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A cocrystal of two Mo^{VI} complexes bearing different diastereomers of the 2,4-di-*tert*-butyl-6-{[(1-oxido-1-phenylpropan-2-yl)(methyl)amino]methyl}-phenolate ligand derived from (+)-ephedrine

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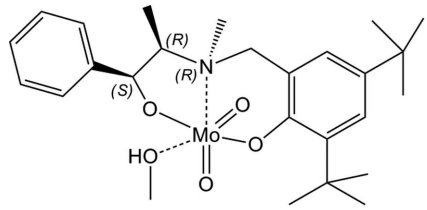
The title cocrystal contains two chiral conformational diastereomers, *viz.* (1*S*,2*R*,R_N)- and (1*S*,2*R*,S_N)-, of [2,4-di-*tert*-butyl-6-{[(1-oxido-1-phenylpropan-2-yl)(methyl)amino]-methyl}phenolato](methanol)-*cis*-dioxidomolybdenum(VI), $[Mo(C_{25}H_{35}NO_2)O_2(CH_3OH)]$, representing the first example of a structurally characterized molybdenum complex with enantiomerically pure ephedrine derivative ligands. The Mo^{VI} cations exhibit differently distorted octahedral coordination environments, with two oxide ligands positioned *cis* to each other. The remainder of the coordination comprises phenoxide, alkoxide and methanol O atoms, with an amine N atom completing the octahedron. The distinct complexes are linked by strong intermolecular O—H···O hydrogen bonds, resulting in one-dimensional molecular chains. Furthermore, the phenyl rings are involved in weak T-shaped/edge-to-face π–π interactions with each other.

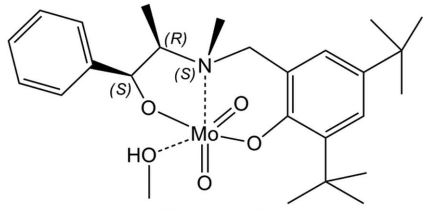
Comment

For the past several decades, high-valent molybdenum complexes have gained considerable attention in various catalytic oxidation reactions (Arzoumanian, 1998) and as biological model compounds (Hille, 1996; Collison *et al.*, 1996). Recently, oxomolybdenum complexes have appeared in novel studies concerning, for example, *X*—H (*X* = Si, B, P or H) bond activation (Sousa *et al.*, 2012) and hydrogen production from water (Karunadasa *et al.*, 2010), previously dominated by more noble metals. Ephedrine and its *N*-substituted derivatives are inexpensive, readily available in enantiomerically pure forms and relatively easy to modify, and thus are an interesting group of chiral ligands for various purposes (Yuan *et al.*, 2003; Kuznetsov *et al.*, 1999; Bouquillon *et al.*, 1999). These two strands of interest are combined in the title compound, (I).

The asymmetric unit of (I) contains two distinct Mo^{VI} complexes, *A* and *B*, which are conformational diastereomers (Fig. 1 and Table 1). The bonding and geometric parameters around the Mo^{VI} cation of *A* and *B* are comparable to some extent, but several differences can be noted, so the different ligand geometries of the two diastereomers will be discussed.



Complex *A*



Complex *B*

(I)

In *A* and *B*, the tridentate doubly deprotonated 2,4-di-*tert*-butyl-6-{[(1-oxido-1-phenylpropan-2-yl)(methyl)amino]-methyl}phenolate ($L1^{2-}$) ligand wraps around the *cis*-$[MoO_2]^{2+}$ fragment, with an additional coordinated methanol molecule producing a heavily distorted octahedral coordina-

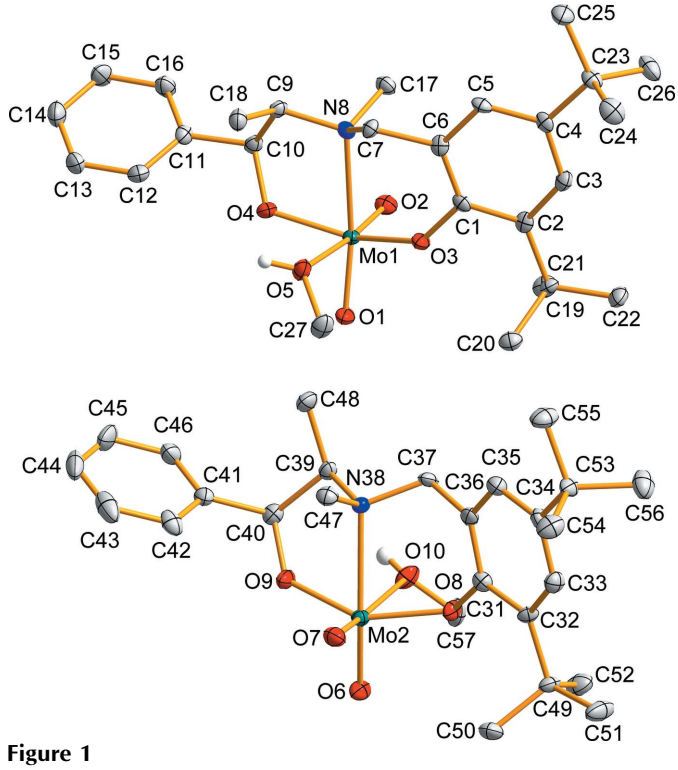


Figure 1
The molecular structure of complexes *A* (top) and *B* (bottom), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and C-bound H atoms have been omitted for clarity.

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tion geometry for the Mo^{VI} cation. The oxide ligands are strongly bound, with Mo═O distances of about 1.7 Å, thus indicating clear double-bond character. The phenoxide and alkoxide O atoms are arranged *trans* to each other, with a marginally longer Mo—O(phenoxide) distance compared with Mo—O(alkoxide), as might be expected from the different electronic nature and steric hindrance of the groups. The amine N and methanol O atoms are *trans* to the Mo═O groups and rather weakly bound to the Mo^{VI} cation, with relatively long bond lengths of over 2.35 Å. This weaker bonding can be explained by the lack of negative partial charge on the coordinating atoms (amine N and methanol O) and by the considerable *trans* influence induced by the oxide ligands. The different character of the coordinating groups emphasizes the distortion of the Mo^{VI} octahedron, which is evident from the *trans* angles around the metal cation (O1—Mo1—N8, O2—Mo1—O5 and O3—Mo1—O4 for *A*, and O6—Mo2—N38, O7—Mo2—O10 and O8—Mo2—O9 for *B*; Table 1) deviating by 7–34° from the value of 180° for a perfect octahedron. In general, the bond lengths and angles involving the Mo^{VI} centres are in good agreement with previous studies concerning $[MoO_2]^{2+}$ aminophenolates (Lehtonen & Sillanpää, 2005; Riisiö *et al.*, 2013) and aminoalcoholates (Cross *et al.*, 1999). The Flack (1983) parameter of 0.00 (3) indicates that the crystals of (I) are enantiomerically pure and the absolute configuration has been determined correctly.

The ligand geometries of complexes *A* and *B* are notably different, which can be seen from an overlay of the molecules (Fig. 2). The two carbon stereocentres of the $L1^{2-}$ ligand have the same configuration (1*S*,2*R*) in both molecules, but the amine N atoms have different stereochemistries due to the conformational change of the ligand, producing *R* (N8) and *S* (N38) configurations for *A* and *B*, respectively. This difference induces major changes in the coordination angles (Mo—O—C), general conformation and 'folding' of the ligand (Table 1). The most significant conformational changes of the ligand are seen when comparing the chelate angles of the ligands (N8—C9—C10—O4 and N38—C39—C40—O9; Table 1). Also, the torsion angles related to the position of methyl substituents C17 and C47 (C17—N8—C9—C18 and C47—N38—C39—C48) are quite dissimilar in *A* and *B* (Table 1). The Mo1—O3 bond is slightly shorter than Mo2—O8, which might be due to the increased π-bonding ability of atom O3 because of the larger Mo—O—C angle. The pyramidality of the amine N atom has been related to the donor ability of the atom (Hänninen *et al.*, 2011). For *B*, the torsion angle indicating the pyramidality of the N atom (C37—C39—C47—N48) is −35.9 (3)°, while the corresponding angle for *A* (C7—C9—C17—N8) is only 33.9 (2)°, indicating a less pyramidalized arrangement. The shorter Mo—N bond for complex *B* is in agreement with the above conclusion. Furthermore, the shorter Mo1—O5 bond compared with Mo2—O10 can be attributed to the tighter hydrogen bonding (O5—H5O···O6^{i} *versus* O10—H10O···O1; see Table 2 for symmetry code).

The solid-state ordering of the complexes is governed by strong intermolecular hydrogen bonds from the coordinated methanol molecule to an oxide ligand of a neighbouring complex (Table 2). These hydrogen bonds bind the molecules together, forming a one-dimensional chain of complexes (Fig. 3*a*). Furthermore, T-shaped/edge-to-face π–π interactions are present between the phenyl rings of the ephedrine part of the ligands (Fig. 3*b*). The distances between phenyl-ring centroids are in the range 4.7–5.2 Å and the angles between the phenyl-ring planes vary from 58 to 89°, thus

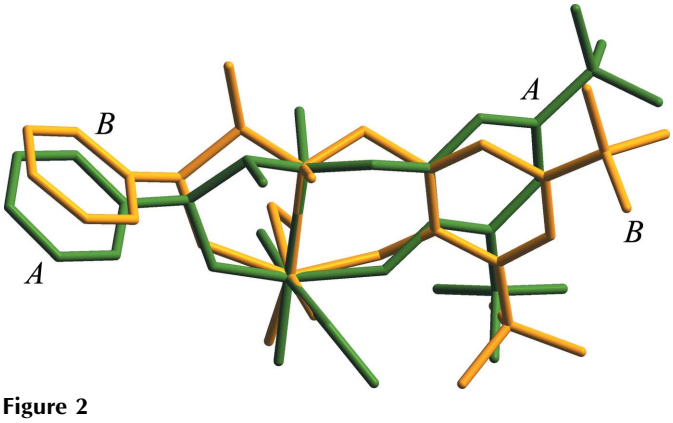

Figure 2
An overlay capped-sticks presentation of complexes *A* (green in the electronic version of the paper) and *B* (yellow). The overlaid atom pairs are C6/C36, N8/N38 and Mo1/Mo2.

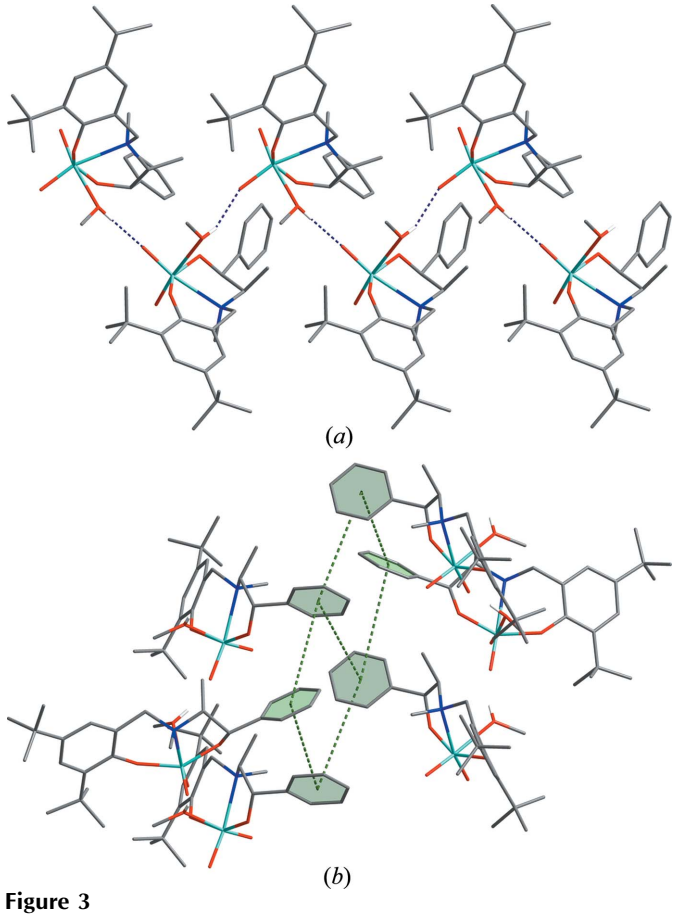

Figure 3
(*a*) A capped-sticks presentation of the O—H···O hydrogen bonds (dashed lines) forming chains of complex molecules in the *b* direction, and (*b*) the T-shaped/edge-to-face π–π interactions (dashed lines and shaded hexagons) in the structure.

supporting the presence of π–π interactions. Both of the preceding effects can be seen to enhance the crystallization of the compounds, thus contributing to the good quality and stability of the crystals. The solid-state structure of the complex does not contain any additional noncoordinating solvents or notable cavities.

Experimental

$MoO_2(acac)_2$ (acac is acetylacetate) was prepared according to the literature procedure of Chen *et al.* (1976). 2,4-Di-*tert*-butyl-6-{[(1-hydroxy-1-phenylpropan-2-yl)(methyl)amino]methyl}phenol (H_2L1) was synthesized by dissolving equimolar amounts of 2,4-di-*tert*-butylphenol, formaldehyde (36.5% water solution) and (1*S*,2*R*)-(+)-ephedrine hydrochloride in methanol. Two equivalents of triethylamine were added and the mixture was refluxed for two weeks, after which time the reaction did not proceed any further and roughly half of the starting materials were converted to products (determined by high-performance liquid chromatography). Small amounts of H_2L1 could be separated by crystallization from the methanol solution in a freezer and were used in the complexation reaction. Cocrystals were prepared by dissolving H_2L1 (0.10 mmol) and $MoO_2(acac)_2$ (0.10 mmol) in methanol (4 ml). The solution was stirred for 20 h, filtered and placed in a freezer. Pale-yellow crystals of (I) suitable for X-ray diffraction formed within a few days.

Crystal data

$[Mo(C_{25}H_{35}NO_2)O_2(CH_4O)]$
M_r = 541.52
Monoclinic, $P2_1$
a = 16.1560 (4) Å
b = 8.4129 (1) Å
c = 20.3545 (6) Å
β = 109.934 (1)°
V = 2600.80 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 123 K
0.36 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.608, T_{max} = 0.746
15118 measured reflections
9756 independent reflections
8963 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.085
S = 1.04
9756 reflections
621 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Absolute structure: Flack (1983), with 4298 Friedel pairs
Flack parameter: 0.00 (3)

All C-bound H atoms were placed in idealized positions and refined in riding mode, with C—H = 0.93 (aromatic), 0.96 (methyl) or 0.97 Å (methylene), and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms or 1.2U_{eq}(C) for aromatic and methylene H atoms. Hydroxy H atoms were located from the electron-density map and their positions were refined isotropically with O—H distances restrained to 0.82 (1) Å. Reflections $\bar{1}02$, 002 and 101 were omitted from the data because the F_o values were considerably smaller than the F_c values, as these reflections were partially obscured by the beam-stop during the data collection.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); molecular graphics: *DIAMOND* (Brandenburg, 1999) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Table 1
Selected geometric parameters (Å, °).

Mo1—O1	1.722 (3)	Mo2—O6	1.722 (2)
Mo1—O2	1.693 (3)	Mo2—O7	1.689 (3)
Mo1—O3	1.917 (2)	Mo2—O8	1.942 (3)
Mo1—O4	1.903 (3)	Mo2—O9	1.904 (3)
Mo1—O5	2.393 (3)	Mo2—O10	2.354 (3)
Mo1—N8	2.442 (3)	Mo2—N38	2.404 (3)
O1—Mo1—O2	104.01 (13)	O6—Mo2—O7	104.38 (13)
O1—Mo1—O3	99.14 (12)	O6—Mo2—O8	99.75 (12)
O1—Mo1—O4	100.84 (12)	O6—Mo2—O9	99.23 (12)
O1—Mo1—O5	83.11 (12)	O6—Mo2—O10	84.26 (11)
O2—Mo1—O5	172.87 (11)	O7—Mo2—O10	169.85 (12)
O3—Mo1—O4	145.84 (13)	O8—Mo2—O9	147.31 (11)
O1—Mo1—N8	169.16 (12)	O6—Mo2—N38	164.80 (11)
Mo1—O3—C1	140.5 (2)	Mo2—O8—C31	129.7 (2)
Mo1—O4—C10	119.6 (2)	Mo2—O9—C40	130.3 (2)
N8—C7—C6	113.0 (3)	N38—C37—C36	113.8 (3)
N8—C9—C10	104.3 (3)	N38—C39—C40	109.3 (3)
C17—N8—C9—C18	−146.5 (3)	C47—N38—C39—C48	45.1 (4)
N8—C9—C10—O4	53.2 (4)	N38—C39—C40—O9	−21.9 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5O···O6^{i}	0.81 (1)	1.96 (3)	2.690 (4)	148 (6)
O10—H10O···O1	0.82 (1)	1.84 (2)	2.633 (4)	165 (5)

Symmetry code: (i) $x, y-1, z$.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LG3109). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2013). C**69**, 509-512 [doi:10.1107/S0108270113010652]

A cocrystal of two Mo^{VI} complexes bearing different diastereomers of the 2,4-di-*tert*-butyl-6-{[(1-oxido-1-phenylpropan-2-yl)(methyl)amino]methyl}phenolate ligand derived from (+)-ephedrine

Reijo Sillanpää and Mikko M. Hänninen

[(1*S*,2*R*,R_N)-2,4-Di-*tert*-butyl-6-{[(1-oxido-1-\ phenylpropan-2-yl)(methyl)amino]methyl}phenolato](methanol)-*cis*-\ dioxidomolybdenum(VI)–[(1*S*,2*R*,S_N)-2,4-di-*tert*-\ butyl-6-{[(1-oxido-1-phenylpropan-2-\ yl)(methyl)amino]methyl}phenolato](methanol)-*cis*-dioxidomolybdenum(VI)

Crystal data

$[Mo(C_{25}H_{35}NO_2)O_2(CH_4O)]$
M_r = 541.52
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 16.1560 (4) Å
b = 8.4129 (1) Å
c = 20.3545 (6) Å
β = 109.934 (1)°
V = 2600.80 (10) Å^3
Z = 4
$F(000)$ = 1136
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4502 reflections
θ = 0.4–28.7°
μ = 0.54 mm^{-1}
T = 123 K
Plate, yellow
0.36 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Enraf–Nonius FR590
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.608, T_{max} = 0.746
15118 measured reflections
9756 independent reflections
8963 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26°, θ_{min} = 2.6°
h = −14→19
k = −10→10
l = −25→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.085
S = 1.04
9756 reflections
621 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 2.3404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Absolute structure: Flack (1983), with 4298 Friedel pairs
Flack parameter: 0.00 (3)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.356925 (17)	0.49021 (4)	0.849500 (15)	0.01348 (7)
Mo2	0.050199 (17)	0.97967 (4)	0.765051 (15)	0.01410 (7)
O1	0.27783 (17)	0.6343 (3)	0.81671 (15)	0.0201 (6)
O2	0.43944 (17)	0.5867 (3)	0.91100 (15)	0.0204 (6)
O3	0.40185 (14)	0.4785 (4)	0.77379 (12)	0.0166 (5)
O4	0.30770 (17)	0.3748 (3)	0.90769 (14)	0.0174 (6)
O5	0.25127 (18)	0.3246 (3)	0.76682 (16)	0.0205 (6)
O6	0.11718 (16)	1.1430 (3)	0.77312 (14)	0.0194 (6)
O7	−0.04313 (18)	1.0539 (3)	0.77283 (15)	0.0220 (6)
O8	0.00846 (15)	0.9542 (3)	0.66421 (13)	0.0163 (6)
O9	0.10678 (18)	0.8852 (3)	0.85415 (14)	0.0215 (6)
O10	0.16371 (19)	0.8432 (3)	0.74084 (16)	0.0232 (6)
N8	0.44682 (19)	0.2510 (3)	0.88457 (17)	0.0153 (7)
N38	−0.0070 (2)	0.7147 (3)	0.76097 (17)	0.0144 (7)
C1	0.4698 (2)	0.4135 (4)	0.7575 (2)	0.0151 (8)
C2	0.5071 (2)	0.4983 (5)	0.71536 (17)	0.0144 (7)
C3	0.5759 (2)	0.4251 (4)	0.7002 (2)	0.0172 (8)
H3	0.6006	0.4785	0.6713	0.021*
C4	0.6100 (2)	0.2761 (4)	0.7259 (2)	0.0152 (8)
C5	0.5708 (2)	0.1958 (4)	0.7672 (2)	0.0156 (8)
H5	0.5921	0.0964	0.7851	0.019*
C6	0.4998 (2)	0.2618 (4)	0.7824 (2)	0.0148 (8)
C7	0.4526 (2)	0.1662 (4)	0.8221 (2)	0.0150 (8)
H7A	0.3936	0.1418	0.791	0.018*
H7B	0.4834	0.0665	0.8369	0.018*
C9	0.4055 (2)	0.1524 (4)	0.9274 (2)	0.0156 (8)
H9	0.4529	0.0989	0.9643	0.019*
C10	0.3633 (2)	0.2756 (4)	0.9622 (2)	0.0165 (8)
H10	0.4104	0.3419	0.9931	0.02*
C11	0.3113 (2)	0.2061 (4)	1.0049 (2)	0.0169 (8)
C12	0.2225 (3)	0.1694 (4)	0.9744 (2)	0.0210 (9)
H12	0.1936	0.1895	0.9272	0.025*
C13	0.1772 (3)	0.1031 (5)	1.0141 (2)	0.0233 (9)
H13	0.1178	0.079	0.9935	0.028*
C14	0.2195 (3)	0.0722 (5)	1.0845 (2)	0.0291 (10)
H14	0.1885	0.0269	1.1108	0.035*
C15	0.3075 (3)	0.1086 (5)	1.1155 (2)	0.0284 (10)

H15	0.3363	0.0877	1.1627	0.034*
C16	0.3528 (3)	0.1773 (5)	1.0753 (2)	0.0226 (9)
H16	0.4119	0.204	1.0962	0.027*
C17	0.5389 (2)	0.2908 (4)	0.9307 (2)	0.0193 (8)
H17A	0.5732	0.1952	0.9424	0.029*
H17B	0.5373	0.3405	0.9728	0.029*
H17C	0.565	0.3622	0.9066	0.029*
C18	0.3408 (3)	0.0267 (4)	0.8868 (2)	0.0204 (9)
H18A	0.2928	0.0769	0.8512	0.031*
H18B	0.3185	−0.0307	0.918	0.031*
H18C	0.3701	−0.0457	0.8655	0.031*
C19	0.4739 (2)	0.6659 (4)	0.6866 (2)	0.0148 (8)
C20	0.3757 (3)	0.6624 (5)	0.6435 (2)	0.0211 (9)
H20A	0.3566	0.7673	0.6265	0.032*
H20B	0.3431	0.6264	0.6722	0.032*
H20C	0.3658	0.5913	0.6047	0.032*
C21	0.4897 (3)	0.7825 (4)	0.7484 (2)	0.0218 (9)
H21A	0.5517	0.7895	0.774	0.033*
H21B	0.4596	0.7448	0.7787	0.033*
H21C	0.4676	0.8857	0.7308	0.033*
C22	0.5231 (3)	0.7302 (5)	0.6399 (2)	0.0234 (9)
H22A	0.5151	0.659	0.6014	0.035*
H22B	0.5847	0.7387	0.6666	0.035*
H22C	0.5004	0.8331	0.6225	0.035*
C23	0.6875 (2)	0.2064 (4)	0.7074 (2)	0.0186 (8)
C24	0.6578 (3)	0.1755 (5)	0.6289 (2)	0.0283 (10)
H24A	0.7057	0.1304	0.6173	0.042*
H24B	0.64	0.2738	0.6041	0.042*
H24C	0.6091	0.1028	0.6159	0.042*
C25	0.7202 (3)	0.0488 (5)	0.7461 (3)	0.0281 (10)
H25A	0.6729	−0.027	0.7337	0.042*
H25B	0.74	0.067	0.7957	0.042*
H25C	0.768	0.0081	0.7332	0.042*
C26	0.7648 (3)	0.3256 (5)	0.7293 (3)	0.0290 (10)
H26A	0.7842	0.341	0.779	0.043*
H26B	0.7457	0.4254	0.7061	0.043*
H26C	0.8125	0.2846	0.7163	0.043*
C27	0.2057 (3)	0.3818 (5)	0.6974 (2)	0.0246 (9)
H27A	0.1675	0.4677	0.6994	0.037*
H27B	0.1714	0.2972	0.6695	0.037*
H27C	0.2477	0.4186	0.677	0.037*
C31	−0.0700 (2)	0.8955 (4)	0.6205 (2)	0.0162 (8)
C32	−0.1222 (2)	0.9814 (5)	0.56211 (17)	0.0153 (6)
C33	−0.2021 (3)	0.9153 (4)	0.5224 (2)	0.0198 (8)
H33	−0.2377	0.9727	0.4842	0.024*
C34	−0.2327 (2)	0.7675 (4)	0.5362 (2)	0.0169 (8)
C35	−0.1763 (3)	0.6818 (4)	0.5922 (2)	0.0192 (8)
H35	−0.1932	0.5818	0.6025	0.023*
C36	−0.0952 (3)	0.7431 (4)	0.6330 (2)	0.0180 (8)

C37	−0.0306 (3)	0.6443 (4)	0.6895 (2)	0.0179 (8)
H37A	−0.0557	0.5397	0.6898	0.021*
H37B	0.0227	0.6311	0.6783	0.021*
C39	0.0661 (2)	0.6177 (4)	0.8105 (2)	0.0162 (8)
H39	0.1064	0.5933	0.7852	0.019*
C40	0.1195 (2)	0.7236 (4)	0.8736 (2)	0.0174 (8)
H40	0.1818	0.7008	0.8821	0.021*
C41	0.1049 (3)	0.6964 (4)	0.9425 (2)	0.0196 (8)
C42	0.0449 (3)	0.7818 (5)	0.9626 (2)	0.0242 (9)
H42	0.0095	0.8569	0.9323	0.029*
C43	0.0364 (3)	0.7575 (6)	1.0275 (3)	0.0371 (12)
H43	−0.0045	0.8161	1.0402	0.045*
C44	0.0882 (4)	0.6474 (6)	1.0731 (3)	0.0459 (15)
H44	0.0831	0.6321	1.1168	0.055*
C45	0.1484 (4)	0.5591 (6)	1.0528 (3)	0.0457 (15)
H45	0.183	0.4826	1.0827	0.055*
C46	0.1568 (3)	0.5850 (5)	0.9886 (2)	0.0301 (10)
H46	0.1979	0.5269	0.9759	0.036*
C47	−0.0864 (2)	0.7173 (4)	0.7812 (2)	0.0190 (8)
H47A	−0.1284	0.7907	0.7518	0.029*
H47B	−0.0707	0.7502	0.8291	0.029*
H47C	−0.1118	0.6129	0.7759	0.029*
C48	0.0382 (3)	0.4583 (5)	0.8310 (2)	0.0247 (9)
H48A	0.0009	0.475	0.8583	0.037*
H48B	0.0894	0.3995	0.8579	0.037*
H48C	0.0066	0.3995	0.7896	0.037*
C49	−0.0901 (2)	1.1420 (4)	0.5419 (2)	0.0183 (8)
C50	−0.0758 (3)	1.2651 (5)	0.6011 (2)	0.0259 (10)
H50A	−0.0635	1.3672	0.5855	0.039*
H50B	−0.0271	1.2327	0.6412	0.039*
H50C	−0.128	1.2719	0.6135	0.039*
C51	−0.0040 (3)	1.1164 (5)	0.5272 (3)	0.0296 (10)
H51A	−0.0139	1.0425	0.4893	0.044*
H51B	0.04	1.0746	0.5682	0.044*
H51C	0.0158	1.2159	0.5148	0.044*
C52	−0.1576 (3)	1.2143 (5)	0.4765 (2)	0.0343 (11)
H52A	−0.1676	1.1425	0.4379	0.051*
H52B	−0.1355	1.3134	0.4658	0.051*
H52C	−0.2119	1.2325	0.4847	0.051*
C53	−0.3239 (3)	0.7063 (4)	0.4909 (2)	0.0199 (9)
C54	−0.3945 (3)	0.8249 (5)	0.4944 (2)	0.0260 (10)
H54A	−0.3926	0.8347	0.5419	0.039*
H54B	−0.4515	0.7874	0.4658	0.039*
H54C	−0.3835	0.9267	0.4777	0.039*
C55	−0.3446 (3)	0.5445 (5)	0.5170 (2)	0.0291 (11)
H55A	−0.3041	0.4661	0.5117	0.044*
H55B	−0.4036	0.5136	0.49	0.044*
H55C	−0.3389	0.5527	0.5653	0.044*
C56	−0.3284 (3)	0.6866 (6)	0.4145 (2)	0.0316 (11)

H56A	−0.3176	0.7873	0.3967	0.047*
H56B	−0.3858	0.6489	0.3868	0.047*
H56C	−0.2847	0.6114	0.4124	0.047*
C57	0.2059 (3)	0.9108 (5)	0.6966 (2)	0.0253 (9)
H57A	0.1622	0.9441	0.6536	0.038*
H57B	0.2437	0.8331	0.687	0.038*
H57C	0.2402	1.0009	0.7194	0.038*
H5O	0.225 (3)	0.264 (6)	0.784 (3)	0.059 (19)*
H10O	0.198 (2)	0.786 (4)	0.7704 (18)	0.025 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01271 (14)	0.01256 (14)	0.01522 (15)	0.00186 (15)	0.00483 (11)	0.00129 (16)
Mo2	0.01390 (14)	0.01320 (14)	0.01498 (15)	−0.00125 (16)	0.00462 (11)	−0.00027 (17)
O1	0.0192 (14)	0.0187 (14)	0.0253 (17)	0.0064 (11)	0.0112 (13)	0.0058 (12)
O2	0.0198 (14)	0.0185 (14)	0.0218 (16)	0.0009 (11)	0.0057 (12)	−0.0018 (11)
O3	0.0162 (11)	0.0170 (11)	0.0183 (12)	0.0046 (13)	0.0080 (10)	0.0024 (14)
O4	0.0175 (14)	0.0164 (13)	0.0167 (15)	0.0030 (10)	0.0038 (12)	0.0048 (11)
O5	0.0201 (15)	0.0209 (15)	0.0186 (16)	−0.0056 (11)	0.0042 (13)	0.0036 (12)
O6	0.0159 (14)	0.0202 (14)	0.0189 (15)	−0.0040 (11)	0.0018 (12)	0.0017 (11)
O7	0.0250 (15)	0.0180 (13)	0.0261 (17)	−0.0022 (11)	0.0126 (13)	−0.0038 (11)
O8	0.0128 (12)	0.0187 (16)	0.0137 (13)	−0.0013 (10)	−0.0004 (10)	0.0025 (10)
O9	0.0281 (16)	0.0177 (14)	0.0133 (15)	−0.0039 (11)	0.0000 (12)	0.0030 (11)
O10	0.0190 (15)	0.0247 (16)	0.0243 (18)	0.0087 (12)	0.0052 (13)	0.0106 (13)
N8	0.0126 (16)	0.0148 (16)	0.0169 (17)	−0.0018 (12)	0.0030 (13)	−0.0029 (13)
N38	0.0151 (16)	0.0124 (15)	0.0152 (17)	0.0000 (12)	0.0044 (14)	0.0012 (12)
C1	0.0107 (18)	0.0165 (17)	0.016 (2)	−0.0013 (14)	0.0022 (16)	−0.0034 (15)
C2	0.0159 (16)	0.0121 (17)	0.0136 (16)	−0.0056 (17)	0.0030 (13)	−0.0054 (18)
C3	0.019 (2)	0.0149 (18)	0.018 (2)	−0.0038 (14)	0.0066 (17)	0.0004 (14)
C4	0.0123 (18)	0.0146 (18)	0.017 (2)	−0.0039 (14)	0.0036 (16)	−0.0061 (15)
C5	0.0131 (19)	0.0142 (18)	0.019 (2)	0.0041 (14)	0.0041 (16)	−0.0020 (15)
C6	0.0171 (19)	0.0136 (18)	0.014 (2)	−0.0039 (14)	0.0054 (16)	−0.0050 (15)
C7	0.0186 (19)	0.0118 (18)	0.015 (2)	−0.0012 (14)	0.0067 (16)	−0.0004 (14)
C9	0.0176 (19)	0.0171 (19)	0.0121 (19)	0.0019 (15)	0.0050 (16)	0.0026 (15)
C10	0.0167 (19)	0.0183 (19)	0.013 (2)	−0.0021 (14)	0.0026 (16)	0.0022 (15)
C11	0.020 (2)	0.0157 (18)	0.018 (2)	0.0030 (14)	0.0110 (17)	0.0002 (15)
C12	0.020 (2)	0.019 (2)	0.024 (2)	0.0037 (15)	0.0065 (18)	0.0061 (16)
C13	0.016 (2)	0.024 (2)	0.030 (3)	−0.0018 (15)	0.0075 (18)	0.0040 (17)
C14	0.028 (2)	0.031 (2)	0.036 (3)	−0.0010 (18)	0.020 (2)	0.006 (2)
C15	0.026 (2)	0.044 (3)	0.017 (2)	0.0033 (19)	0.0083 (19)	0.0087 (19)
C16	0.017 (2)	0.031 (2)	0.018 (2)	−0.0010 (16)	0.0041 (17)	0.0013 (17)
C17	0.0128 (19)	0.020 (2)	0.023 (2)	−0.0003 (15)	0.0033 (17)	−0.0006 (16)
C18	0.021 (2)	0.020 (2)	0.024 (2)	−0.0021 (14)	0.0115 (17)	0.0007 (15)
C19	0.0174 (19)	0.0120 (18)	0.014 (2)	0.0013 (14)	0.0035 (16)	0.0031 (14)
C20	0.024 (2)	0.0166 (19)	0.022 (2)	0.0026 (15)	0.0063 (18)	0.0006 (16)
C21	0.026 (2)	0.0152 (19)	0.022 (2)	−0.0018 (16)	0.0051 (19)	−0.0045 (17)
C22	0.026 (2)	0.016 (2)	0.031 (3)	0.0010 (16)	0.014 (2)	0.0065 (17)
C23	0.017 (2)	0.0172 (19)	0.023 (2)	0.0019 (15)	0.0079 (17)	−0.0044 (16)
C24	0.028 (2)	0.031 (2)	0.027 (3)	0.0009 (18)	0.011 (2)	−0.0077 (19)

C25	0.026 (2)	0.025 (2)	0.038 (3)	0.0091 (17)	0.017 (2)	0.0059 (18)
C26	0.017 (2)	0.026 (2)	0.047 (3)	−0.0009 (17)	0.014 (2)	−0.006 (2)
C27	0.027 (2)	0.029 (2)	0.015 (2)	−0.0055 (17)	0.0037 (18)	0.0007 (17)
C31	0.0136 (19)	0.0174 (19)	0.018 (2)	0.0000 (14)	0.0059 (16)	−0.0021 (16)
C32	0.0140 (15)	0.0122 (15)	0.0189 (17)	0.0026 (19)	0.0045 (13)	0.003 (2)
C33	0.019 (2)	0.0200 (19)	0.017 (2)	0.0016 (15)	0.0016 (17)	0.0027 (16)
C34	0.0168 (19)	0.0192 (19)	0.013 (2)	−0.0027 (15)	0.0029 (16)	−0.0030 (15)
C35	0.023 (2)	0.0171 (19)	0.018 (2)	−0.0027 (15)	0.0067 (17)	−0.0021 (15)
C36	0.026 (2)	0.0142 (19)	0.015 (2)	0.0052 (15)	0.0078 (17)	−0.0001 (15)
C37	0.024 (2)	0.0082 (17)	0.017 (2)	0.0001 (14)	0.0010 (17)	−0.0009 (15)
C39	0.0186 (19)	0.0157 (19)	0.014 (2)	0.0041 (15)	0.0051 (16)	0.0021 (15)
C40	0.0142 (19)	0.0194 (19)	0.018 (2)	0.0003 (15)	0.0051 (16)	0.0023 (16)
C41	0.025 (2)	0.0170 (19)	0.017 (2)	−0.0049 (15)	0.0082 (18)	−0.0019 (15)
C42	0.018 (2)	0.030 (2)	0.023 (2)	−0.0072 (16)	0.0057 (18)	−0.0093 (18)
C43	0.036 (3)	0.049 (3)	0.035 (3)	−0.022 (2)	0.023 (2)	−0.021 (2)
C44	0.073 (4)	0.051 (3)	0.018 (3)	−0.037 (3)	0.020 (3)	−0.010 (2)
C45	0.078 (4)	0.034 (3)	0.015 (3)	−0.011 (3)	0.003 (3)	0.008 (2)
C46	0.046 (3)	0.020 (2)	0.019 (2)	0.0014 (19)	0.004 (2)	−0.0032 (17)
C47	0.017 (2)	0.0187 (19)	0.021 (2)	−0.0033 (15)	0.0062 (17)	−0.0008 (16)
C48	0.033 (2)	0.018 (2)	0.019 (2)	0.0001 (17)	0.0041 (17)	0.0056 (17)
C49	0.020 (2)	0.0127 (18)	0.019 (2)	−0.0022 (15)	0.0024 (17)	0.0041 (15)
C50	0.035 (2)	0.0132 (19)	0.028 (3)	−0.0007 (17)	0.009 (2)	−0.0025 (17)
C51	0.034 (3)	0.024 (2)	0.037 (3)	−0.0005 (18)	0.021 (2)	0.0058 (19)
C52	0.038 (3)	0.023 (2)	0.028 (3)	−0.0079 (18)	−0.007 (2)	0.0092 (19)
C53	0.022 (2)	0.0177 (19)	0.015 (2)	−0.0060 (15)	−0.0004 (17)	−0.0012 (15)
C54	0.017 (2)	0.028 (2)	0.031 (3)	−0.0026 (16)	0.0049 (19)	0.0046 (19)
C55	0.026 (2)	0.023 (2)	0.031 (3)	−0.0084 (16)	0.001 (2)	0.0040 (18)
C56	0.030 (2)	0.042 (3)	0.022 (2)	−0.013 (2)	0.007 (2)	−0.007 (2)
C57	0.026 (2)	0.034 (2)	0.020 (2)	0.0051 (17)	0.0127 (19)	0.0019 (18)

Geometric parameters (Å, º)

Mo1—O1	1.722 (3)	C23—C26	1.543 (5)
Mo1—O2	1.693 (3)	C24—H24A	0.96
Mo1—O3	1.917 (2)	C24—H24B	0.96
Mo1—O4	1.903 (3)	C24—H24C	0.96
Mo1—O5	2.393 (3)	C25—H25A	0.96
Mo1—N8	2.442 (3)	C25—H25B	0.96
Mo2—O6	1.722 (2)	C25—H25C	0.96
Mo2—O7	1.689 (3)	C26—H26A	0.96
Mo2—O8	1.942 (3)	C26—H26B	0.96
Mo2—O9	1.904 (3)	C26—H26C	0.96
Mo2—O10	2.354 (3)	C27—H27A	0.96
Mo2—N38	2.404 (3)	C27—H27B	0.96
O3—C1	1.365 (4)	C27—H27C	0.96
O4—C10	1.433 (4)	C31—C36	1.395 (5)
O5—C27	1.435 (5)	C31—C32	1.401 (5)
O5—H5O	0.814 (10)	C32—C33	1.384 (5)
O8—C31	1.368 (4)	C32—C49	1.552 (5)
O9—C40	1.411 (4)	C33—C34	1.402 (5)

O10—C57	1.419 (5)	C33—H33	0.93
O10—H10O	0.817 (10)	C34—C35	1.394 (5)
N8—C7	1.488 (5)	C34—C53	1.535 (5)
N8—C17	1.502 (5)	C35—C36	1.389 (5)
N8—C9	1.514 (5)	C35—H35	0.93
N38—C47	1.475 (5)	C36—C37	1.511 (5)
N38—C37	1.495 (5)	C37—H37A	0.97
N38—C39	1.505 (5)	C37—H37B	0.97
C1—C6	1.398 (5)	C39—C48	1.517 (5)
C1—C2	1.400 (5)	C39—C40	1.560 (5)
C2—C3	1.394 (5)	C39—H39	0.98
C2—C19	1.551 (5)	C40—C41	1.517 (6)
C3—C4	1.396 (5)	C40—H40	0.98
C3—H3	0.93	C41—C42	1.377 (6)
C4—C5	1.388 (5)	C41—C46	1.387 (6)
C4—C23	1.542 (5)	C42—C43	1.389 (6)
C5—C6	1.401 (5)	C42—H42	0.93
C5—H5	0.93	C43—C44	1.374 (7)
C6—C7	1.517 (5)	C43—H43	0.93
C7—H7A	0.97	C44—C45	1.395 (8)
C7—H7B	0.97	C44—H44	0.93
C9—C18	1.519 (5)	C45—C46	1.377 (7)
C9—C10	1.538 (5)	C45—H45	0.93
C9—H9	0.98	C46—H46	0.93
C10—C11	1.518 (5)	C47—H47A	0.96
C10—H10	0.98	C47—H47B	0.96
C11—C16	1.380 (6)	C47—H47C	0.96
C11—C12	1.390 (5)	C48—H48A	0.96
C12—C13	1.380 (6)	C48—H48B	0.96
C12—H12	0.93	C48—H48C	0.96
C13—C14	1.385 (6)	C49—C52	1.530 (5)
C13—H13	0.93	C49—C51	1.535 (5)
C14—C15	1.380 (6)	C49—C50	1.546 (6)
C14—H14	0.93	C50—H50A	0.96
C15—C16	1.396 (6)	C50—H50B	0.96
C15—H15	0.93	C50—H50C	0.96
C16—H16	0.93	C51—H51A	0.96
C17—H17A	0.96	C51—H51B	0.96
C17—H17B	0.96	C51—H51C	0.96
C17—H17C	0.96	C52—H52A	0.96
C18—H18A	0.96	C52—H52B	0.96
C18—H18B	0.96	C52—H52C	0.96
C18—H18C	0.96	C53—C54	1.535 (6)
C19—C20	1.529 (5)	C53—C55	1.538 (5)
C19—C22	1.531 (5)	C53—C56	1.541 (6)
C19—C21	1.546 (5)	C54—H54A	0.96
C20—H20A	0.96	C54—H54B	0.96
C20—H20B	0.96	C54—H54C	0.96
C20—H20C	0.96	C55—H55A	0.96

C21—H21A	0.96	C55—H55B	0.96
C21—H21B	0.96	C55—H55C	0.96
C21—H21C	0.96	C56—H56A	0.96
C22—H22A	0.96	C56—H56B	0.96
C22—H22B	0.96	C56—H56C	0.96
C22—H22C	0.96	C57—H57A	0.96
C23—C24	1.526 (6)	C57—H57B	0.96
C23—C25	1.542 (6)	C57—H57C	0.96

O1—Mo1—O2	104.01 (13)	C4—C23—C25	111.5 (3)
O1—Mo1—O3	99.14 (12)	C24—C23—C26	110.4 (4)
O1—Mo1—O4	100.84 (12)	C4—C23—C26	109.0 (3)
O1—Mo1—O5	83.11 (12)	C25—C23—C26	108.0 (3)
O2—Mo1—O5	172.87 (11)	C23—C24—H24A	109.5
O3—Mo1—O4	145.84 (13)	C23—C24—H24B	109.5
O1—Mo1—N8	169.16 (12)	H24A—C24—H24B	109.5
Mo1—O3—C1	140.5 (2)	C23—C24—H24C	109.5
Mo1—O4—C10	119.6 (2)	H24A—C24—H24C	109.5
N8—C7—C6	113.0 (3)	H24B—C24—H24C	109.5
N8—C9—C10	104.3 (3)	C23—C25—H25A	109.5
O6—Mo2—O7	104.38 (13)	C23—C25—H25B	109.5
O6—Mo2—O8	99.75 (12)	H25A—C25—H25B	109.5
O6—Mo2—O9	99.23 (12)	C23—C25—H25C	109.5
O6—Mo2—O10	84.26 (11)	H25A—C25—H25C	109.5
O7—Mo2—O10	169.85 (12)	H25B—C25—H25C	109.5
O8—Mo2—O9	147.31 (11)	C23—C26—H26A	109.5
O6—Mo2—N38	164.80 (11)	C23—C26—H26B	109.5
Mo2—O8—C31	129.7 (2)	H26A—C26—H26B	109.5
Mo2—O9—C40	130.3 (2)	C23—C26—H26C	109.5
N38—C37—C36	113.8 (3)	H26A—C26—H26C	109.5
N38—C39—C40	109.3 (3)	H26B—C26—H26C	109.5
O2—Mo1—O3	101.57 (12)	O5—C27—H27A	109.5
O2—Mo1—O4	100.10 (13)	O5—C27—H27B	109.5
O4—Mo1—O5	77.96 (11)	H27A—C27—H27B	109.5
O3—Mo1—O5	77.27 (11)	O5—C27—H27C	109.5
O2—Mo1—N8	86.52 (11)	H27A—C27—H27C	109.5
O4—Mo1—N8	74.37 (11)	H27B—C27—H27C	109.5
O3—Mo1—N8	80.93 (12)	O8—C31—C36	118.4 (3)
O5—Mo1—N8	86.35 (10)	O8—C31—C32	121.5 (3)
O7—Mo2—O9	102.66 (13)	C36—C31—C32	120.0 (4)
O7—Mo2—O8	98.08 (12)	C33—C32—C31	117.4 (4)
O9—Mo2—O10	80.82 (11)	C33—C32—C49	121.6 (3)
O8—Mo2—O10	74.90 (10)	C31—C32—C49	121.0 (3)
O7—Mo2—N38	90.07 (11)	C32—C33—C34	124.2 (4)
O9—Mo2—N38	72.52 (11)	C32—C33—H33	117.9
O8—Mo2—N38	82.54 (11)	C34—C33—H33	117.9
O10—Mo2—N38	81.84 (10)	C35—C34—C33	116.4 (3)
C27—O5—Mo1	120.0 (2)	C35—C34—C53	122.9 (3)
C27—O5—H5O	118 (4)	C33—C34—C53	120.6 (3)

Mo1—O5—H5O	114 (4)	C36—C35—C34	121.2 (3)
C57—O10—Mo2	120.9 (2)	C36—C35—H35	119.4
C57—O10—H10O	111 (3)	C34—C35—H35	119.4
Mo2—O10—H10O	120 (3)	C35—C36—C31	120.4 (4)
C7—N8—C17	107.7 (3)	C35—C36—C37	121.1 (3)
C7—N8—C9	113.5 (3)	C31—C36—C37	118.4 (4)
C17—N8—C9	107.4 (3)	N38—C37—H37A	108.8
C7—N8—Mo1	110.5 (2)	C36—C37—H37A	108.8
C17—N8—Mo1	111.3 (2)	N38—C37—H37B	108.8
C9—N8—Mo1	106.6 (2)	C36—C37—H37B	108.8
C47—N38—C37	108.7 (3)	H37A—C37—H37B	107.7
C47—N38—C39	112.3 (3)	N38—C39—C48	115.1 (3)
C37—N38—C39	108.6 (3)	C48—C39—C40	114.3 (3)
C47—N38—Mo2	109.7 (2)	N38—C39—H39	105.7
C37—N38—Mo2	111.6 (2)	C48—C39—H39	105.7
C39—N38—Mo2	106.0 (2)	C40—C39—H39	105.7
O3—C1—C6	119.1 (3)	O9—C40—C41	110.6 (3)
O3—C1—C2	119.6 (3)	O9—C40—C39	109.4 (3)
C6—C1—C2	121.3 (3)	C41—C40—C39	117.3 (3)
C3—C2—C1	116.9 (4)	O9—C40—H40	106.3
C3—C2—C19	121.2 (3)	C41—C40—H40	106.3
C1—C2—C19	121.9 (3)	C39—C40—H40	106.3
C2—C3—C4	123.8 (4)	C42—C41—C46	118.5 (4)
C2—C3—H3	118.1	C42—C41—C40	122.9 (4)
C4—C3—H3	118.1	C46—C41—C40	118.6 (4)
C5—C4—C3	117.4 (3)	C41—C42—C43	120.9 (4)
C5—C4—C23	122.6 (3)	C41—C42—H42	119.6
C3—C4—C23	120.0 (3)	C43—C42—H42	119.6
C4—C5—C6	121.2 (3)	C44—C43—C42	120.5 (5)
C4—C5—H5	119.4	C44—C43—H43	119.7
C6—C5—H5	119.4	C42—C43—H43	119.7
C1—C6—C5	119.3 (3)	C43—C44—C45	119.0 (5)
C1—C6—C7	120.1 (3)	C43—C44—H44	120.5
C5—C6—C7	120.4 (3)	C45—C44—H44	120.5
N8—C7—H7A	109	C46—C45—C44	120.1 (5)
C6—C7—H7A	109	C46—C45—H45	120
N8—C7—H7B	109	C44—C45—H45	120
C6—C7—H7B	109	C45—C46—C41	121.1 (5)
H7A—C7—H7B	107.8	C45—C46—H46	119.4
N8—C9—C18	115.1 (3)	C41—C46—H46	119.4
C18—C9—C10	112.9 (3)	N38—C47—H47A	109.5
N8—C9—H9	108.1	N38—C47—H47B	109.5
C18—C9—H9	108.1	H47A—C47—H47B	109.5
C10—C9—H9	108.1	N38—C47—H47C	109.5
O4—C10—C11	110.1 (3)	H47A—C47—H47C	109.5
O4—C10—C9	107.2 (3)	H47B—C47—H47C	109.5
C11—C10—C9	115.0 (3)	C39—C48—H48A	109.5
O4—C10—H10	108.1	C39—C48—H48B	109.5
C11—C10—H10	108.1	H48A—C48—H48B	109.5

C9—C10—H10	108.1	C39—C48—H48C	109.5
C16—C11—C12	119.2 (4)	H48A—C48—H48C	109.5
C16—C11—C10	119.6 (3)	H48B—C48—H48C	109.5
C12—C11—C10	121.2 (4)	C52—C49—C51	108.1 (4)
C13—C12—C11	120.1 (4)	C52—C49—C50	106.6 (3)
C13—C12—H12	120	C51—C49—C50	110.0 (3)
C11—C12—H12	120	C52—C49—C32	111.9 (3)
C12—C13—C14	120.6 (4)	C51—C49—C32	109.7 (3)
C12—C13—H13	119.7	C50—C49—C32	110.6 (3)
C14—C13—H13	119.7	C49—C50—H50A	109.5
C15—C14—C13	120.0 (4)	C49—C50—H50B	109.5
C15—C14—H14	120	H50A—C50—H50B	109.5
C13—C14—H14	120	C49—C50—H50C	109.5
C14—C15—C16	119.3 (4)	H50A—C50—H50C	109.5
C14—C15—H15	120.4	H50B—C50—H50C	109.5
C16—C15—H15	120.4	C49—C51—H51A	109.5
C11—C16—C15	121.0 (4)	C49—C51—H51B	109.5
C11—C16—H16	119.5	H51A—C51—H51B	109.5
C15—C16—H16	119.5	C49—C51—H51C	109.5
N8—C17—H17A	109.5	H51A—C51—H51C	109.5
N8—C17—H17B	109.5	H51B—C51—H51C	109.5
H17A—C17—H17B	109.5	C49—C52—H52A	109.5
N8—C17—H17C	109.5	C49—C52—H52B	109.5
H17A—C17—H17C	109.5	H52A—C52—H52B	109.5
H17B—C17—H17C	109.5	C49—C52—H52C	109.5
C9—C18—H18A	109.5	H52A—C52—H52C	109.5
C9—C18—H18B	109.5	H52B—C52—H52C	109.5
H18A—C18—H18B	109.5	C34—C53—C54	109.3 (3)
C9—C18—H18C	109.5	C34—C53—C55	111.5 (3)
H18A—C18—H18C	109.5	C54—C53—C55	107.9 (3)
H18B—C18—H18C	109.5	C34—C53—C56	110.4 (3)
C20—C19—C22	107.9 (3)	C54—C53—C56	109.6 (4)
C20—C19—C21	109.3 (3)	C55—C53—C56	108.1 (3)
C22—C19—C21	107.7 (3)	C53—C54—H54A	109.5
C20—C19—C2	110.9 (3)	C53—C54—H54B	109.5
C22—C19—C2	111.7 (3)	H54A—C54—H54B	109.5
C21—C19—C2	109.3 (3)	C53—C54—H54C	109.5
C19—C20—H20A	109.5	H54A—C54—H54C	109.5
C19—C20—H20B	109.5	H54B—C54—H54C	109.5
H20A—C20—H20B	109.5	C53—C55—H55A	109.5
C19—C20—H20C	109.5	C53—C55—H55B	109.5
H20A—C20—H20C	109.5	H55A—C55—H55B	109.5
H20B—C20—H20C	109.5	C53—C55—H55C	109.5
C19—C21—H21A	109.5	H55A—C55—H55C	109.5
C19—C21—H21B	109.5	H55B—C55—H55C	109.5
H21A—C21—H21B	109.5	C53—C56—H56A	109.5
C19—C21—H21C	109.5	C53—C56—H56B	109.5
H21A—C21—H21C	109.5	H56A—C56—H56B	109.5
H21B—C21—H21C	109.5	C53—C56—H56C	109.5

C19—C22—H22A	109.5	H56A—C56—H56C	109.5
C19—C22—H22B	109.5	H56B—C56—H56C	109.5
H22A—C22—H22B	109.5	O10—C57—H57A	109.5
C19—C22—H22C	109.5	O10—C57—H57B	109.5
H22A—C22—H22C	109.5	H57A—C57—H57B	109.5
H22B—C22—H22C	109.5	O10—C57—H57C	109.5
C24—C23—C4	109.2 (3)	H57A—C57—H57C	109.5
C24—C23—C25	108.6 (3)	H57B—C57—H57C	109.5

C17—N8—C9—C18	−146.5 (3)	C17—N8—C9—C10	89.2 (3)
N8—C9—C10—O4	53.2 (4)	Mo1—N8—C9—C10	−30.1 (3)
C47—N38—C39—C48	45.1 (4)	Mo1—O4—C10—C11	175.6 (2)
N38—C39—C40—O9	−21.9 (4)	Mo1—O4—C10—C9	−58.6 (3)
O2—Mo1—O3—C1	63.4 (4)	C18—C9—C10—O4	−72.5 (4)
O1—Mo1—O3—C1	169.8 (4)	N8—C9—C10—C11	176.0 (3)
O4—Mo1—O3—C1	−65.0 (4)	C18—C9—C10—C11	50.3 (4)
O5—Mo1—O3—C1	−109.4 (4)	O4—C10—C11—C16	−147.7 (3)
N8—Mo1—O3—C1	−21.2 (4)	C9—C10—C11—C16	91.1 (4)
O2—Mo1—O4—C10	−53.7 (3)	O4—C10—C11—C12	32.9 (5)
O1—Mo1—O4—C10	−160.2 (3)	C9—C10—C11—C12	−88.3 (4)
O3—Mo1—O4—C10	75.1 (3)	C16—C11—C12—C13	−0.8 (6)
O5—Mo1—O4—C10	119.4 (3)	C10—C11—C12—C13	178.6 (4)
N8—Mo1—O4—C10	29.8 (2)	C11—C12—C13—C14	−0.1 (6)
O1—Mo1—O5—C27	43.7 (3)	C12—C13—C14—C15	0.4 (6)
O4—Mo1—O5—C27	146.4 (3)	C13—C14—C15—C16	0.3 (7)
O3—Mo1—O5—C27	−57.3 (3)	C12—C11—C16—C15	1.6 (6)
N8—Mo1—O5—C27	−138.8 (3)	C10—C11—C16—C15	−177.9 (4)
O7—Mo2—O8—C31	−43.4 (3)	C14—C15—C16—C11	−1.3 (7)
O6—Mo2—O8—C31	−149.6 (3)	C3—C2—C19—C20	122.5 (4)
O9—Mo2—O8—C31	85.7 (3)	C1—C2—C19—C20	−57.5 (5)
O10—Mo2—O8—C31	129.1 (3)	C3—C2—C19—C22	2.1 (5)
N38—Mo2—O8—C31	45.6 (3)	C1—C2—C19—C22	−177.9 (3)
O7—Mo2—O9—C40	108.6 (3)	C3—C2—C19—C21	−117.0 (4)
O6—Mo2—O9—C40	−144.3 (3)	C1—C2—C19—C21	63.0 (4)
O8—Mo2—O9—C40	−19.5 (4)	C5—C4—C23—C24	114.4 (4)
O10—Mo2—O9—C40	−61.7 (3)	C3—C4—C23—C24	−65.0 (4)
N38—Mo2—O9—C40	22.6 (3)	C5—C4—C23—C25	−5.7 (5)
O7—Mo2—O10—C57	106.3 (7)	C3—C4—C23—C25	174.9 (4)
O6—Mo2—O10—C57	−42.5 (3)	C5—C4—C23—C26	−125.0 (4)
O9—Mo2—O10—C57	−142.8 (3)	C3—C4—C23—C26	55.7 (5)
O8—Mo2—O10—C57	59.3 (3)	Mo2—O8—C31—C36	−56.0 (4)
N38—Mo2—O10—C57	143.7 (3)	Mo2—O8—C31—C32	127.3 (3)
O2—Mo1—N8—C7	−131.5 (2)	O8—C31—C32—C33	−177.5 (3)
O1—Mo1—N8—C7	62.0 (7)	C36—C31—C32—C33	5.8 (5)
O4—Mo1—N8—C7	127.0 (2)	O8—C31—C32—C49	4.2 (5)
O3—Mo1—N8—C7	−29.2 (2)	C36—C31—C32—C49	−172.4 (3)
O5—Mo1—N8—C7	48.5 (2)	C31—C32—C33—C34	−1.3 (6)
O2—Mo1—N8—C17	−11.9 (3)	C49—C32—C33—C34	176.9 (4)
O1—Mo1—N8—C17	−178.4 (6)	C32—C33—C34—C35	−2.4 (6)

O4—Mo1—N8—C17	−113.4 (3)	C32—C33—C34—C53	177.7 (4)
O3—Mo1—N8—C17	90.4 (3)	C33—C34—C35—C36	1.7 (6)
O5—Mo1—N8—C17	168.0 (3)	C53—C34—C35—C36	−178.4 (4)
O2—Mo1—N8—C9	104.8 (2)	C34—C35—C36—C31	2.7 (6)
O1—Mo1—N8—C9	−61.7 (7)	C34—C35—C36—C37	−174.3 (4)
O4—Mo1—N8—C9	3.3 (2)	O8—C31—C36—C35	176.6 (3)
O3—Mo1—N8—C9	−152.9 (2)	C32—C31—C36—C35	−6.6 (6)
O5—Mo1—N8—C9	−75.2 (2)	O8—C31—C36—C37	−6.2 (5)
O7—Mo2—N38—C47	−11.8 (3)	C32—C31—C36—C37	170.5 (3)
O6—Mo2—N38—C47	150.3 (4)	C47—N38—C37—C36	65.0 (4)
O9—Mo2—N38—C47	91.4 (3)	C39—N38—C37—C36	−172.6 (3)
O8—Mo2—N38—C47	−110.0 (2)	Mo2—N38—C37—C36	−56.1 (4)
O10—Mo2—N38—C47	174.3 (3)	C35—C36—C37—N38	−120.4 (4)
O7—Mo2—N38—C37	108.7 (2)	C31—C36—C37—N38	62.5 (5)
O6—Mo2—N38—C37	−89.3 (5)	C37—N38—C39—C48	−75.1 (4)
O9—Mo2—N38—C37	−148.1 (3)	Mo2—N38—C39—C48	164.8 (3)
O8—Mo2—N38—C37	10.5 (2)	C47—N38—C39—C40	−85.2 (4)
O10—Mo2—N38—C37	−65.2 (2)	C37—N38—C39—C40	154.6 (3)
O7—Mo2—N38—C39	−133.2 (2)	Mo2—N38—C39—C40	34.5 (3)
O6—Mo2—N38—C39	28.8 (6)	Mo2—O9—C40—C41	−139.2 (3)
O9—Mo2—N38—C39	−30.0 (2)	Mo2—O9—C40—C39	−8.6 (5)
O8—Mo2—N38—C39	128.6 (2)	C48—C39—C40—O9	−152.6 (3)
O10—Mo2—N38—C39	52.9 (2)	N38—C39—C40—C41	105.0 (4)
Mo1—O3—C1—C6	37.5 (6)	C48—C39—C40—C41	−25.7 (5)
Mo1—O3—C1—C2	−143.6 (3)	O9—C40—C41—C42	32.9 (5)
O3—C1—C2—C3	−179.6 (3)	C39—C40—C41—C42	−93.5 (4)
C6—C1—C2—C3	−0.7 (5)	O9—C40—C41—C46	−144.5 (4)
O3—C1—C2—C19	0.4 (5)	C39—C40—C41—C46	89.2 (5)
C6—C1—C2—C19	179.3 (3)	C46—C41—C42—C43	0.1 (6)
C1—C2—C3—C4	−1.6 (6)	C40—C41—C42—C43	−177.2 (4)
C19—C2—C3—C4	178.5 (3)	C41—C42—C43—C44	0.1 (6)
C2—C3—C4—C5	1.9 (6)	C42—C43—C44—C45	−0.8 (7)
C2—C3—C4—C23	−178.8 (3)	C43—C44—C45—C46	1.4 (7)
C3—C4—C5—C6	0.0 (5)	C44—C45—C46—C41	−1.2 (7)
C23—C4—C5—C6	−179.3 (3)	C42—C41—C46—C45	0.4 (6)
O3—C1—C6—C5	−178.6 (3)	C40—C41—C46—C45	177.9 (4)
C2—C1—C6—C5	2.5 (6)	C33—C32—C49—C52	1.6 (5)
O3—C1—C6—C7	4.7 (5)	C31—C32—C49—C52	179.8 (4)
C2—C1—C6—C7	−174.2 (3)	C33—C32—C49—C51	−118.3 (4)
C4—C5—C6—C1	−2.2 (6)	C31—C32—C49—C51	59.9 (5)
C4—C5—C6—C7	174.5 (3)	C33—C32—C49—C50	120.3 (4)
C17—N8—C7—C6	−57.3 (4)	C31—C32—C49—C50	−61.5 (5)
C9—N8—C7—C6	−176.0 (3)	C35—C34—C53—C54	121.0 (4)
Mo1—N8—C7—C6	64.4 (3)	C33—C34—C53—C54	−59.1 (5)
C1—C6—C7—N8	−56.5 (5)	C35—C34—C53—C55	1.8 (6)
C5—C6—C7—N8	126.8 (4)	C33—C34—C53—C55	−178.3 (4)
C7—N8—C9—C18	−27.6 (4)	C35—C34—C53—C56	−118.4 (4)
Mo1—N8—C9—C18	94.2 (3)	C33—C34—C53—C56	61.5 (5)
C7—N8—C9—C10	−151.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*O*···O6^{i}	0.81 (1)	1.96 (3)	2.690 (4)	148 (6)
O10—H10*O*···O1	0.82 (1)	1.84 (2)	2.633 (4)	165 (5)

Symmetry code: (i) *x*, *y*−1, *z*.