Octakis(dibutylammonium) decamolybdate(VI)

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In the title salt, $(C_8H_{20}N)_8[Mo_{10}O_{34}]$, the $[Mo_{10}O_{34}]^{8-}$ polyanion is located about an inversion centre and can be considered as a β-type octamolybdate anion to which two additional MoO_4 tetrahedra are linked via common corners. The $[Mo_{10}O_{34}]^{8-}$ polyanions are packed in rows extending parallel to [001] and are connected to the dibutylammonium counter-cations through N—H···O hydrogen-bonding interactions.

3D view

Chemical scheme
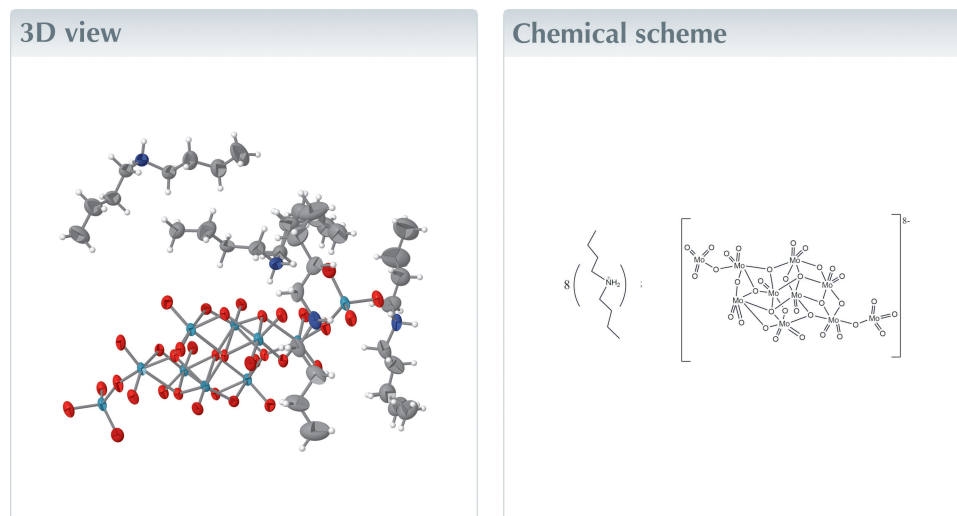
Structure description

Polyoxometalates (POMs) are obtained by self-assembly of transition-metal oxide units $[MO_n]^{p-}$ in acidic media (M = metal; n = 3, 4, 6, ⋯; p = 0, 1, 2, 3, ⋯). POMs and their derivatives are an important group of materials that have attracted considerable interest in areas such as electrochemistry (Zhang et al., 2021), materials science (Hao et al., 2007; Li et al., 2007), and medicine (Cronin et al., 2002; Müller et al., 1999). In recent years, research on organic–inorganic hybrid POMs has experienced significant growth, supported by possible modifications and/or functionalizations of the oxide surface of the POM with preselected organic moieties (Xu et al., 2003). The structural diversity of the corresponding isopolyoxomolybdates is due to characteristic large polyanionic units and organic ammonium cations, which consolidate the crystal structures through non-covalent supramolecular interactions. In this regard, several octamolybdate polyanions $[Mo_8O_{26}]^{4-}$, charge-balanced by organic counter-ions, have been synthesized and structurally characterized (Allis et al., 2004; Harchani & Haddad, 2015). For the current study, we used diisobutylammonium as a counter-cation and obtained the hybrid organic–inorganic decamolybdate $(C_8H_{20}N)_8[Mo_{10}O_{34}]$.

The asymmetric unit of $(C_8H_{20}N)_8[Mo_{10}O_{34}]$ is shown in Fig. 1. The $[Mo_{10}O_{34}]^{8-}$ anion is located about an inversion centre and is displayed in Fig. 2. Such kind of deca-
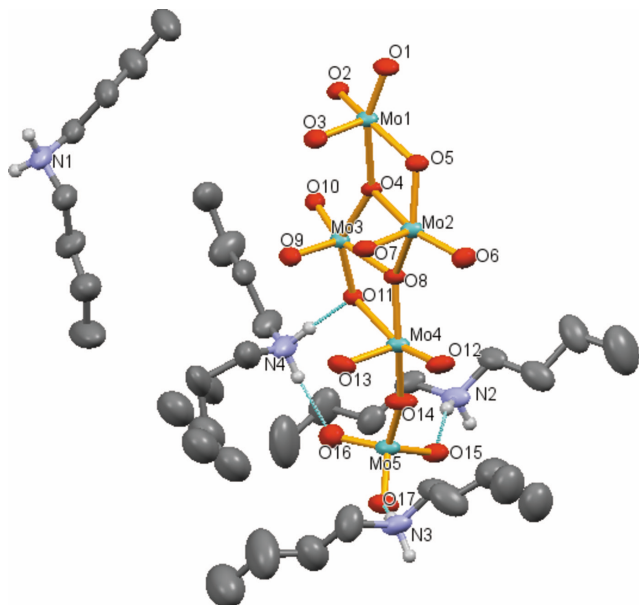
Figure 1
The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Dotted lines indicate N—H···O hydrogen-bonding interactions. The C-bound H atoms are omitted for clarity.

molybdate anion is known from other ammonium salts and has been reported for the first time for $(NH_4)_8[Mo_{10}O_{34}]$ (Fuchs *et al.*, 1975). The $[Mo_{10}O_{34}]^{8-}$ anion can be considered as a β-type octamolydate to which two additional MoO_4 tetrahedra are added *via* vertex-sharing. Two types of β-octamolybdate anions can be distinguished, type *A* with the
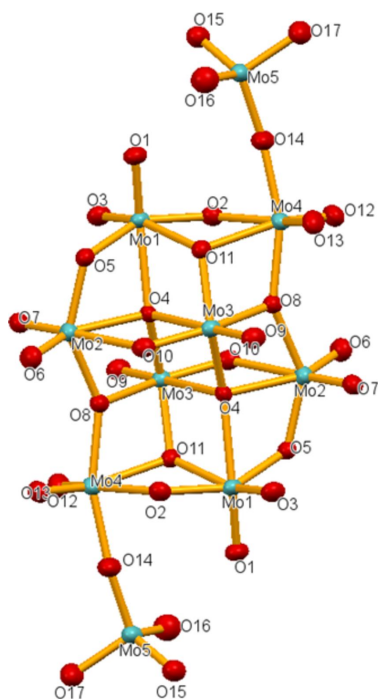
Table 1
Hydrogen-bond geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
N1—H1$A\cdots$O2^i	0.91	1.72	2.627 (6)	172
N1—H1$B\cdots$O10^i	0.91	1.97	2.755 (6)	143
N2—H2$C\cdots$O15	0.91	1.91	2.778 (6)	160
N2—H2$D\cdots$O7^{ii}	0.91	1.90	2.812 (6)	176
N3—H3$A\cdots$O15^{iii}	0.91	1.87	2.770 (6)	172
N3—H3$B\cdots$O17	0.91	1.88	2.786 (6)	176
N4—H4$A\cdots$O11	0.91	1.80	2.695 (6)	166
N4—H4$B\cdots$O16	0.91	1.86	2.762 (7)	172

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y+1, -z+2$.

general formula $[Mo_8O_{26}]^{4-}$ and type *B* with the general formula $[H_xMo_8O_{28}]^{(8-x)}$ (Pavani *et al.*, 2007). Thus, the $[Mo_{10}O_{34}]^{8-}$ anion of the title compound can be considered as of the β-octamolybdate *B* type (Du *et al.* 2011; Isobe *et al.* 1978).

The $[Mo_{10}O_{34}]^{8-}$ polyanion is made up of eight MoO_6 octahedra linked to each other by edge and/or vertex sharing, building up an octamolybdate anion. Similar POMs with an Mo_8 core linked to the ends by Mo_xO_y groups are found in the crystal structures of $[NH_3(CH_2)_2NH_2(CH_2)_2NH_3]_2[Mo_9O_{30}]$ and $[NH_3(CH_2)_2NH_2(CH_2)_3NH_3]_2[Mo_{10}O_{33}]$ (Chakrabarti & Natarajan, 2002). In $(C_8H_{20}N)_8[Mo_{10}O_{34}]$, the β-octamolybdate polyanion is linked with two additional MoO_4 tetrahedra, which can be expressed by the formula $[(MoO_3)_2\beta-Mo_8O_{28}]^{8-}$. Bond-valence calculations show that the five crystallographically unique Mo atoms are in the +VI oxidation state. According to the role of the oxygen ligands (terminal or bridging) in the β-octamolybdate moiety, the corresponding Mo—O bond lengths for Mo1–Mo4 range from 1.703 (3) to 2.451 (3) Å and the O—Mo—O bond angles from 71.01 (12) to 179.57 (14)°. These values are in the range expected for octahedrally coordinated Mo^{VI} atoms and in agreement with those in the previously reported octamolybdate structure. (Pavani & Ramanan, 2005; Wu *et al.*, 2002). The Mo5 site is tetrahedrally surrounded by three terminal oxygen atoms (O15, O16, O17) with bond lengths between 1.738 (4) and 1.767 (4) Å and a bridging oxygen atom O14 to the β-octamolybdate anion with 1.804 (4) Å. The angles of the tetrahedron range from 107.5 (2) to 112.0 (2)°.

Figure 2
The centrosymmetric $[Mo_{10}O_{34}]^{8-}$ polyanion in the title compound. Displacement ellipsoids are drawn at the 50% probability level.
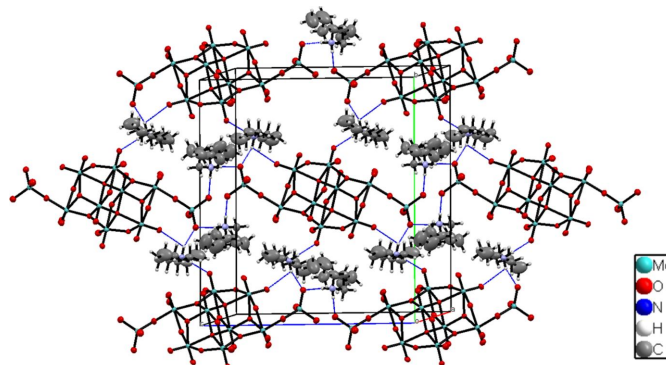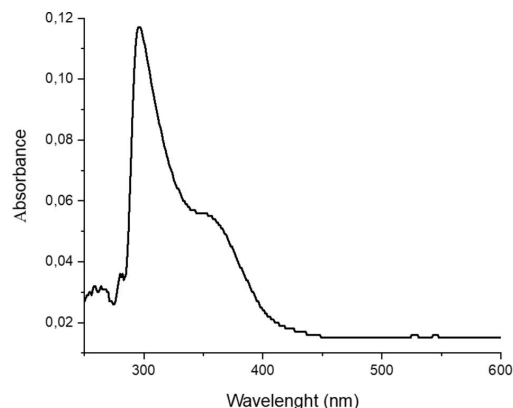

Figure 3
The unit-cell packing viewed down [001] with hydrogen bonds indicated by blue dashed lines.

Figure 4
UV/Vis spectrum of the title compound.

In the crystal, the $[Mo_{10}O_{34}]^{8-}$ polyanions are stacked into rows parallel to [001] and surrounded by dibutylammonium counter-cations. Next to Coulombic interactions, cations and anions are linked through rather strong N—H···O hydrogen bonds between the ammonium cations and the terminal oxygen atoms of the MoO_4 tetrahedra (O15···H3A—N3 and O17···H3B—N3; Table 1, Fig. 3). The other ammonium groups are involved in hydrogen-bonding interactions with the terminal O atoms of the β-octamolybdate moiety (Table 1).

The UV-vis absorption spectrum of the title compound was recorded in the range 250–700 nm in aqueous solution (0.1 N) and is shown in Fig. 4. It shows two absorption bands at 297 nm and 353 nm. The strongest band at 297 nm is attributed to a charge-transfer transition of the type O_t —Mo and the shoulder peak at 353 nm to a charge-transfer transition of the type Mo—O—Mo (Gong *et al.*, 2006; Zhang *et al.*, 1997)

Synthesis and crystallization

Ammonium heptamolybdate, $(NH_4)_6[Mo_7O_{24}]\cdot 4H_2O$ (4.943 g), and dibutylamine, $C_8H_{19}N$ (1.559 g), were dissolved in 40 ml of hot water. The mixture was heated for 2 h at 473 K under reflux and then filtered. The filtrate was kept for three months at ambient conditions, affording colourless crystals in about 8% yield (based on Mo).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

References

Allis, D. G., Burkholder, E. & Zubieta, J. A. (2004). *Polyhedron*, **23**, 1145–1152.
Chakrabarti, S. & Natarajan, S. (2002). *Cryst. Growth Des.* **2**, 333–335.
Cronin, L., Beugholt, C., Krickemeyer, E., Schmidtmann, M., Bögge, H., Kögerler, P., Luong, T. K. K. & Müller, A. (2002). *Angew. Chem. Int. Ed.* **41**, 2805–2808.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_8H_{20}N)_8[Mo_{10}O_{34}]$
M_r	2545.39
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	130
a, b, c (Å)	14.21628 (18), 20.7477 (2), 18.2210 (2)
β (°)	110.0785 (15)
V (Å³)	5047.74 (12)
Z	2
Radiation type	Cu $K\alpha$
μ (mm⁻¹)	10.44
Crystal size (mm)	$0.15 \times 0.03 \times 0.02$
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.500, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	42024, 9853, 8753
R_{int}	0.043
$(\sin \theta/\lambda)_{max}$ (Å⁻¹)	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.119, 1.02
No. of reflections	9853
No. of parameters	541
No. of restraints	18
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å⁻³)	1.65, −1.09

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

Du, J., Yu, J., Tang, J., Wang, J., Zhang, W., Thiel, W. R. & Jia, M. (2011). *Eur. J. Inorg. Chem.* pp. 2361–2365.
Fuchs, J., Hartl, H., Hunnius, W.-D. & Mahjour, S. (1975). *Angew. Chem.* **87**, 634–635.
Gong, Y., Hu, C., Li, H., Tang, W., Huang, K. & Hou, W. (2006). *J. Mol. Struct.* **784**, 228–238.
Hao, J., Ruhlmann, L., Zhu, Y., Li, Q. & Wei, Y. (2007). *Inorg. Chem.* **46**, 4960–4967.
Harchani, A. & Haddad, A. (2015). *J. Clust. Sci.* **26**, 1773–1785.
Isobe, M., Marumo, F., Yamase, T. & Ikawa, T. (1978). *Acta Cryst.* **B34**, 2728–2731.
Li, T., Lü, J., Gao, S., Li, F. & Cao, R. (2007). *Chem. Lett.* **36**, 356–357.
Müller, A., Shah, S. Q. N., Bögge, H. & Schmidtmann, M. (1999). *Nature*, **397**, 48–50.
Pavani, K., Lofland, S. E., Ramanujachary, K. V. & Ramanan, A. (2007). *Eur. J. Inorg. Chem.* pp. 568–578.
Pavani, K. & Ramanan, A. (2005). *Eur. J. Inorg. Chem.* pp. 3080–3087.
Rigaju OD (2021). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2015a). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015b). *Acta Cryst.* C**71**, 3–8.
Wu, C.-D., Lu, C.-Z., Zhuang, H.-H. & Huang, J.-S. (2002). *Inorg. Chem.* **41**, 5636–5637.
Xu, L., Qin, C., Wang, X., Wei, Y. & Wang, E. (2003). *Inorg. Chem.* **42**, 7342–7344.
Zhang, J. Y., Chang, Z. H., Wang, X. L., Wang, X. & Lin, H. Y. (2021). *New J. Chem.* **45**, 3328–3334.
Zhang, X.-M., Shan, B.-Z., You, X.-Z. & Fun, H.-K. (1997). *Polyhedron*, **16**, 95–102.

full crystallographic data

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Crystal data

$(C_8H_{20}N)_8[Mo_{10}O_{34}]$
$M_r = 2545.39$
Monoclinic, $P2_1/c$
$a = 14.21628 (18)$ Å
$b = 20.7477 (2)$ Å
$c = 18.2210 (2)$ Å
$\beta = 110.0785 (15)°$
$V = 5047.74 (12)$ Å^3
$Z = 2$

$F(000) = 2584$
$D_x = 1.675$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 25536 reflections
$\theta = 3.3–76.7°$
$\mu = 10.44$ mm^{-1}
$T = 130$ K
Needle, clear colourless
$0.15 \times 0.03 \times 0.02$ mm

Data collection

XtaLAB Synergy, Dualflex, HyPix
    diffractometer
Radiation source: micro-focus sealed X-ray
    tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan
    (CrysAlisPro; Rigaku OD, 2023)

$T_{min} = 0.500$, $T_{max} = 1.000$
42024 measured reflections
9853 independent reflections
8753 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 74.5°$, $\theta_{min} = 3.3°$
$h = -17 \rightarrow 14$
$k = -24 \rightarrow 24$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.119$
$S = 1.02$
9853 reflections
541 parameters
18 restraints
Primary atom site location: dual

Hydrogen site location: inferred from
    neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 16.9839P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.65$ e Å^{-3}
$\Delta\rho_{min} = -1.09$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.46738 (3)	0.39489 (2)	0.35188 (2)	0.02715 (10)	
Mo2	0.39496 (3)	0.38023 (2)	0.50285 (2)	0.02920 (10)	
Mo3	0.60253 (3)	0.46487 (2)	0.57062 (2)	0.02504 (10)	
Mo4	0.50325 (3)	0.45478 (2)	0.70777 (2)	0.02984 (10)	
Mo5	0.60971 (3)	0.54579 (2)	0.89668 (2)	0.03279 (11)	
O1	0.3980 (3)	0.37372 (16)	0.25734 (19)	0.0367 (8)	
O2	0.5564 (3)	0.45633 (15)	0.33548 (19)	0.0323 (7)	
O3	0.5479 (3)	0.33261 (16)	0.3883 (2)	0.0358 (7)	
O4	0.5043 (2)	0.43977 (14)	0.47119 (17)	0.0248 (6)	
O5	0.3602 (3)	0.37118 (15)	0.39495 (18)	0.0298 (7)	
O6	0.2865 (3)	0.37049 (17)	0.5231 (2)	0.0386 (8)	
O7	0.4603 (3)	0.30910 (16)	0.53384 (19)	0.0343 (7)	
O8	0.4764 (2)	0.43107 (15)	0.59639 (18)	0.0284 (6)	
O9	0.6765 (3)	0.39936 (16)	0.60011 (19)	0.0345 (7)	
O10	0.6746 (2)	0.51676 (16)	0.53399 (19)	0.0305 (7)	
O11	0.6152 (2)	0.50842 (14)	0.66534 (18)	0.0284 (6)	
O12	0.3977 (3)	0.41825 (18)	0.7121 (2)	0.0425 (9)	
O13	0.5989 (3)	0.39987 (17)	0.7488 (2)	0.0419 (8)	
O14	0.5422 (4)	0.50320 (18)	0.8079 (2)	0.0483 (10)	
O15	0.5518 (3)	0.61967 (17)	0.9041 (2)	0.0406 (8)	
O16	0.7336 (3)	0.5584 (2)	0.9045 (2)	0.0486 (9)	
O17	0.6081 (3)	0.49797 (18)	0.9747 (2)	0.0473 (9)	
N1	1.2542 (4)	0.5261 (3)	0.5803 (3)	0.0528 (13)	
H1A	1.317838	0.533197	0.613310	0.063*	
H1B	1.257981	0.498154	0.542852	0.063*	
C1	1.0075 (8)	0.6326 (6)	0.3483 (6)	0.103 (4)	
H1C	1.003411	0.670808	0.315617	0.155*	
H1D	0.944901	0.627889	0.358998	0.155*	
H1E	1.018427	0.594290	0.320836	0.155*	
C2	1.0942 (7)	0.6403 (5)	0.4248 (5)	0.087 (3)	
H2A	1.152069	0.659291	0.414106	0.104*	
H2B	1.074403	0.670627	0.458930	0.104*	
C3	1.1249 (5)	0.5784 (4)	0.4665 (4)	0.068 (2)	
H3C	1.143849	0.547696	0.432336	0.081*	
H3D	1.067847	0.559794	0.478613	0.081*	
C4	1.2132 (5)	0.5878 (4)	0.5420 (4)	0.0581 (16)	
H4C	1.266756	0.611691	0.530394	0.070*	
H4D	1.191445	0.614235	0.578467	0.070*	
C5	1.1975 (5)	0.4946 (4)	0.6249 (4)	0.0613 (17)	
H5A	1.217167	0.448646	0.633571	0.074*	
H5B	1.124920	0.496506	0.594592	0.074*	
C6	1.2188 (7)	0.5285 (5)	0.7041 (5)	0.078 (2)	
H6A	1.292066	0.533544	0.729856	0.093*	
H6B	1.188595	0.572041	0.695217	0.093*	
C7	1.1781 (8)	0.4919 (6)	0.7566 (6)	0.097 (3)	

H7A	1.204890	0.447427	0.763302	0.116*
H7B	1.104202	0.489454	0.733008	0.116*
C8	1.2075 (11)	0.5256 (7)	0.8371 (6)	0.132 (5)
H8A	1.164148	0.563035	0.833396	0.197*
H8B	1.277455	0.539697	0.853090	0.197*
H8C	1.199623	0.495291	0.875880	0.197*
N2	0.5610 (4)	0.7400 (2)	0.8400 (3)	0.0435 (11)
H2C	0.558726	0.697106	0.849875	0.052*
H2D	0.551162	0.761770	0.880137	0.052*
C9	0.9281 (8)	0.7162 (6)	0.9733 (10)	0.136 (5)
H9A	0.925536	0.730830	1.023715	0.204*
H9B	0.993739	0.726632	0.969919	0.204*
H9C	0.917704	0.669441	0.968751	0.204*
C10	0.8498 (7)	0.7484 (4)	0.9101 (6)	0.083 (3)
H10A	0.856978	0.736603	0.859627	0.099*
H10B	0.858736	0.795591	0.916640	0.099*
C11	0.7436 (5)	0.7304 (3)	0.9077 (4)	0.0569 (16)
H11A	0.737917	0.682872	0.908117	0.068*
H11B	0.733897	0.747021	0.955539	0.068*
C12	0.6631 (5)	0.7559 (3)	0.8388 (4)	0.0496 (14)
H12A	0.670511	0.737780	0.790857	0.059*
H12B	0.669932	0.803313	0.837023	0.059*
C13	0.4791 (5)	0.7553 (3)	0.7679 (3)	0.0521 (15)
H13A	0.487501	0.730037	0.724489	0.063*
H13B	0.482772	0.801585	0.755759	0.063*
C14	0.3794 (6)	0.7416 (4)	0.7727 (5)	0.071 (2)
H14A	0.369298	0.768421	0.814244	0.085*
H14B	0.376330	0.695772	0.786885	0.085*
C15	0.2959 (6)	0.7553 (4)	0.6953 (6)	0.085 (3)
H15A	0.301443	0.800569	0.680112	0.102*
H15B	0.305073	0.727183	0.654378	0.102*
C16	0.1939 (9)	0.7446 (7)	0.6986 (9)	0.138 (5)
H16A	0.187367	0.767691	0.743506	0.207*
H16B	0.183356	0.698427	0.703895	0.207*
H16C	0.143702	0.760723	0.650403	0.207*
N3	0.5165 (4)	0.3786 (2)	0.9706 (3)	0.0484 (12)
H3A	0.498144	0.376434	1.013761	0.058*
H3B	0.548926	0.416775	0.972730	0.058*
C18	0.2616 (9)	0.3213 (6)	0.8363 (6)	0.105 (3)
H18A	0.261341	0.352706	0.795295	0.126*	0.333 (15)
H18B	0.244014	0.278560	0.811063	0.126*	0.333 (15)
H18C	0.227094	0.359599	0.847226	0.126*	0.667 (15)
H18D	0.267273	0.327310	0.784033	0.126*	0.667 (15)
C19	0.3625 (8)	0.3181 (4)	0.8945 (6)	0.091 (3)
H19A	0.398549	0.281599	0.881310	0.110*
H19B	0.356831	0.309039	0.946131	0.110*
C20	0.4248 (7)	0.3793 (4)	0.9009 (4)	0.0668 (19)
H20A	0.443553	0.383491	0.853603	0.080*

H20B	0.383793	0.417232	0.903352	0.080*	
C21	0.5880 (6)	0.3257 (4)	0.9748 (6)	0.080 (2)	
H21A	0.558598	0.284238	0.983043	0.096*	
H21B	0.600695	0.322988	0.924752	0.096*	
C22	0.6830 (8)	0.3368 (6)	1.0391 (7)	0.102 (3)	
H22A	0.668767	0.339739	1.088525	0.123*	
H22B	0.710307	0.378925	1.030655	0.123*	
C23	0.7605 (11)	0.2871 (8)	1.0484 (10)	0.156 (6)	
H23A	0.775827	0.284082	0.999411	0.187*	
H23B	0.734182	0.244800	1.057416	0.187*	
C24	0.8592 (12)	0.3022 (9)	1.1178 (9)	0.163 (7)	
H24A	0.891581	0.340187	1.105114	0.245*	
H24B	0.904591	0.265168	1.126657	0.245*	
H24C	0.843257	0.310613	1.165148	0.245*	
C17X	0.189 (2)	0.3390 (16)	0.868 (2)	0.106 (5)	0.333 (15)
H17A	0.124953	0.344533	0.825605	0.159*	0.333 (15)
H17B	0.208992	0.379747	0.896216	0.159*	0.333 (15)
H17C	0.183507	0.305427	0.903659	0.159*	0.333 (15)
C17	0.2031 (13)	0.2674 (8)	0.8340 (12)	0.106 (5)	0.667 (15)
H17D	0.234106	0.229559	0.819533	0.159*	0.667 (15)
H17E	0.136177	0.273997	0.795289	0.159*	0.667 (15)
H17F	0.197592	0.260561	0.885594	0.159*	0.667 (15)
N4	0.7926 (4)	0.5318 (3)	0.7782 (3)	0.0639 (17)	
H4A	0.733073	0.530722	0.737974	0.077*	
H4B	0.779178	0.542145	0.822093	0.077*	
C25	0.9612 (7)	0.6165 (5)	0.5961 (6)	0.088 (3)	
H25A	0.897792	0.615423	0.552036	0.132*	
H25B	0.997253	0.575933	0.598168	0.132*	
H25C	1.002122	0.652488	0.589347	0.132*	
C26	0.9400 (8)	0.6254 (5)	0.6717 (7)	0.097 (3)	
H26A	1.004535	0.628966	0.715395	0.116*	
H26B	0.903308	0.666367	0.668837	0.116*	
C27	0.8807 (5)	0.5722 (4)	0.6890 (4)	0.0587 (17)	
H27A	0.919594	0.531675	0.695964	0.070*	
H27B	0.818304	0.566349	0.643671	0.070*	
C28	0.8530 (6)	0.5842 (5)	0.7627 (5)	0.076 (2)	
H28A	0.915332	0.588959	0.808361	0.091*	
H28B	0.815198	0.625050	0.756318	0.091*	
C29	0.8358 (6)	0.4650 (5)	0.7886 (5)	0.084 (3)	
H29C	0.851243	0.451059	0.742069	0.100*	0.354 (12)
H29D	0.789472	0.433582	0.798999	0.100*	0.354 (12)
H29A	0.787341	0.437231	0.801932	0.100*	0.646 (12)
H29B	0.834884	0.450959	0.736475	0.100*	0.646 (12)
C30	0.9371 (9)	0.4479 (14)	0.8450 (7)	0.116 (9)	0.646 (12)
H30A	0.951345	0.402023	0.838008	0.140*	0.646 (12)
H30B	0.988524	0.474333	0.833678	0.140*	0.646 (12)
C31	0.9435 (14)	0.4588 (10)	0.9272 (9)	0.118 (4)	0.646 (12)
H31A	0.928863	0.504727	0.933306	0.142*	0.646 (12)

H31B	1.013140	0.450351	0.961847	0.142*	0.646 (12)
C32	0.8742 (14)	0.4181 (10)	0.9538 (11)	0.118 (4)	0.646 (12)
H32A	0.811153	0.411778	0.910258	0.178*	0.646 (12)
H32B	0.860589	0.439740	0.996901	0.178*	0.646 (12)
H32C	0.905380	0.376152	0.971561	0.178*	0.646 (12)
C30X	0.930 (2)	0.4727 (16)	0.8584 (18)	0.116 (9)	0.354 (12)
H30C	0.914077	0.496308	0.900096	0.140*	0.354 (12)
H30D	0.979275	0.498075	0.843703	0.140*	0.354 (12)
C31X	0.973 (2)	0.4078 (16)	0.888 (2)	0.118 (4)	0.354 (12)
H31C	1.040851	0.413603	0.926996	0.142*	0.354 (12)
H31D	0.978545	0.382011	0.844473	0.142*	0.354 (12)
C32X	0.910 (2)	0.3715 (18)	0.926 (2)	0.118 (4)	0.354 (12)
H32D	0.846812	0.358446	0.886039	0.178*	0.354 (12)
H32E	0.895563	0.399259	0.964503	0.178*	0.354 (12)
H32F	0.946241	0.333117	0.952337	0.178*	0.354 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0428 (2)	0.01922 (17)	0.02010 (17)	0.00043 (13)	0.01172 (15)	−0.00267 (12)
Mo2	0.0424 (2)	0.02309 (19)	0.02335 (18)	−0.00671 (14)	0.01292 (15)	−0.00140 (13)
Mo3	0.03428 (19)	0.01990 (17)	0.02015 (17)	0.00191 (13)	0.00831 (14)	−0.00127 (12)
Mo4	0.0462 (2)	0.02416 (19)	0.01977 (18)	−0.00317 (14)	0.01209 (15)	−0.00028 (13)
Mo5	0.0532 (2)	0.02420 (19)	0.02242 (19)	−0.00053 (15)	0.01481 (17)	−0.00126 (13)
O1	0.056 (2)	0.0296 (17)	0.0240 (16)	−0.0013 (15)	0.0126 (15)	−0.0059 (13)
O2	0.0432 (19)	0.0299 (17)	0.0275 (16)	0.0011 (13)	0.0169 (14)	−0.0018 (13)
O3	0.052 (2)	0.0268 (17)	0.0303 (17)	0.0063 (14)	0.0166 (15)	−0.0031 (13)
O4	0.0383 (16)	0.0193 (14)	0.0198 (14)	−0.0021 (12)	0.0137 (12)	0.0006 (11)
O5	0.0451 (18)	0.0216 (15)	0.0237 (15)	−0.0047 (13)	0.0130 (14)	−0.0026 (12)
O6	0.047 (2)	0.0343 (19)	0.0380 (19)	−0.0090 (15)	0.0195 (16)	−0.0030 (15)
O7	0.052 (2)	0.0274 (17)	0.0259 (16)	−0.0035 (14)	0.0164 (15)	0.0004 (13)
O8	0.0424 (18)	0.0222 (15)	0.0220 (15)	−0.0007 (13)	0.0130 (13)	0.0001 (12)
O9	0.0438 (19)	0.0296 (17)	0.0290 (17)	0.0063 (14)	0.0110 (14)	−0.0011 (13)
O10	0.0340 (17)	0.0302 (17)	0.0277 (16)	0.0014 (13)	0.0111 (13)	−0.0039 (13)
O11	0.0402 (17)	0.0216 (15)	0.0224 (15)	−0.0015 (12)	0.0097 (13)	−0.0010 (12)
O12	0.065 (2)	0.037 (2)	0.0317 (18)	−0.0111 (17)	0.0241 (17)	−0.0015 (15)
O13	0.061 (2)	0.0318 (18)	0.0295 (18)	−0.0034 (16)	0.0103 (16)	0.0049 (14)
O14	0.086 (3)	0.0331 (19)	0.0272 (18)	−0.0084 (19)	0.0214 (19)	−0.0041 (15)
O15	0.062 (2)	0.0308 (18)	0.0322 (18)	0.0013 (16)	0.0202 (17)	−0.0009 (14)
O16	0.057 (2)	0.044 (2)	0.045 (2)	0.0032 (18)	0.0185 (19)	−0.0007 (18)
O17	0.076 (3)	0.0330 (19)	0.0311 (19)	−0.0027 (18)	0.0162 (18)	0.0021 (15)
N1	0.042 (3)	0.078 (4)	0.041 (3)	0.005 (2)	0.017 (2)	−0.016 (3)
C1	0.089 (7)	0.137 (9)	0.067 (6)	0.042 (6)	0.005 (5)	−0.010 (6)
C2	0.082 (6)	0.102 (7)	0.070 (5)	0.033 (5)	0.018 (4)	−0.002 (5)
C3	0.054 (4)	0.088 (6)	0.060 (4)	0.018 (4)	0.017 (3)	−0.008 (4)
C4	0.046 (3)	0.082 (5)	0.048 (3)	0.009 (3)	0.018 (3)	−0.009 (3)
C5	0.052 (4)	0.080 (5)	0.054 (4)	0.003 (3)	0.021 (3)	−0.007 (3)
C6	0.091 (6)	0.087 (6)	0.068 (5)	0.018 (5)	0.043 (5)	−0.006 (4)

C7	0.088 (6)	0.138 (9)	0.069 (6)	0.022 (6)	0.034 (5)	0.010 (6)
C8	0.148 (11)	0.186 (13)	0.058 (6)	0.072 (10)	0.031 (6)	−0.005 (7)
N2	0.075 (3)	0.024 (2)	0.035 (2)	0.003 (2)	0.022 (2)	−0.0035 (17)
C9	0.067 (6)	0.115 (10)	0.221 (17)	−0.008 (6)	0.044 (8)	0.018 (10)
C10	0.080 (5)	0.057 (5)	0.125 (8)	−0.010 (4)	0.051 (6)	0.006 (5)
C11	0.071 (4)	0.034 (3)	0.075 (5)	−0.003 (3)	0.037 (4)	0.000 (3)
C12	0.082 (4)	0.028 (3)	0.051 (3)	−0.007 (3)	0.039 (3)	−0.008 (2)
C13	0.082 (4)	0.033 (3)	0.039 (3)	0.007 (3)	0.018 (3)	−0.007 (2)
C14	0.089 (5)	0.044 (4)	0.064 (5)	−0.003 (3)	0.005 (4)	−0.002 (3)
C15	0.070 (5)	0.064 (5)	0.095 (7)	0.000 (4)	−0.005 (4)	−0.012 (4)
C16	0.086 (8)	0.120 (10)	0.179 (15)	−0.008 (7)	0.010 (8)	0.010 (10)
N3	0.076 (3)	0.040 (3)	0.042 (3)	−0.004 (2)	0.035 (3)	−0.001 (2)
C18	0.103 (8)	0.138 (10)	0.080 (7)	0.005 (7)	0.038 (6)	0.004 (6)
C19	0.108 (7)	0.066 (5)	0.074 (6)	−0.014 (5)	−0.002 (5)	0.000 (4)
C20	0.103 (6)	0.054 (4)	0.042 (4)	0.008 (4)	0.023 (4)	0.000 (3)
C21	0.071 (5)	0.064 (5)	0.109 (7)	0.000 (4)	0.035 (5)	−0.018 (5)
C22	0.090 (7)	0.112 (8)	0.100 (8)	0.013 (6)	0.026 (6)	0.019 (6)
C23	0.120 (10)	0.151 (13)	0.176 (15)	0.045 (9)	0.025 (10)	−0.044 (11)
C24	0.143 (13)	0.201 (18)	0.128 (12)	0.051 (12)	0.023 (10)	−0.022 (12)
C17X	0.094 (10)	0.100 (10)	0.135 (14)	−0.002 (8)	0.053 (9)	0.015 (9)
C17	0.094 (10)	0.100 (10)	0.135 (14)	−0.002 (8)	0.053 (9)	0.015 (9)
N4	0.040 (3)	0.114 (5)	0.038 (3)	−0.007 (3)	0.013 (2)	−0.019 (3)
C25	0.063 (5)	0.129 (8)	0.076 (6)	−0.003 (5)	0.029 (4)	0.022 (5)
C26	0.090 (6)	0.107 (8)	0.106 (8)	−0.035 (6)	0.051 (6)	−0.028 (6)
C27	0.039 (3)	0.085 (5)	0.047 (3)	0.001 (3)	0.009 (3)	−0.007 (3)
C28	0.055 (4)	0.116 (7)	0.057 (4)	−0.021 (4)	0.019 (3)	−0.034 (4)
C29	0.058 (4)	0.131 (8)	0.065 (5)	0.012 (5)	0.025 (4)	0.019 (5)
C30	0.058 (6)	0.20 (2)	0.088 (10)	0.030 (9)	0.021 (6)	0.058 (12)
C31	0.110 (9)	0.138 (12)	0.098 (9)	0.018 (7)	0.023 (6)	0.028 (8)
C32	0.110 (9)	0.138 (12)	0.098 (9)	0.018 (7)	0.023 (6)	0.028 (8)
C30X	0.058 (6)	0.20 (2)	0.088 (10)	0.030 (9)	0.021 (6)	0.058 (12)
C31X	0.110 (9)	0.138 (12)	0.098 (9)	0.018 (7)	0.023 (6)	0.028 (8)
C32X	0.110 (9)	0.138 (12)	0.098 (9)	0.018 (7)	0.023 (6)	0.028 (8)

Geometric parameters (Å, °)

Mo1—O1	1.723 (3)	C15—H15B	0.9900
Mo1—O2	1.891 (3)	C15—C16	1.489 (16)
Mo1—O3	1.703 (3)	C16—H16A	0.9800
Mo1—O4	2.255 (3)	C16—H16B	0.9800
Mo1—O5	2.001 (3)	C16—H16C	0.9800
Mo1—O11[i]	2.291 (3)	N3—H3A	0.9100
Mo2—O4	2.211 (3)	N3—H3B	0.9100
Mo2—O5	1.865 (3)	N3—C20	1.475 (10)
Mo2—O6	1.717 (4)	N3—C21	1.481 (9)
Mo2—O7	1.732 (3)	C18—H18A	0.9900
Mo2—O8	2.001 (3)	C18—H18B	0.9900
Mo2—O10[i]	2.354 (3)	C18—H18C	0.9900

Mo3—O4	1.941 (3)	C18—H18D	0.9900
Mo3—O4^i	2.451 (3)	C18—C19	1.463 (14)
Mo3—O8	2.122 (3)	C18—C17X	1.386 (18)
Mo3—O9	1.690 (3)	C18—C17	1.386 (16)
Mo3—O10	1.767 (3)	C19—H19A	0.9900
Mo3—O11	1.901 (3)	C19—H19B	0.9900
Mo4—O2^i	2.070 (3)	C19—C20	1.529 (12)
Mo4—O8	1.994 (3)	C20—H20A	0.9900
Mo4—O11	2.284 (3)	C20—H20B	0.9900
Mo4—O12	1.706 (4)	C21—H21A	0.9900
Mo4—O13	1.734 (4)	C21—H21B	0.9900
Mo4—O14	1.987 (4)	C21—C22	1.471 (14)
Mo5—O14	1.804 (4)	C22—H22A	0.9900
Mo5—O15	1.767 (4)	C22—H22B	0.9900
Mo5—O16	1.738 (4)	C22—C23	1.476 (16)
Mo5—O17	1.741 (4)	C23—H23A	0.9900
N1—H1A	0.9100	C23—H23B	0.9900
N1—H1B	0.9100	C23—C24	1.56 (2)
N1—C4	1.479 (9)	C24—H24A	0.9800
N1—C5	1.480 (9)	C24—H24B	0.9800
C1—H1C	0.9800	C24—H24C	0.9800
C1—H1D	0.9800	C17X—H17A	0.9800
C1—H1E	0.9800	C17X—H17B	0.9800
C1—C2	1.520 (12)	C17X—H17C	0.9800
C2—H2A	0.9900	C17—H17D	0.9800
C2—H2B	0.9900	C17—H17E	0.9800
C2—C3	1.480 (13)	C17—H17F	0.9800
C3—H3C	0.9900	N4—H4A	0.9100
C3—H3D	0.9900	N4—H4B	0.9100
C3—C4	1.523 (10)	N4—C28	1.471 (11)
C4—H4C	0.9900	N4—C29	1.500 (12)
C4—H4D	0.9900	C25—H25A	0.9800
C5—H5A	0.9900	C25—H25B	0.9800
C5—H5B	0.9900	C25—H25C	0.9800
C5—C6	1.540 (10)	C25—C26	1.518 (13)
C6—H6A	0.9900	C26—H26A	0.9900
C6—H6B	0.9900	C26—H26B	0.9900
C6—C7	1.485 (13)	C26—C27	1.488 (12)
C7—H7A	0.9900	C27—H27A	0.9900
C7—H7B	0.9900	C27—H27B	0.9900
C7—C8	1.547 (14)	C27—C28	1.543 (10)
C8—H8A	0.9800	C28—H28A	0.9900
C8—H8B	0.9800	C28—H28B	0.9900
C8—H8C	0.9800	C29—H29C	0.9900
N2—H2C	0.9100	C29—H29D	0.9900
N2—H2D	0.9100	C29—H29A	0.9900
N2—C12	1.496 (8)	C29—H29B	0.9900
N2—C13	1.461 (8)	C29—C30	1.497 (11)

C9—H9A	0.9800	C29—C30X	1.502 (12)
C9—H9B	0.9800	C30—H30A	0.9900
C9—H9C	0.9800	C30—H30B	0.9900
C9—C10	1.460 (16)	C30—C31	1.486 (12)
C10—H10A	0.9900	C31—H31A	0.9900
C10—H10B	0.9900	C31—H31B	0.9900
C10—C11	1.541 (10)	C31—C32	1.500 (12)
C11—H11A	0.9900	C32—H32A	0.9800
C11—H11B	0.9900	C32—H32B	0.9800
C11—C12	1.476 (10)	C32—H32C	0.9800
C12—H12A	0.9900	C30X—H30C	0.9900
C12—H12B	0.9900	C30X—H30D	0.9900
C13—H13A	0.9900	C30X—C31X	1.502 (13)
C13—H13B	0.9900	C31X—H31C	0.9900
C13—C14	1.477 (11)	C31X—H31D	0.9900
C14—H14A	0.9900	C31X—C32X	1.502 (12)
C14—H14B	0.9900	C32X—H32D	0.9800
C14—C15	1.526 (11)	C32X—H32E	0.9800
C15—H15A	0.9900	C32X—H32F	0.9800
O1—Mo1—O2	101.45 (16)	N2—C13—H13B	109.0
O1—Mo1—O4	158.94 (15)	N2—C13—C14	112.9 (6)
O1—Mo1—O5	94.45 (16)	H13A—C13—H13B	107.8
O1—Mo1—O11^{i}	89.75 (14)	C14—C13—H13A	109.0
O2—Mo1—O4	85.83 (12)	C14—C13—H13B	109.0
O2—Mo1—O5	150.37 (13)	C13—C14—H14A	109.3
O2—Mo1—O11^{i}	74.19 (13)	C13—C14—H14B	109.3
O3—Mo1—O1	105.88 (17)	C13—C14—C15	111.6 (7)
O3—Mo1—O2	100.13 (16)	H14A—C14—H14B	108.0
O3—Mo1—O4	92.02 (14)	C15—C14—H14A	109.3
O3—Mo1—O5	99.31 (15)	C15—C14—H14B	109.3
O3—Mo1—O11^{i}	164.24 (14)	C14—C15—H15A	108.9
O4—Mo1—O11^{i}	73.09 (11)	C14—C15—H15B	108.9
O5—Mo1—O4	71.33 (12)	H15A—C15—H15B	107.7
O5—Mo1—O11^{i}	81.10 (12)	C16—C15—C14	113.3 (10)
O4—Mo2—O10^{i}	71.49 (11)	C16—C15—H15A	108.9
O5—Mo2—O4	74.75 (12)	C16—C15—H15B	108.9
O5—Mo2—O8	144.85 (13)	C15—C16—H16A	109.5
O5—Mo2—O10^{i}	81.80 (12)	C15—C16—H16B	109.5
O6—Mo2—O4	152.33 (15)	C15—C16—H16C	109.5
O6—Mo2—O5	106.39 (17)	H16A—C16—H16B	109.5
O6—Mo2—O7	104.51 (17)	H16A—C16—H16C	109.5
O6—Mo2—O8	100.31 (15)	H16B—C16—H16C	109.5
O6—Mo2—O10^{i}	81.24 (15)	H3A—N3—H3B	107.4
O7—Mo2—O4	102.40 (14)	C20—N3—H3A	108.3
O7—Mo2—O5	100.16 (15)	C20—N3—H3B	108.3
O7—Mo2—O8	94.73 (15)	C20—N3—C21	116.0 (6)
O7—Mo2—O10^{i}	172.96 (14)	C21—N3—H3A	108.3

O8—Mo2—O4	71.01 (12)	C21—N3—H3B	108.3
O8—Mo2—O10ⁱ	80.09 (12)	H18A—C18—H18B	107.8
O4—Mo3—O4ⁱ	75.88 (12)	H18C—C18—H18D	107.6
O4—Mo3—O8	74.10 (12)	C19—C18—H18A	109.0
O8—Mo3—O4ⁱ	81.68 (11)	C19—C18—H18B	109.0
O9—Mo3—O4ⁱ	179.57 (15)	C19—C18—H18C	108.6
O9—Mo3—O4	104.54 (15)	C19—C18—H18D	108.6
O9—Mo3—O8	98.32 (15)	C17X—C18—H18A	109.0
O9—Mo3—O10	103.39 (16)	C17X—C18—H18B	109.0
O9—Mo3—O11	103.82 (15)	C17X—C18—C19	113.1 (19)
O10—Mo3—O4	97.80 (14)	C17—C18—H18C	108.6
O10—Mo3—O4ⁱ	76.62 (12)	C17—C18—H18D	108.6
O10—Mo3—O8	158.14 (14)	C17—C18—C19	114.5 (12)
O10—Mo3—O11	99.60 (14)	C18—C19—H19A	108.6
O11—Mo3—O4ⁱ	75.77 (12)	C18—C19—H19B	108.6
O11—Mo3—O4	142.06 (14)	C18—C19—C20	114.7 (9)
O11—Mo3—O8	77.31 (13)	H19A—C19—H19B	107.6
O2ⁱ—Mo4—O11	71.28 (12)	C20—C19—H19A	108.6
O8—Mo4—O2ⁱ	85.53 (13)	C20—C19—H19B	108.6
O8—Mo4—O11	71.79 (12)	N3—C20—C19	112.1 (6)
O12—Mo4—O2ⁱ	99.29 (17)	N3—C20—H20A	109.2
O12—Mo4—O8	94.19 (15)	N3—C20—H20B	109.2
O12—Mo4—O11	163.32 (15)	C19—C20—H20A	109.2
O12—Mo4—O13	105.07 (19)	C19—C20—H20B	109.2
O12—Mo4—O14	98.93 (17)	H20A—C20—H20B	107.9
O13—Mo4—O2ⁱ	155.20 (16)	N3—C21—H21A	109.5
O13—Mo4—O8	97.15 (15)	N3—C21—H21B	109.5
O13—Mo4—O11	86.08 (15)	H21A—C21—H21B	108.1
O13—Mo4—O14	90.34 (18)	C22—C21—N3	110.5 (8)
O14—Mo4—O2ⁱ	81.24 (15)	C22—C21—H21A	109.5
O14—Mo4—O8	162.67 (14)	C22—C21—H21B	109.5
O14—Mo4—O11	93.27 (14)	C21—C22—H22A	108.5
O15—Mo5—O14	112.07 (19)	C21—C22—H22B	108.5
O16—Mo5—O14	111.0 (2)	C21—C22—C23	115.3 (11)
O16—Mo5—O15	110.43 (19)	H22A—C22—H22B	107.5
O16—Mo5—O17	108.5 (2)	C23—C22—H22A	108.5
O17—Mo5—O14	107.55 (18)	C23—C22—H22B	108.5
O17—Mo5—O15	107.13 (18)	C22—C23—H23A	109.1
Mo1—O2—Mo4ⁱ	116.86 (17)	C22—C23—H23B	109.1
Mo1—O4—Mo3ⁱ	96.86 (11)	C22—C23—C24	112.3 (12)
Mo2—O4—Mo1	93.97 (11)	H23A—C23—H23B	107.9
Mo2—O4—Mo3ⁱ	96.67 (11)	C24—C23—H23A	109.1
Mo3—O4—Mo1	149.92 (16)	C24—C23—H23B	109.1
Mo3—O4—Mo2	104.56 (12)	C23—C24—H24A	109.5
Mo3—O4—Mo3ⁱ	104.12 (12)	C23—C24—H24B	109.5
Mo2—O5—Mo1	115.25 (16)	C23—C24—H24C	109.5
Mo2—O8—Mo3	105.76 (13)	H24A—C24—H24B	109.5
Mo4—O8—Mo2	148.17 (17)	H24A—C24—H24C	109.5

Mo4—O8—Mo3	105.24 (14)	H24B—C24—H24C	109.5
Mo3—O10—Mo2i	114.80 (15)	C18—C17X—H17A	109.5
Mo3—O11—Mo1i	114.04 (14)	C18—C17X—H17B	109.5
Mo3—O11—Mo4	102.46 (13)	C18—C17X—H17C	109.5
Mo4—O11—Mo1i	95.11 (12)	H17A—C17X—H17B	109.5
Mo5—O14—Mo4	165.0 (3)	H17A—C17X—H17C	109.5
H1A—N1—H1B	107.4	H17B—C17X—H17C	109.5
C4—N1—H1A	108.2	C18—C17—H17D	109.5
C4—N1—H1B	108.2	C18—C17—H17E	109.5
C4—N1—C5	116.2 (5)	C18—C17—H17F	109.5
C5—N1—H1A	108.2	H17D—C17—H17E	109.5
C5—N1—H1B	108.2	H17D—C17—H17F	109.5
H1C—C1—H1D	109.5	H17E—C17—H17F	109.5
H1C—C1—H1E	109.5	H4A—N4—H4B	107.2
H1D—C1—H1E	109.5	C28—N4—H4A	107.9
C2—C1—H1C	109.5	C28—N4—H4B	107.9
C2—C1—H1D	109.5	C28—N4—C29	117.8 (6)
C2—C1—H1E	109.5	C29—N4—H4A	107.9
C1—C2—H2A	109.1	C29—N4—H4B	107.9
C1—C2—H2B	109.1	H25A—C25—H25B	109.5
H2A—C2—H2B	107.8	H25A—C25—H25C	109.5
C3—C2—C1	112.5 (9)	H25B—C25—H25C	109.5
C3—C2—H2A	109.1	C26—C25—H25A	109.5
C3—C2—H2B	109.1	C26—C25—H25B	109.5
C2—C3—H3C	109.4	C26—C25—H25C	109.5
C2—C3—H3D	109.4	C25—C26—H26A	108.8
C2—C3—C4	111.0 (7)	C25—C26—H26B	108.8
H3C—C3—H3D	108.0	H26A—C26—H26B	107.7
C4—C3—H3C	109.4	C27—C26—C25	114.0 (8)
C4—C3—H3D	109.4	C27—C26—H26A	108.8
N1—C4—C3	112.6 (6)	C27—C26—H26B	108.8
N1—C4—H4C	109.1	C26—C27—H27A	108.9
N1—C4—H4D	109.1	C26—C27—H27B	108.9
C3—C4—H4C	109.1	C26—C27—C28	113.4 (7)
C3—C4—H4D	109.1	H27A—C27—H27B	107.7
H4C—C4—H4D	107.8	C28—C27—H27A	108.9
N1—C5—H5A	109.6	C28—C27—H27B	108.9
N1—C5—H5B	109.6	N4—C28—C27	112.8 (6)
N1—C5—C6	110.1 (6)	N4—C28—H28A	109.0
H5A—C5—H5B	108.1	N4—C28—H28B	109.0
C6—C5—H5A	109.6	C27—C28—H28A	109.0
C6—C5—H5B	109.6	C27—C28—H28B	109.0
C5—C6—H6A	109.2	H28A—C28—H28B	107.8
C5—C6—H6B	109.2	N4—C29—H29C	111.3
H6A—C6—H6B	107.9	N4—C29—H29D	111.3
C7—C6—C5	112.1 (8)	N4—C29—H29A	106.3
C7—C6—H6A	109.2	N4—C29—H29B	106.3
C7—C6—H6B	109.2	N4—C29—C30X	102.4 (16)

C6—C7—H7A	109.7	H29C—C29—H29D	109.2
C6—C7—H7B	109.7	H29A—C29—H29B	106.4
C6—C7—C8	110.0 (11)	C30—C29—N4	124.2 (13)
H7A—C7—H7B	108.2	C30—C29—H29A	106.3
C8—C7—H7A	109.7	C30—C29—H29B	106.3
C8—C7—H7B	109.7	C30X—C29—H29C	111.3
C7—C8—H8A	109.5	C30X—C29—H29D	111.3
C7—C8—H8B	109.5	C29—C30—H30A	109.3
C7—C8—H8C	109.5	C29—C30—H30B	109.3
H8A—C8—H8B	109.5	H30A—C30—H30B	108.0
H8A—C8—H8C	109.5	C31—C30—C29	111.5 (13)
H8B—C8—H8C	109.5	C31—C30—H30A	109.3
H2C—N2—H2D	107.6	C31—C30—H30B	109.3
C12—N2—H2C	108.6	C30—C31—H31A	108.5
C12—N2—H2D	108.6	C30—C31—H31B	108.5
C13—N2—H2C	108.6	C30—C31—C32	114.9 (13)
C13—N2—H2D	108.6	H31A—C31—H31B	107.5
C13—N2—C12	114.6 (5)	C32—C31—H31A	108.5
H9A—C9—H9B	109.5	C32—C31—H31B	108.5
H9A—C9—H9C	109.5	C31—C32—H32A	109.5
H9B—C9—H9C	109.5	C31—C32—H32B	109.5
C10—C9—H9A	109.5	C31—C32—H32C	109.5
C10—C9—H9B	109.5	H32A—C32—H32B	109.5
C10—C9—H9C	109.5	H32A—C32—H32C	109.5
C9—C10—H10A	109.0	H32B—C32—H32C	109.5
C9—C10—H10B	109.0	C29—C30X—H30C	109.6
C9—C10—C11	112.7 (8)	C29—C30X—H30D	109.6
H10A—C10—H10B	107.8	C29—C30X—C31X	110 (2)
C11—C10—H10A	109.0	H30C—C30X—H30D	108.1
C11—C10—H10B	109.0	C31X—C30X—H30C	109.6
C10—C11—H11A	108.8	C31X—C30X—H30D	109.6
C10—C11—H11B	108.8	C30X—C31X—H31C	109.1
H11A—C11—H11B	107.7	C30X—C31X—H31D	109.1
C12—C11—C10	113.8 (6)	C30X—C31X—C32X	112.5 (14)
C12—C11—H11A	108.8	H31C—C31X—H31D	107.8
C12—C11—H11B	108.8	C32X—C31X—H31C	109.1
N2—C12—H12A	109.1	C32X—C31X—H31D	109.1
N2—C12—H12B	109.1	C31X—C32X—H32D	109.5
C11—C12—N2	112.4 (5)	C31X—C32X—H32E	109.5
C11—C12—H12A	109.1	C31X—C32X—H32F	109.5
C11—C12—H12B	109.1	H32D—C32X—H32E	109.5
H12A—C12—H12B	107.9	H32D—C32X—H32F	109.5
N2—C13—H13A	109.0	H32E—C32X—H32F	109.5
O1—Mo1—O2—Mo4ⁱ	72.5 (2)	C5—C6—C7—C8	176.2 (8)
O3—Mo1—O2—Mo4ⁱ	−178.82 (17)	N2—C13—C14—C15	−177.5 (6)
O4—Mo1—O2—Mo4ⁱ	−87.51 (17)	C9—C10—C11—C12	−172.9 (9)
O4—Mo2—O5—Mo1	18.83 (15)	C10—C11—C12—N2	−177.6 (6)

O4—Mo3—O10—Mo2[i]	78.67 (16)	C12—N2—C13—C14	−177.5 (5)
O4[i]—Mo3—O10—Mo2[i]	5.27 (13)	C13—N2—C12—C11	−171.1 (5)
O5—Mo1—O2—Mo4[i]	−48.5 (4)	C13—C14—C15—C16	−177.6 (8)
O6—Mo2—O5—Mo1	170.08 (17)	N3—C21—C22—C23	−179.5 (12)
O7—Mo2—O5—Mo1	−81.38 (19)	C18—C19—C20—N3	168.1 (8)
O8—Mo2—O5—Mo1	32.2 (3)	C20—N3—C21—C22	170.1 (8)
O8—Mo3—O10—Mo2[i]	12.3 (4)	C21—N3—C20—C19	60.7 (9)
O9—Mo3—O10—Mo2[i]	−174.29 (16)	C21—C22—C23—C24	180.0 (14)
O10[i]—Mo2—O5—Mo1	91.78 (17)	C17X—C18—C19—C20	−95.2 (19)
O11[i]—Mo1—O2—Mo4[i]	−13.91 (15)	C17—C18—C19—C20	−176.1 (12)
O11—Mo3—O10—Mo2[i]	−67.48 (17)	N4—C29—C30—C31	64 (2)
O15—Mo5—O14—Mo4	130.5 (8)	N4—C29—C30X—C31X	168 (2)
O16—Mo5—O14—Mo4	6.5 (9)	C25—C26—C27—C28	−175.7 (8)
O17—Mo5—O14—Mo4	−112.0 (9)	C26—C27—C28—N4	178.8 (8)
N1—C5—C6—C7	−170.0 (7)	C28—N4—C29—C30	52.8 (12)
C1—C2—C3—C4	178.8 (7)	C28—N4—C29—C30X	60.7 (19)
C2—C3—C4—N1	−172.8 (6)	C29—N4—C28—C27	57.9 (9)
C4—N1—C5—C6	−77.2 (8)	C29—C30—C31—C32	63 (3)
C5—N1—C4—C3	−78.5 (7)	C29—C30X—C31X—C32X	−70 (4)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2[ii]	0.91	1.72	2.627 (6)	172
N1—H1B···O10[ii]	0.91	1.97	2.755 (6)	143
N2—H2C···O15	0.91	1.91	2.778 (6)	160
N2—H2D···O7[iii]	0.91	1.90	2.812 (5)	176
N3—H3A···O15[iv]	0.91	1.87	2.770 (6)	172
N3—H3B···O17	0.91	1.88	2.786 (6)	176
N4—H4A···O11	0.91	1.80	2.695 (6)	166
N4—H4B···O16	0.91	1.86	2.762 (7)	172

Symmetry codes: (ii) −x+2, −y+1, −z+1; (iii) −x+1, y+1/2, −z+3/2; (iv) −x+1, −y+1, −z+2.