17)	C6—H6	0.95
Mo4—O13	2.3396 (17)	C7—C8	1.387 (4)
Mo4—O17	2.3515 (17)	C7—H7	0.95
Mo5—O16	1.7020 (18)	C8—C9	1.390 (4)
Mo5—O19	1.7084 (18)	C9—C10	1.379 (4)
Mo5—O11	1.8962 (18)	C9—H9	0.95
Mo5—O15	1.9532 (17)	C10—H10	0.95
Mo5—O14	2.2703 (17)	C11—C12	1.374 (4)
Mo5—O18	2.3952 (17)	C11—H11	0.95
P1—O14	1.5134 (18)	C12—C13	1.391 (4)
P1—O17	1.5365 (18)	C12—H12	0.95

P1—O2	1.5369 (17)	C13—C14	1.396 (4)
P1—O4	1.5606 (18)	C13—C18	1.479 (3)
P2—O9	1.5152 (17)	C14—C15	1.380 (4)
P2—O13	1.5242 (17)	C14—H14	0.95
P2—O18	1.5333 (17)	C15—H15	0.95
P2—O7	1.5674 (17)	C16—C17	1.363 (4)
O4—H21	0.84	C16—H16	0.95
O7—H22	0.84	C17—C18	1.385 (4)
O1W—H5A	0.97	C17—H17	0.95
O1W—H5B	0.96	C18—C19	1.399 (4)
O1W—H5C	0.97	C19—C20	1.379 (4)
O2W—H6A	0.99	C19—H19	0.95
O2W—H6B	0.99	C20—H20	0.95
N1—C1	1.328 (4)		
O6—Mo1—O8	105.34 (10)	P2—O9—Mo2	122.16 (9)
O6—Mo1—O10	101.73 (8)	Mo1—O10—Mo4	123.52 (9)
O8—Mo1—O10	98.53 (8)	Mo5—O11—Mo2	150.13 (10)
O6—Mo1—O15	94.65 (8)	Mo4—O12—Mo3	125.75 (9)
O8—Mo1—O15	100.26 (8)	P2—O13—Mo4	127.63 (10)
O10—Mo1—O15	150.77 (7)	P2—O13—Mo3	123.99 (9)
O6—Mo1—O18	158.81 (8)	Mo4—O13—Mo3	90.12 (6)
O8—Mo1—O18	93.99 (8)	P1—O14—Mo5	119.48 (9)
O10—Mo1—O18	83.52 (7)	Mo1—O15—Mo5	122.24 (8)
O15—Mo1—O18	73.04 (6)	P1—O17—Mo4	125.47 (9)
O6—Mo1—O17	89.01 (8)	P1—O17—Mo1	128.83 (10)
O8—Mo1—O17	164.57 (8)	Mo4—O17—Mo1	90.13 (6)
O10—Mo1—O17	72.50 (6)	P2—O18—Mo1	129.10 (10)
O15—Mo1—O17	83.87 (6)	P2—O18—Mo5	133.78 (10)
O18—Mo1—O17	72.85 (6)	Mo1—O18—Mo5	95.56 (6)
O1—Mo2—O23	102.46 (9)	Mo2—O21—Mo3	124.29 (9)
O1—Mo2—O21	101.49 (9)	H5A—O1W—H5B	109.9
O23—Mo2—O21	100.41 (8)	H5A—O1W—H5C	105.2
O1—Mo2—O11	98.20 (9)	H5B—O1W—H5C	144.7
O23—Mo2—O11	103.47 (8)	H6A—O2W—H6B	106.9
O21—Mo2—O11	144.79 (7)	C1—N1—C5	122.5 (3)
O1—Mo2—O9	174.32 (8)	C1—N1—H1	118.8
O23—Mo2—O9	83.16 (8)	C5—N1—H1	118.8
O21—Mo2—O9	78.04 (7)	C10—N2—C6	122.0 (2)
O11—Mo2—O9	79.53 (7)	C10—N2—H2	119.0
O1—Mo2—O2	89.87 (8)	C6—N2—H2	119.0
O23—Mo2—O2	164.98 (8)	C11—N3—C15	123.2 (2)
O21—Mo2—O2	68.32 (6)	C11—N3—H3	118.4
O11—Mo2—O2	82.92 (7)	C15—N3—H3	118.4
O9—Mo2—O2	84.70 (6)	C16—N4—C20	123.2 (3)
O3—Mo3—O5	104.46 (12)	C16—N4—H4	118.4
O3—Mo3—O21	98.28 (9)	C20—N4—H4	118.4
O5—Mo3—O21	100.25 (9)	N1—C1—C2	120.3 (3)
O3—Mo3—O12	101.29 (9)	N1—C1—H1A	119.9
O5—Mo3—O12	96.34 (9)	C2—C1—H1A	119.9

O21—Mo3—O12	150.23 (7)	C1—C2—C3	119.1 (3)
O3—Mo3—O2	154.95 (9)	C1—C2—H2A	120.5
O5—Mo3—O2	99.78 (10)	C3—C2—H2A	120.5
O21—Mo3—O2	70.99 (7)	C4—C3—C2	118.8 (2)
O12—Mo3—O2	81.98 (7)	C4—C3—C8	120.5 (2)
O3—Mo3—O13	85.16 (9)	C2—C3—C8	120.7 (2)
O5—Mo3—O13	164.92 (9)	C5—C4—C3	119.7 (3)
O21—Mo3—O13	89.61 (7)	C5—C4—H4A	120.2
O12—Mo3—O13	70.08 (6)	C3—C4—H4A	120.2
O2—Mo3—O13	72.52 (6)	N1—C5—C4	119.7 (3)
O20—Mo4—O22	102.57 (9)	N1—C5—H5	120.2
O20—Mo4—O12	101.94 (8)	C4—C5—H5	120.2
O22—Mo4—O12	102.86 (8)	N2—C6—C7	120.4 (3)
O20—Mo4—O10	99.22 (8)	N2—C6—H6	119.8
O22—Mo4—O10	98.78 (8)	C7—C6—H6	119.8
O12—Mo4—O10	145.43 (7)	C6—C7—C8	119.1 (2)
O20—Mo4—O13	89.28 (7)	C6—C7—H7	120.5
O22—Mo4—O13	168.14 (7)	C8—C7—H7	120.5
O12—Mo4—O13	73.99 (7)	C7—C8—C9	119.3 (2)
O10—Mo4—O13	79.29 (7)	C7—C8—C3	120.7 (2)
O20—Mo4—O17	169.55 (7)	C9—C8—C3	120.0 (2)
O22—Mo4—O17	86.33 (7)	C10—C9—C8	119.1 (3)
O12—Mo4—O17	81.07 (7)	C10—C9—H9	120.4
O10—Mo4—O17	73.72 (7)	C8—C9—H9	120.4
O13—Mo4—O17	81.89 (6)	N2—C10—C9	120.1 (3)
O16—Mo5—O19	102.73 (9)	N2—C10—H10	119.9
O16—Mo5—O11	106.15 (9)	C9—C10—H10	119.9
O19—Mo5—O11	100.13 (8)	N3—C11—C12	119.5 (3)
O16—Mo5—O15	99.01 (8)	N3—C11—H11	120.2
O19—Mo5—O15	98.37 (8)	C12—C11—H11	120.2
O11—Mo5—O15	144.41 (7)	C11—C12—C13	119.8 (2)
O16—Mo5—O14	85.93 (8)	C11—C12—H12	120.1
O19—Mo5—O14	170.78 (8)	C13—C12—H12	120.1
O11—Mo5—O14	80.16 (7)	C12—C13—C14	118.6 (2)
O15—Mo5—O14	76.91 (7)	C12—C13—C18	120.8 (2)
O16—Mo5—O18	167.66 (8)	C14—C13—C18	120.5 (2)
O19—Mo5—O18	82.72 (8)	C15—C14—C13	119.0 (3)
O11—Mo5—O18	83.45 (6)	C15—C14—H14	120.5
O15—Mo5—O18	69.06 (6)	C13—C14—H14	120.5
O14—Mo5—O18	88.17 (6)	N3—C15—C14	119.8 (3)
O14—P1—O17	110.84 (10)	N3—C15—H15	120.1
O14—P1—O2	109.63 (10)	C14—C15—H15	120.1
O17—P1—O2	111.07 (9)	N4—C16—C17	119.5 (3)
O14—P1—O4	110.65 (11)	N4—C16—H16	120.2
O17—P1—O4	109.33 (11)	C17—C16—H16	120.2
O2—P1—O4	105.19 (11)	C16—C17—C18	120.1 (3)
O9—P2—O13	111.86 (10)	C16—C17—H17	120.0
O9—P2—O18	109.36 (10)	C18—C17—H17	120.0
O13—P2—O18	112.63 (10)	C17—C18—C19	119.0 (2)

O9—P2—O7	108.03 (10)	C17—C18—C13	120.8 (2)
O13—P2—O7	106.11 (10)	C19—C18—C13	120.2 (2)
O18—P2—O7	108.65 (10)	C20—C19—C18	118.8 (3)
P1—O2—Mo3	129.94 (10)	C20—C19—H19	120.6
P1—O2—Mo2	133.75 (10)	C18—C19—H19	120.6
Mo3—O2—Mo2	95.91 (6)	N4—C20—C19	119.4 (3)
P1—O4—H21	109.5	N4—C20—H20	120.3
P2—O7—H22	109.5	C19—C20—H20	120.3
O14—P1—O2—Mo3	−176.12 (11)	O11—Mo5—O15—Mo1	−39.14 (17)
O17—P1—O2—Mo3	−53.29 (15)	O14—Mo5—O15—Mo1	−90.36 (10)
O4—P1—O2—Mo3	64.89 (15)	O18—Mo5—O15—Mo1	2.73 (8)
O14—P1—O2—Mo2	−5.27 (16)	O14—P1—O17—Mo4	155.65 (10)
O17—P1—O2—Mo2	117.56 (13)	O2—P1—O17—Mo4	33.52 (14)
O4—P1—O2—Mo2	−124.26 (14)	O4—P1—O17—Mo4	−82.12 (13)
O3—Mo3—O2—P1	110.1 (2)	O14—P1—O17—Mo1	30.40 (15)
O5—Mo3—O2—P1	−84.55 (14)	O2—P1—O17—Mo1	−91.73 (13)
O21—Mo3—O2—P1	177.89 (15)	O4—P1—O17—Mo1	152.64 (11)
O12—Mo3—O2—P1	10.55 (13)	O20—Mo4—O17—P1	−89.7 (4)
O13—Mo3—O2—P1	82.10 (13)	O22—Mo4—O17—P1	121.51 (13)
O3—Mo3—O2—Mo2	−63.3 (2)	O12—Mo4—O17—P1	17.86 (11)
O5—Mo3—O2—Mo2	102.08 (8)	O10—Mo4—O17—P1	−138.24 (13)
O21—Mo3—O2—Mo2	4.52 (6)	O13—Mo4—O17—P1	−57.09 (11)
O12—Mo3—O2—Mo2	−162.82 (7)	O20—Mo4—O17—Mo1	50.8 (4)
O13—Mo3—O2—Mo2	−91.27 (6)	O22—Mo4—O17—Mo1	−98.01 (7)
O1—Mo2—O2—P1	79.95 (15)	O12—Mo4—O17—Mo1	158.35 (7)
O23—Mo2—O2—P1	−134.6 (3)	O10—Mo4—O17—Mo1	2.25 (6)
O21—Mo2—O2—P1	−177.62 (16)	O13—Mo4—O17—Mo1	83.40 (6)
O11—Mo2—O2—P1	−18.33 (13)	O6—Mo1—O17—P1	−121.35 (13)
O9—Mo2—O2—P1	−98.41 (14)	O8—Mo1—O17—P1	79.9 (3)
O1—Mo2—O2—Mo3	−107.09 (8)	O10—Mo1—O17—P1	136.03 (13)
O23—Mo2—O2—Mo3	38.4 (3)	O15—Mo1—O17—P1	−26.56 (12)
O21—Mo2—O2—Mo3	−4.66 (7)	O18—Mo1—O17—P1	47.55 (11)
O11—Mo2—O2—Mo3	154.63 (7)	O6—Mo1—O17—Mo4	100.35 (8)
O9—Mo2—O2—Mo3	74.56 (6)	O8—Mo1—O17—Mo4	−58.4 (3)
O13—P2—O9—Mo2	−64.72 (13)	O10—Mo1—O17—Mo4	−2.28 (6)
O18—P2—O9—Mo2	60.76 (13)	O15—Mo1—O17—Mo4	−164.87 (6)
O7—P2—O9—Mo2	178.85 (10)	O18—Mo1—O17—Mo4	−90.76 (6)
O1—Mo2—O9—P2	−4.4 (9)	O9—P2—O18—Mo1	−171.70 (11)
O23—Mo2—O9—P2	−176.36 (13)	O13—P2—O18—Mo1	−46.66 (15)
O21—Mo2—O9—P2	81.43 (12)	O7—P2—O18—Mo1	70.59 (14)
O11—Mo2—O9—P2	−71.25 (11)	O9—P2—O18—Mo5	−9.58 (16)
O2—Mo2—O9—P2	12.51 (11)	O13—P2—O18—Mo5	115.46 (13)
O6—Mo1—O10—Mo4	−81.83 (12)	O7—P2—O18—Mo5	−127.29 (13)
O8—Mo1—O10—Mo4	170.45 (11)	O6—Mo1—O18—P2	112.6 (2)
O15—Mo1—O10—Mo4	40.9 (2)	O8—Mo1—O18—P2	−91.35 (14)
O18—Mo1—O10—Mo4	77.36 (10)	O10—Mo1—O18—P2	6.79 (12)
O17—Mo1—O10—Mo4	3.35 (9)	O15—Mo1—O18—P2	169.12 (14)
O20—Mo4—O10—Mo1	−175.50 (11)	O17—Mo1—O18—P2	80.43 (12)
O22—Mo4—O10—Mo1	80.13 (11)	O6—Mo1—O18—Mo5	−54.5 (2)

O12—Mo4—O10—Mo1	−48.27 (18)	O8—Mo1—O18—Mo5	101.52 (8)
O13—Mo4—O10—Mo1	−88.00 (10)	O10—Mo1—O18—Mo5	−160.34 (7)
O17—Mo4—O10—Mo1	−3.41 (9)	O15—Mo1—O18—Mo5	1.99 (6)
O16—Mo5—O11—Mo2	139.11 (19)	O17—Mo1—O18—Mo5	−86.70 (6)
O19—Mo5—O11—Mo2	−114.3 (2)	O16—Mo5—O18—P2	−153.0 (3)
O15—Mo5—O11—Mo2	5.9 (3)	O19—Mo5—O18—P2	89.92 (14)
O14—Mo5—O11—Mo2	56.30 (19)	O11—Mo5—O18—P2	−11.21 (13)
O18—Mo5—O11—Mo2	−32.97 (19)	O15—Mo5—O18—P2	−168.19 (15)
O1—Mo2—O11—Mo5	−114.2 (2)	O14—Mo5—O18—P2	−91.51 (14)
O23—Mo2—O11—Mo5	140.78 (19)	O16—Mo5—O18—Mo1	13.2 (4)
O21—Mo2—O11—Mo5	9.4 (3)	O19—Mo5—O18—Mo1	−103.93 (8)
O9—Mo2—O11—Mo5	60.50 (19)	O11—Mo5—O18—Mo1	154.94 (7)
O2—Mo2—O11—Mo5	−25.39 (19)	O15—Mo5—O18—Mo1	−2.04 (6)
O20—Mo4—O12—Mo3	83.35 (12)	O14—Mo5—O18—Mo1	74.64 (6)
O22—Mo4—O12—Mo3	−170.60 (11)	O1—Mo2—O21—Mo3	91.66 (12)
O10—Mo4—O12—Mo3	−43.20 (19)	O23—Mo2—O21—Mo3	−163.19 (11)
O13—Mo4—O12—Mo3	−2.40 (10)	O11—Mo2—O21—Mo3	−31.05 (19)
O17—Mo4—O12—Mo3	−86.47 (11)	O9—Mo2—O21—Mo3	−82.55 (11)
O3—Mo3—O12—Mo4	−78.23 (14)	O2—Mo2—O21—Mo3	6.45 (9)
O5—Mo3—O12—Mo4	175.61 (13)	O3—Mo3—O21—Mo2	149.94 (13)
O21—Mo3—O12—Mo4	51.9 (2)	O5—Mo3—O21—Mo2	−103.64 (13)
O2—Mo3—O12—Mo4	76.57 (11)	O12—Mo3—O21—Mo2	19.2 (2)
O13—Mo3—O12—Mo4	2.34 (9)	O2—Mo3—O21—Mo2	−6.72 (10)
O9—P2—O13—Mo4	149.46 (10)	O13—Mo3—O21—Mo2	64.89 (11)
O18—P2—O13—Mo4	25.80 (15)	C5—N1—C1—C2	−0.2 (5)
O7—P2—O13—Mo4	−92.95 (13)	N1—C1—C2—C3	0.4 (4)
O9—P2—O13—Mo3	27.92 (13)	C1—C2—C3—C4	−0.2 (4)
O18—P2—O13—Mo3	−95.74 (12)	C1—C2—C3—C8	180.0 (3)
O7—P2—O13—Mo3	145.51 (10)	C2—C3—C4—C5	−0.1 (4)
O20—Mo4—O13—P2	123.86 (13)	C8—C3—C4—C5	179.7 (3)
O22—Mo4—O13—P2	−57.4 (4)	C1—N1—C5—C4	−0.1 (5)
O12—Mo4—O13—P2	−133.50 (13)	C3—C4—C5—N1	0.2 (5)
O10—Mo4—O13—P2	24.33 (12)	C10—N2—C6—C7	0.1 (4)
O17—Mo4—O13—P2	−50.53 (12)	N2—C6—C7—C8	0.7 (4)
O20—Mo4—O13—Mo3	−101.10 (7)	C6—C7—C8—C9	−0.6 (4)
O22—Mo4—O13—Mo3	77.7 (4)	C6—C7—C8—C3	179.6 (2)
O12—Mo4—O13—Mo3	1.54 (6)	C4—C3—C8—C7	35.2 (4)
O10—Mo4—O13—Mo3	159.37 (7)	C2—C3—C8—C7	−145.0 (3)
O17—Mo4—O13—Mo3	84.51 (6)	C4—C3—C8—C9	−144.6 (3)
O3—Mo3—O13—P2	−120.11 (13)	C2—C3—C8—C9	35.2 (4)
O5—Mo3—O13—P2	109.4 (3)	C7—C8—C9—C10	−0.2 (4)
O21—Mo3—O13—P2	−21.76 (11)	C3—C8—C9—C10	179.6 (2)
O12—Mo3—O13—P2	136.03 (12)	C6—N2—C10—C9	−0.9 (4)
O2—Mo3—O13—P2	48.40 (11)	C8—C9—C10—N2	1.0 (4)
O3—Mo3—O13—Mo4	102.35 (9)	C15—N3—C11—C12	1.2 (4)
O5—Mo3—O13—Mo4	−28.1 (4)	N3—C11—C12—C13	−0.7 (4)
O21—Mo3—O13—Mo4	−159.31 (7)	C11—C12—C13—C14	−0.2 (4)
O12—Mo3—O13—Mo4	−1.52 (6)	C11—C12—C13—C18	−178.0 (2)
O2—Mo3—O13—Mo4	−89.15 (6)	C12—C13—C14—C15	0.5 (4)

O17—P1—O14—Mo5	−61.89 (13)	C18—C13—C14—C15	178.4 (3)
O2—P1—O14—Mo5	61.08 (13)	C11—N3—C15—C14	−0.9 (5)
O4—P1—O14—Mo5	176.65 (11)	C13—C14—C15—N3	0.0 (5)
O16—Mo5—O14—P1	179.84 (13)	C20—N4—C16—C17	0.5 (5)
O19—Mo5—O14—P1	19.6 (5)	N4—C16—C17—C18	0.2 (4)
O11—Mo5—O14—P1	−72.97 (12)	C16—C17—C18—C19	−1.0 (4)
O15—Mo5—O14—P1	79.61 (12)	C16—C17—C18—C13	179.0 (2)
O18—Mo5—O14—P1	10.69 (11)	C12—C13—C18—C17	−154.0 (2)
O6—Mo1—O15—Mo5	159.52 (11)	C14—C13—C18—C17	28.2 (4)
O8—Mo1—O15—Mo5	−93.95 (11)	C12—C13—C18—C19	26.0 (4)
O10—Mo1—O15—Mo5	35.3 (2)	C14—C13—C18—C19	−151.8 (3)
O18—Mo1—O15—Mo5	−2.88 (9)	C17—C18—C19—C20	1.2 (4)
O17—Mo1—O15—Mo5	71.02 (10)	C13—C18—C19—C20	−178.9 (3)
O16—Mo5—O15—Mo1	−174.01 (11)	C16—N4—C20—C19	−0.4 (5)
O19—Mo5—O15—Mo1	81.58 (11)	C18—C19—C20—N4	−0.5 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H5A···O8^{i}	0.97	2.07	3.008 (3)	162
O1W—H5B···N4^{ii}	0.96	2.44	3.135 (4)	129
O2W—H6A···O1^{iii}	0.99	2.54	3.059 (3)	112
O2W—H6B···O11^{iii}	0.99	2.25	3.221 (3)	166
O2W—H6B···O16^{iii}	0.99	2.53	3.121 (4)	118
N1—H1···O22^{iv}	0.88	1.86	2.728 (3)	170
N3—H3···O9^{v}	0.88	1.89	2.761 (3)	169
N3—H3···O21^{v}	0.88	2.43	2.907 (3)	115
N4—H4···O19^{vi}	0.88	2.17	2.680 (3)	116
N4—H4···O1W^{iii}	0.88	2.47	3.135 (4)	132
O4—H21···O2W^{vii}	0.84	1.74	2.538 (3)	159
O7—H22···O1W^{viii}	0.84	1.98	2.805 (3)	169
N2—H2···O15	0.88	1.75	2.618 (3)	166
O1W—H5C···O23	0.97	2.05	3.009 (3)	173

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

Fig. 1

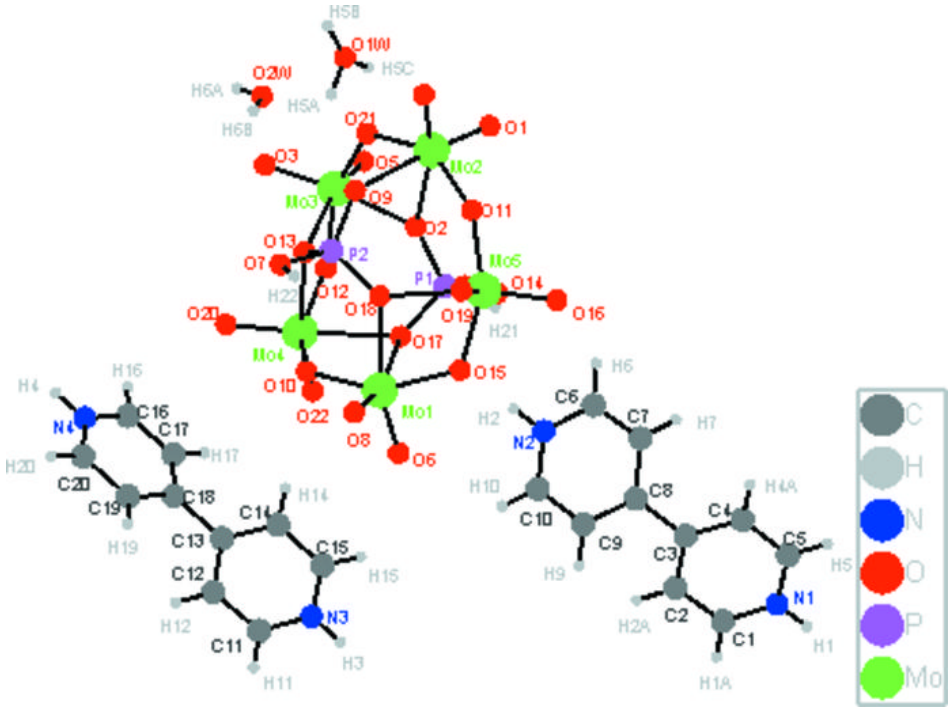

H5B
O1W
H5C
O2W
H6A
H5A
H6B
O21
O1
O3
O5
Mo2
Mo3
O9
O11
O2
O13
P2
O7
P1
Mo5
H22
O12
O18
O14
O16
O20
O19
H21
O17
Mo4
O15
O10
O22
Mo1
O8
O6
H4
H16
C16
N4
C17
H17
H20
C20
C19
C18
H14
C14
H19
C13
C15
H15
C12
N3
H12
C11
H3
H11
H6
C6
C7
H7
H2
N2
H10
C8
H4A
C10
C4
C9
C5
H5
C3
N1
H9
C2
H2A
C1
H1
H1A
C
H
N
O
P
Mo

Fig. 2

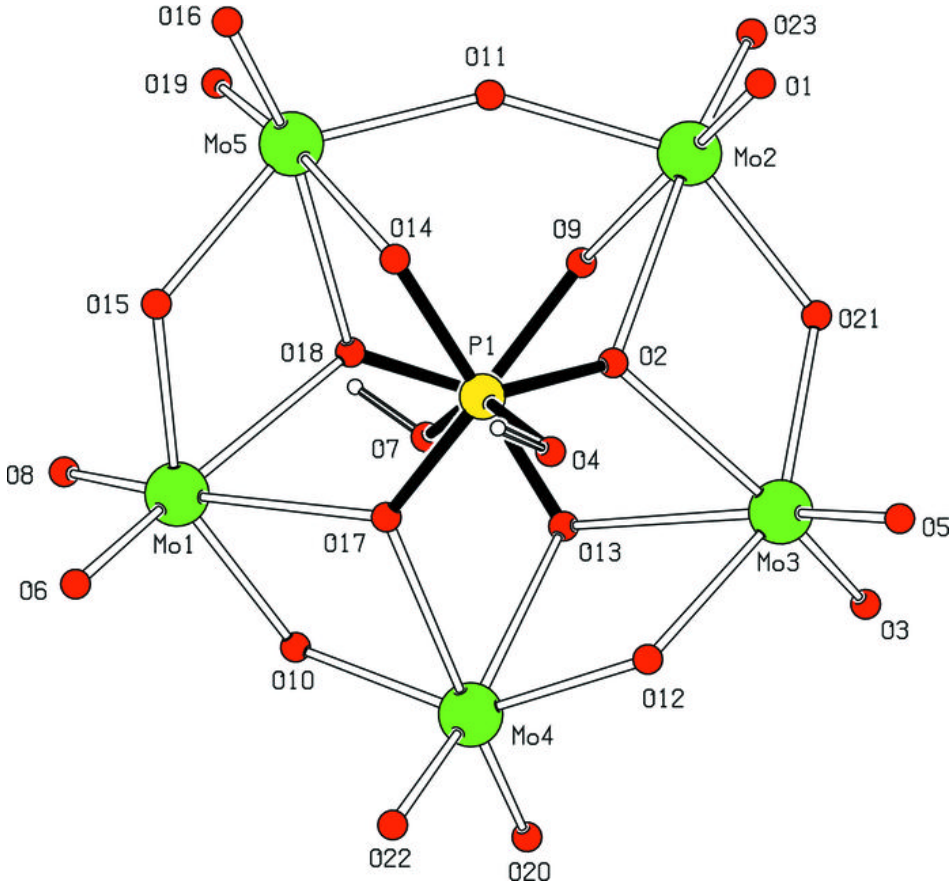

O16
O19
O11
O23
O1
Mo5
Mo2
O14
O9
O15
O21
O18
P1
O2
O7
O4
O8
Mo1
O17
O13
Mo3
O5
O6
O3
O10
O12
Mo4
O22
O20

Fig. 3