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{1,1′-[(2,2-Dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]di-2-naphtholato}dioxidomolybdenum(VI) dichloromethane 1.75-solvate

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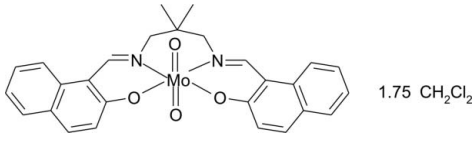
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.035; wR factor = 0.106; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $[Mo(C_{27}H_{24}N_2O_2)O_2]\cdot1.75CH_2Cl_2$, the Mo^{VI} ion is coordinated by two oxide O atoms and by two O and two N atoms of the tetradentate 1,1′-[(2,2-dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]di-2-naphtholate Schiff base ligand in a distorted octahedral configuration. The compound crystallizes with 1.75 molecules of dichloromethane per complex molecule. In the crystal, symmetry-related molecules are linked by a number of C—H···O interactions involving both the Schiff base ligand and the partly disordered dichloromethane solvent molecules, leading to the formation of a two-dimensional network extending parallel to (101).

Related literature

For the chemistry of molybdenum(VI)–Schiff base complexes and related structures with O═Mo═O units (metal oxidation state +VI), see: Abbasi *et al.* (2008); Arnaiz *et al.* (2000); Holm *et al.* (1996); Maurya *et al.* (1997); Nakayima *et al.* (1998); Rao *et al.* (1998); Sheikhshoaie *et al.* (2009); Syamal & Maurya (1989).



Experimental

Crystal data

$[Mo(C_{27}H_{24}N_2O_2)O_2]\cdot1.75CH_2Cl_2$
M_r = 685.04
Monoclinic, $C2/c$
a = 27.6049 (18) Å
b = 10.7743 (8) Å
c = 21.6474 (14) Å
β = 112.861 (7)°
V = 5932.7 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.79 mm^{-1}
T = 173 K
0.38 × 0.23 × 0.18 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: multi-scan (*MULscanABS* in *PLATON*; Spek, 2009)
T_{min} = 0.833, T_{max} = 0.864
21863 measured reflections
5537 independent reflections
4789 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.106
S = 1.12
5537 reflections
372 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—O1	2.0888 (19)	Mo1—O4	1.713 (2)
Mo1—O2	1.957 (2)	Mo1—N1	2.118 (2)
Mo1—O3	1.702 (2)	Mo1—N2	2.297 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3^{i}	0.95	2.43	3.328 (5)	159
C11—H11···O4^{ii}	0.95	2.38	3.297 (3)	161
C12—H12A···O4	0.99	2.47	2.974 (4)	111
C27—H27A···O3^{ii}	0.98	2.55	3.490 (4)	161
C28—H28A···O2^{iii}	0.99	2.40	3.213 (5)	139
C28—H28A···O4^{iii}	0.99	2.53	3.429 (6)	151

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

Data collection: *EXPOSE* in *IPDS-I* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS-I* (Stoe & Cie, 2000); data reduction: *INTEGRATE* in *IPDS-I* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2248).

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supplementary materials

Acta Cryst. (2009). E**65**, m1124-m1125 [**doi:10.1107/S1600536809032759**]

{1,1'-[(2,2-Dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]di-2-naphtholato}dioxidomolybdenum(VI) dichloromethane 1.75-solvate

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Comment

Numerous molybdenum(VI) Schiff base complexes have been extensively investigated for over the past twenty years, due to their importance in the domains of stereochemistry (Maurya *et al.*, 1997), structural chemistry (Nakayima, *et al.*, 1998; Syamal & Maurya, 1989), analytical chemistry (Rao *et al.*, 1998), bioinorganic chemistry (Holm *et al.*, 1996), and oxidation catalysis (Abbasi *et al.*, 2008; Arnaiz *et al.*, 2000; Sheikhshoaie *et al.*, 2009). Continuing our interest in the structural chemistry of dioxidomolybdenum(VI) Schiff base complexes, we have synthesized and structurally characterized the title compound.

The molecular structure of the title complex is illustrated in Fig. 1, and geometrical parameters are available in the archived CIF. The Mo^{VI} atom is in a distorted octahedral environment being coordinated by two oxido O atoms (O1 and O2) and four atoms (two oxygen and two nitrogen atoms) of the tetradentate Schiff base ligand 1,1'-[(2,2-dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]di-2-naphtholate. The Mo—O distances of the oxido ligands (Mo1O3 and Mo1O4) are significantly shorter [1.702 (2) and 1.713 (2) Å, respectively] than the corresponding Mo—O distances to the O-atoms (O2 and O1) of the tetradentate Schiff base ligand, 1.957 (2) and 2.0888 (19) Å, respectively. The Mo—N distances, to atoms N1 and N2, are even longer being 2.118 (2) and 2.297 (2) Å, respectively.

In the crystal, molecules are linked by a number of C—H···O interactions, involving both the Schiff base ligand and the solvent molecules of crystallization, leading to the formation of a two-dimensional network extending in the (101) plane (Table 1 and Fig. 2).

Experimental

The title dioxidomolybdenum (VI) complex was prepared by mixing $MoO_2(acac)_2$ with the ligand, 2,2'-[(2,2-dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]-dinaphtholate, in a 1:1 molar ratio using 25 ml of dry methanol as solvent, followed by refluxing the solution for 3 h. The small reddish crystals that formed were filtered off and recrystallized from dichloromethane.

Refinement

All H-atoms were placed in the calculated positions and treated as riding atoms: C—H = 0.95 - 0.99 Å with U_{iso}(H) = k × U_{eq}(parent C-atom), where k = 1.2 for aromatic H-atoms, and 1.5 for methyl H-atoms. The compound crystallizes with 1.75 molecules of dichloromethane per molecule of complex. In one of these molecules a chlorine atom (Cl1A/Cl1B) is positionally disordered with occupancies of 0.75/0.25. Using the one-circle Stoe Image Plate Diffraction System it is not possible to measure 100% of the Ewald sphere, and here only 95% was accessible.

Figures

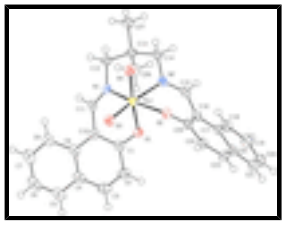


Fig. 1. A view of the molecular structure of the title complex, with the thermal ellipsoids drawn at the 50% probability level. The dichloromethane molecules of crystallization have been omitted for clarity.

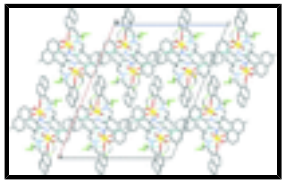


Fig. 2. A view along the *b* axis of the crystal packing of the title complex. The H-atoms not involved in C—H···O interactions (dotted blue lines) were omitted for clarity.

{1,1'-[(2,2-Dimethylpropane-1,3-diyl)bis(nitrilomethylidyne)]di-2- naphtholato}dioxidomolybdenum(VI) dichloromethane 1.75-solvate

Crystal data

$[Mo(C_{27}H_{24}N_2O_2)O_2]\cdot 1.75CH_2Cl_2$	F_{000} = 2780
M_r = 685.04	D_x = 1.534 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 7998 reflections
a = 27.6049 (18) Å	θ = 2.0–26.1º
b = 10.7743 (8) Å	μ = 0.79 mm^{-1}
c = 21.6474 (14) Å	*T* = 173 K
β = 112.861 (7)º	Block, red
V = 5932.7 (7) Å^3	0.38 × 0.23 × 0.18 mm
Z = 8	

Data collection

Stoe IPDS diffractometer	5537 independent reflections
Radiation source: fine-focus sealed tube	4789 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 173 K	θ_{max} = 26.0º
φ rotation scans	θ_{min} = 2.2º
Absorption correction: multi-scan (MULscanABS in PLATON; Spek, 2009)	*h* = −33→33
T_{min} = 0.833, T_{max} = 0.864	*k* = −13→13
21863 measured reflections	*l* = −26→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 13.6226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} = 0.001$
5537 reflections	$\Delta\rho_{max} = 1.08$ e Å^{-3}
372 parameters	$\Delta\rho_{min} = -0.83$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.15195 (1)	0.99201 (2)	0.16605 (1)	0.0203 (1)	
O1	0.11880 (7)	0.83854 (17)	0.10537 (10)	0.0244 (5)	
O2	0.07965 (7)	1.05359 (17)	0.13420 (10)	0.0243 (5)	
O3	0.17351 (8)	1.04666 (19)	0.10747 (10)	0.0294 (6)	
O4	0.18137 (8)	1.08694 (18)	0.23387 (10)	0.0292 (6)	
N1	0.21210 (9)	0.8581 (2)	0.20744 (11)	0.0230 (7)	
N2	0.11991 (9)	0.8840 (2)	0.23353 (11)	0.0226 (6)	
C1	0.14254 (11)	0.7716 (2)	0.07556 (14)	0.0240 (8)	
C2	0.11155 (12)	0.7170 (3)	0.01261 (15)	0.0320 (9)	
C3	0.13435 (13)	0.6494 (3)	−0.02156 (16)	0.0351 (9)	
C4	0.18908 (13)	0.6260 (3)	0.00395 (15)	0.0297 (9)	
C5	0.21229 (15)	0.5592 (3)	−0.03334 (16)	0.0359 (10)	
C6	0.26506 (15)	0.5380 (3)	−0.00885 (17)	0.0378 (10)	
C7	0.29652 (14)	0.5838 (3)	0.05388 (18)	0.0373 (10)	
C8	0.27554 (13)	0.6506 (3)	0.09176 (16)	0.0312 (9)	
C9	0.22085 (12)	0.6729 (2)	0.06788 (14)	0.0258 (8)	
C10	0.19693 (11)	0.7471 (2)	0.10402 (14)	0.0240 (8)	
C11	0.22523 (11)	0.7765 (2)	0.17248 (14)	0.0236 (8)	
C12	0.23835 (11)	0.8489 (3)	0.28089 (14)	0.0259 (8)	
C13	0.20409 (11)	0.7852 (3)	0.31281 (14)	0.0264 (8)	
C14	0.15487 (11)	0.8626 (3)	0.30383 (14)	0.0265 (8)	
C15	0.07193 (11)	0.8512 (2)	0.21471 (14)	0.0243 (8)	
C16	0.03019 (11)	0.8852 (3)	0.15189 (14)	0.0237 (8)	
C17	−0.01851 (11)	0.8180 (3)	0.12751 (14)	0.0258 (8)	
C18	−0.02482 (13)	0.7013 (3)	0.15323 (16)	0.0317 (9)	
C19	−0.07116 (14)	0.6372 (3)	0.12614 (18)	0.0396 (11)	
C20	−0.11356 (13)	0.6855 (3)	0.07190 (19)	0.0412 (11)	
C21	−0.10943 (12)	0.7981 (3)	0.04577 (16)	0.0347 (9)	

C22	−0.06213 (11)	0.8663 (3)	0.07250 (15)	0.0277 (8)	
C23	−0.05656 (12)	0.9810 (3)	0.04395 (16)	0.0311 (9)	
C24	−0.00985 (11)	1.0411 (3)	0.06555 (15)	0.0276 (8)	
C25	0.03480 (11)	0.9914 (2)	0.11817 (15)	0.0231 (8)	
C26	0.18840 (13)	0.6549 (3)	0.28459 (17)	0.0347 (9)	
C27	0.23717 (13)	0.7789 (3)	0.38843 (15)	0.0342 (9)	
Cl1A	0.11134 (12)	0.36651 (18)	0.28031 (10)	0.1016 (8)	0.800
Cl2	0.06716 (6)	0.14580 (16)	0.31642 (8)	0.0913 (6)	
C28	0.0768 (2)	0.2286 (5)	0.2529 (2)	0.0657 (17)	
Cl1B	0.0605 (5)	0.3839 (7)	0.2649 (4)	0.102 (4)	0.200
Cl3	0.02059 (6)	0.4230 (2)	0.09019 (10)	0.0919 (8)	0.750
Cl4	0.13068 (6)	0.4167 (3)	0.11352 (11)	0.0993 (8)	0.750
C29	0.0693 (3)	0.3744 (8)	0.0643 (4)	0.088 (3)	0.750
H2	0.07450	0.72800	−0.00560	0.0380*	
H3	0.11280	0.61660	−0.06420	0.0420*	
H5	0.19070	0.52830	−0.07640	0.0430*	
H6	0.28020	0.49240	−0.03440	0.0450*	
H7	0.33330	0.56880	0.07100	0.0450*	
H8	0.29800	0.68190	0.13430	0.0370*	
H11	0.25700	0.73220	0.19510	0.0280*	
H12A	0.24750	0.93330	0.29990	0.0310*	
H12B	0.27150	0.80150	0.29240	0.0310*	
H14A	0.13480	0.81950	0.32670	0.0320*	
H14B	0.16620	0.94380	0.32610	0.0320*	
H15	0.06280	0.80030	0.24440	0.0290*	
H18	0.00360	0.66670	0.19000	0.0380*	
H19	−0.07450	0.55900	0.14440	0.0480*	
H20	−0.14540	0.63980	0.05320	0.0490*	
H21	−0.13860	0.83090	0.00930	0.0420*	
H23	−0.08610	1.01640	0.00910	0.0370*	
H24	−0.00690	1.11740	0.04530	0.0330*	
H26A	0.22010	0.60530	0.29290	0.0520*	
H26B	0.16780	0.65990	0.23620	0.0520*	
H26C	0.16720	0.61560	0.30650	0.0520*	
H27A	0.26850	0.72800	0.39650	0.0510*	
H27B	0.24790	0.86290	0.40570	0.0510*	
H27C	0.21620	0.74190	0.41130	0.0510*	
H28A	0.09620	0.17560	0.23290	0.0790*	0.800
H28B	0.04210	0.24780	0.21730	0.0790*	0.800
H28C	0.11240	0.21750	0.25530	0.0790*	0.200
H28D	0.05230	0.20120	0.20920	0.0790*	0.200
H29A	0.06810	0.28270	0.06100	0.1060*	0.750
H29B	0.06140	0.40740	0.01870	0.1060*	0.750

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0189 (1)	0.0204 (1)	0.0216 (1)	−0.0010 (1)	0.0080 (1)	−0.0015 (1)

O1	0.0227 (9)	0.0256 (9)	0.0249 (10)	−0.0011 (7)	0.0092 (8)	−0.0046 (7)
O2	0.0215 (9)	0.0231 (9)	0.0287 (10)	0.0001 (7)	0.0103 (8)	0.0010 (8)
O3	0.0272 (10)	0.0320 (11)	0.0308 (11)	−0.0018 (8)	0.0133 (9)	0.0012 (8)
O4	0.0278 (10)	0.0257 (10)	0.0315 (11)	−0.0013 (8)	0.0086 (9)	−0.0053 (8)
N1	0.0193 (11)	0.0271 (12)	0.0220 (11)	0.0005 (9)	0.0074 (9)	−0.0015 (9)
N2	0.0246 (11)	0.0235 (11)	0.0205 (11)	0.0036 (9)	0.0097 (10)	0.0005 (9)
C1	0.0292 (14)	0.0198 (12)	0.0249 (13)	−0.0016 (10)	0.0127 (12)	0.0000 (10)
C2	0.0312 (15)	0.0290 (15)	0.0301 (15)	0.0010 (12)	0.0058 (13)	−0.0057 (12)
C3	0.0465 (18)	0.0275 (15)	0.0261 (15)	−0.0007 (13)	0.0086 (14)	−0.0046 (11)
C4	0.0446 (17)	0.0215 (13)	0.0280 (14)	−0.0004 (12)	0.0196 (14)	0.0005 (11)
C5	0.060 (2)	0.0229 (14)	0.0318 (16)	−0.0016 (13)	0.0254 (16)	−0.0004 (12)
C6	0.062 (2)	0.0243 (14)	0.0428 (18)	0.0024 (14)	0.0375 (18)	0.0004 (13)
C7	0.0467 (19)	0.0271 (15)	0.0501 (19)	0.0051 (13)	0.0318 (17)	0.0059 (13)
C8	0.0372 (16)	0.0276 (14)	0.0352 (16)	0.0024 (12)	0.0212 (14)	0.0019 (12)
C9	0.0358 (15)	0.0181 (12)	0.0290 (14)	0.0015 (11)	0.0186 (13)	0.0029 (10)
C10	0.0277 (14)	0.0205 (12)	0.0243 (13)	−0.0003 (10)	0.0107 (12)	0.0001 (10)
C11	0.0215 (13)	0.0247 (13)	0.0278 (14)	0.0021 (10)	0.0132 (12)	0.0015 (10)
C12	0.0224 (13)	0.0310 (14)	0.0203 (13)	0.0046 (11)	0.0038 (11)	−0.0023 (11)
C13	0.0263 (14)	0.0291 (14)	0.0221 (13)	0.0048 (11)	0.0077 (12)	−0.0005 (11)
C14	0.0271 (14)	0.0318 (14)	0.0206 (13)	0.0033 (11)	0.0094 (12)	0.0012 (11)
C15	0.0267 (14)	0.0241 (13)	0.0257 (14)	0.0008 (10)	0.0141 (12)	0.0019 (10)
C16	0.0222 (13)	0.0257 (13)	0.0243 (13)	0.0024 (10)	0.0104 (11)	−0.0016 (10)
C17	0.0267 (14)	0.0285 (14)	0.0252 (14)	−0.0004 (11)	0.0133 (12)	−0.0051 (11)
C18	0.0350 (16)	0.0318 (15)	0.0327 (16)	−0.0036 (12)	0.0180 (14)	−0.0024 (12)
C19	0.0454 (19)	0.0353 (17)	0.0453 (19)	−0.0133 (14)	0.0254 (17)	−0.0073 (14)
C20	0.0330 (17)	0.0463 (19)	0.0472 (19)	−0.0173 (14)	0.0188 (16)	−0.0143 (15)
C21	0.0243 (14)	0.0477 (18)	0.0331 (16)	−0.0042 (13)	0.0121 (13)	−0.0106 (14)
C22	0.0216 (13)	0.0359 (15)	0.0274 (14)	−0.0016 (11)	0.0116 (12)	−0.0072 (12)
C23	0.0238 (14)	0.0383 (16)	0.0279 (15)	0.0042 (12)	0.0065 (13)	−0.0009 (12)
C24	0.0261 (14)	0.0284 (14)	0.0283 (14)	0.0043 (11)	0.0106 (12)	0.0036 (11)
C25	0.0209 (13)	0.0230 (13)	0.0263 (14)	0.0011 (10)	0.0101 (12)	−0.0035 (10)
C26	0.0378 (17)	0.0294 (15)	0.0369 (17)	0.0023 (13)	0.0145 (14)	0.0008 (12)
C27	0.0332 (16)	0.0433 (18)	0.0248 (15)	0.0124 (13)	0.0097 (13)	0.0043 (12)
Cl1A	0.150 (2)	0.0683 (11)	0.0748 (11)	−0.0216 (12)	0.0309 (13)	−0.0188 (9)
Cl2	0.0686 (8)	0.1124 (12)	0.0813 (9)	0.0082 (8)	0.0163 (7)	0.0270 (8)
C28	0.067 (3)	0.073 (3)	0.064 (3)	−0.009 (2)	0.033 (2)	−0.027 (2)
Cl1B	0.166 (9)	0.060 (4)	0.073 (4)	0.011 (5)	0.040 (5)	−0.022 (3)
Cl3	0.0444 (8)	0.150 (2)	0.0799 (12)	0.0113 (10)	0.0225 (8)	−0.0222 (12)
Cl4	0.0402 (8)	0.157 (2)	0.0875 (13)	0.0037 (10)	0.0105 (8)	0.0133 (13)
C29	0.057 (4)	0.114 (6)	0.100 (6)	−0.009 (4)	0.038 (4)	−0.048 (5)

Geometric parameters (Å, °)

Mo1—O1	2.0888 (19)	C18—C19	1.369 (5)
Mo1—O2	1.957 (2)	C19—C20	1.397 (5)
Mo1—O3	1.702 (2)	C20—C21	1.362 (5)
Mo1—O4	1.713 (2)	C21—C22	1.412 (5)
Mo1—N1	2.118 (2)	C22—C23	1.417 (5)
Mo1—N2	2.297 (2)	C23—C24	1.354 (5)

Cl1A—C28	1.741 (6)	C24—C25	1.419 (4)
Cl1B—C28	1.778 (10)	C2—H2	0.9500
Cl2—C28	1.744 (5)	C3—H3	0.9500
Cl3—C29	1.727 (9)	C5—H5	0.9500
Cl4—C29	1.677 (9)	C6—H6	0.9500
O1—C1	1.302 (3)	C7—H7	0.9500
O2—C25	1.330 (4)	C8—H8	0.9500
N1—C12	1.472 (4)	C11—H11	0.9500
N1—C11	1.300 (4)	C12—H12A	0.9900
N2—C14	1.470 (4)	C12—H12B	0.9900
N2—C15	1.275 (4)	C14—H14B	0.9900
C1—C10	1.409 (4)	C14—H14A	0.9900
C1—C2	1.425 (4)	C15—H15	0.9500
C2—C3	1.355 (5)	C18—H18	0.9500
C3—C4	1.415 (5)	C19—H19	0.9500
C4—C5	1.408 (5)	C20—H20	0.9500
C4—C9	1.413 (4)	C21—H21	0.9500
C5—C6	1.362 (6)	C23—H23	0.9500
C6—C7	1.388 (5)	C24—H24	0.9500
C7—C8	1.377 (5)	C26—H26C	0.9800
C8—C9	1.413 (5)	C26—H26B	0.9800
C9—C10	1.445 (4)	C26—H26A	0.9800
C10—C11	1.417 (4)	C27—H27A	0.9800
C12—C13	1.533 (4)	C27—H27B	0.9800
C13—C26	1.525 (5)	C27—H27C	0.9800
C13—C27	1.535 (4)	C28—H28A	0.9900
C13—C14	1.541 (5)	C28—H28D	0.9700
C15—C16	1.447 (4)	C28—H28B	0.9900
C16—C25	1.389 (4)	C28—H28C	0.9700
C16—C17	1.435 (5)	C29—H29A	0.9900
C17—C18	1.413 (5)	C29—H29B	0.9900
C17—C22	1.423 (4)		
O1—Mo1—O2	84.79 (8)	C1—C2—H2	120.00
O1—Mo1—O3	90.00 (9)	C3—C2—H2	120.00
O1—Mo1—O4	163.25 (9)	C2—C3—H3	119.00
O1—Mo1—N1	78.85 (8)	C4—C3—H3	119.00
O1—Mo1—N2	79.39 (8)	C4—C5—H5	119.00
O2—Mo1—O3	102.96 (10)	C6—C5—H5	119.00
O2—Mo1—O4	100.28 (10)	C5—C6—H6	120.00
O2—Mo1—N1	154.77 (9)	C7—C6—H6	120.00
O2—Mo1—N2	78.68 (9)	C6—C7—H7	119.00
O3—Mo1—O4	104.21 (10)	C8—C7—H7	119.00
O3—Mo1—N1	96.16 (10)	C7—C8—H8	120.00
O3—Mo1—N2	169.12 (9)	C9—C8—H8	120.00
O4—Mo1—N1	90.65 (9)	N1—C11—H11	117.00
O4—Mo1—N2	85.92 (9)	C10—C11—H11	117.00
N1—Mo1—N2	79.52 (9)	N1—C12—H12A	109.00
Mo1—O1—C1	124.64 (18)	N1—C12—H12B	109.00
Mo1—O2—C25	129.78 (16)	C13—C12—H12A	109.00

Mo1—N1—C11	124.36 (19)	C13—C12—H12B	109.00
Mo1—N1—C12	118.61 (18)	H12A—C12—H12B	108.00
C11—N1—C12	116.8 (2)	N2—C14—H14A	109.00
Mo1—N2—C14	118.55 (19)	N2—C14—H14B	109.00
Mo1—N2—C15	123.10 (18)	C13—C14—H14A	109.00
C14—N2—C15	118.0 (2)	C13—C14—H14B	109.00
O1—C1—C2	118.2 (3)	H14A—C14—H14B	108.00
O1—C1—C10	122.9 (2)	N2—C15—H15	117.00
C2—C1—C10	118.9 (3)	C16—C15—H15	117.00
C1—C2—C3	120.7 (3)	C17—C18—H18	119.00
C2—C3—C4	122.3 (3)	C19—C18—H18	119.00
C3—C4—C5	121.5 (3)	C18—C19—H19	120.00
C3—C4—C9	118.7 (3)	C20—C19—H19	120.00
C5—C4—C9	119.8 (3)	C19—C20—H20	120.00
C4—C5—C6	121.3 (3)	C21—C20—H20	120.00
C5—C6—C7	119.3 (3)	C20—C21—H21	120.00
C6—C7—C8	121.4 (4)	C22—C21—H21	120.00
C7—C8—C9	120.5 (3)	C22—C23—H23	119.00
C4—C9—C8	117.8 (3)	C24—C23—H23	119.00
C4—C9—C10	119.2 (3)	C23—C24—H24	120.00
C8—C9—C10	122.9 (3)	C25—C24—H24	120.00
C1—C10—C9	120.0 (3)	C13—C26—H26A	110.00
C1—C10—C11	118.5 (3)	C13—C26—H26B	110.00
C9—C10—C11	120.5 (3)	C13—C26—H26C	109.00
N1—C11—C10	126.5 (3)	H26A—C26—H26B	109.00
N1—C12—C13	112.7 (2)	H26A—C26—H26C	109.00
C12—C13—C14	111.7 (3)	H26B—C26—H26C	109.00
C12—C13—C26	111.1 (3)	C13—C27—H27A	109.00
C12—C13—C27	106.6 (3)	C13—C27—H27B	109.00
C14—C13—C26	110.4 (3)	C13—C27—H27C	109.00
C14—C13—C27	106.8 (2)	H27A—C27—H27B	109.00
C26—C13—C27	110.2 (3)	H27A—C27—H27C	110.00
N2—C14—C13	114.1 (2)	H27B—C27—H27C	109.00
N2—C15—C16	125.4 (3)	Cl1A—C28—Cl2	113.5 (2)
C15—C16—C17	120.5 (3)	Cl1B—C28—Cl2	104.0 (4)
C15—C16—C25	120.0 (3)	Cl1A—C28—H28A	109.00
C17—C16—C25	119.1 (3)	Cl1A—C28—H28B	109.00
C16—C17—C18	123.1 (3)	Cl2—C28—H28A	109.00
C16—C17—C22	119.4 (3)	Cl2—C28—H28B	109.00
C18—C17—C22	117.5 (3)	Cl2—C28—H28C	111.00
C17—C18—C19	121.2 (3)	Cl2—C28—H28D	111.00
C18—C19—C20	120.7 (3)	Cl1B—C28—H28C	115.00
C19—C20—C21	120.2 (3)	Cl1B—C28—H28D	108.00
C20—C21—C22	120.5 (3)	H28A—C28—H28B	108.00
C17—C22—C21	120.0 (3)	H28C—C28—H28D	109.00
C17—C22—C23	119.0 (3)	Cl3—C29—Cl4	115.7 (5)
C21—C22—C23	121.0 (3)	Cl3—C29—H29A	109.00
C22—C23—C24	121.1 (3)	Cl3—C29—H29B	108.00
C23—C24—C25	120.7 (3)	Cl4—C29—H29A	108.00

O2—C25—C16	123.0 (3)	Cl4—C29—H29B	108.00
O2—C25—C24	116.7 (2)	H29A—C29—H29B	107.00
C16—C25—C24	120.3 (3)		
O2—Mo1—O1—C1	149.5 (2)	C9—C4—C5—C6	0.4 (5)
O3—Mo1—O1—C1	46.5 (2)	C3—C4—C9—C8	−178.7 (3)
N1—Mo1—O1—C1	−49.8 (2)	C3—C4—C9—C10	−2.4 (4)
N2—Mo1—O1—C1	−131.1 (2)	C5—C4—C9—C8	0.3 (4)
O1—Mo1—O2—C25	33.1 (2)	C5—C4—C9—C10	176.6 (3)
O3—Mo1—O2—C25	121.9 (2)	C4—C5—C6—C7	−0.4 (5)
O4—Mo1—O2—C25	−130.7 (2)	C5—C6—C7—C8	−0.3 (5)
N1—Mo1—O2—C25	−16.4 (4)	C6—C7—C8—C9	0.9 (5)
N2—Mo1—O2—C25	−47.1 (2)	C7—C8—C9—C4	−0.9 (4)
O1—Mo1—N1—C11	37.9 (2)	C7—C8—C9—C10	−177.1 (3)
O1—Mo1—N1—C12	−137.0 (2)	C4—C9—C10—C1	0.2 (4)
O2—Mo1—N1—C11	88.4 (3)	C4—C9—C10—C11	168.5 (3)
O2—Mo1—N1—C12	−86.5 (3)	C8—C9—C10—C1	176.3 (3)
O3—Mo1—N1—C11	−50.9 (2)	C8—C9—C10—C11	−15.4 (4)
O3—Mo1—N1—C12	134.2 (2)	C1—C10—C11—N1	−23.9 (4)
O4—Mo1—N1—C11	−155.3 (2)	C9—C10—C11—N1	167.7 (3)
O4—Mo1—N1—C12	29.9 (2)	N1—C12—C13—C14	−65.6 (3)
N2—Mo1—N1—C11	119.0 (2)	N1—C12—C13—C26	58.2 (3)
N2—Mo1—N1—C12	−55.9 (2)	N1—C12—C13—C27	178.2 (2)
O1—Mo1—N2—C14	131.0 (2)	C12—C13—C14—N2	60.7 (3)
O1—Mo1—N2—C15	−55.5 (2)	C26—C13—C14—N2	−63.5 (3)
O2—Mo1—N2—C14	−142.3 (2)	C27—C13—C14—N2	176.8 (3)
O2—Mo1—N2—C15	31.3 (2)	N2—C15—C16—C17	163.0 (3)
O4—Mo1—N2—C14	−40.9 (2)	N2—C15—C16—C25	−23.8 (4)
O4—Mo1—N2—C15	132.6 (2)	C15—C16—C17—C18	−14.9 (5)
N1—Mo1—N2—C14	50.5 (2)	C15—C16—C17—C22	168.7 (3)
N1—Mo1—N2—C15	−136.0 (2)	C25—C16—C17—C18	171.9 (3)
Mo1—O1—C1—C2	−147.8 (2)	C25—C16—C17—C22	−4.6 (4)
Mo1—O1—C1—C10	34.8 (3)	C15—C16—C25—O2	12.1 (4)
Mo1—O2—C25—C16	35.8 (4)	C15—C16—C25—C24	−165.6 (3)
Mo1—O2—C25—C24	−146.5 (2)	C17—C16—C25—O2	−174.6 (3)
Mo1—N1—C11—C10	−11.9 (4)	C17—C16—C25—C24	7.7 (4)
C12—N1—C11—C10	163.0 (3)	C16—C17—C18—C19	−176.5 (3)
Mo1—N1—C12—C13	75.1 (3)	C22—C17—C18—C19	0.1 (5)
C11—N1—C12—C13	−100.2 (3)	C16—C17—C22—C21	176.7 (3)
Mo1—N2—C14—C13	−61.8 (3)	C16—C17—C22—C23	−1.1 (5)
C15—N2—C14—C13	124.3 (3)	C18—C17—C22—C21	0.1 (5)
Mo1—N2—C15—C16	−5.7 (4)	C18—C17—C22—C23	−177.8 (3)
C14—N2—C15—C16	167.9 (3)	C17—C18—C19—C20	0.3 (6)
O1—C1—C2—C3	178.1 (3)	C18—C19—C20—C21	−0.8 (6)
C10—C1—C2—C3	−4.4 (4)	C19—C20—C21—C22	0.9 (5)
O1—C1—C10—C9	−179.4 (2)	C20—C21—C22—C17	−0.6 (5)
O1—C1—C10—C11	12.0 (4)	C20—C21—C22—C23	177.3 (3)
C2—C1—C10—C9	3.1 (4)	C17—C22—C23—C24	3.9 (5)
C2—C1—C10—C11	−165.4 (2)	C21—C22—C23—C24	−174.0 (3)
C1—C2—C3—C4	2.2 (5)	C22—C23—C24—C25	−0.8 (5)

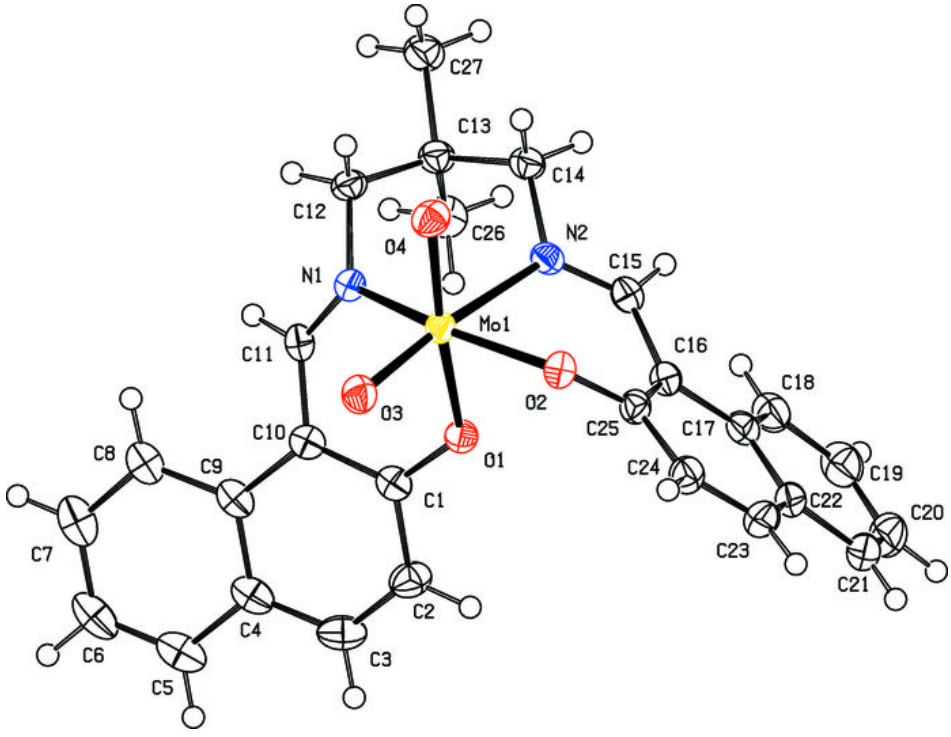
C2—C3—C4—C5	−177.8 (3)	C23—C24—C25—O2	177.1 (3)
C2—C3—C4—C9	1.2 (5)	C23—C24—C25—C16	−5.1 (5)
C3—C4—C5—C6	179.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O3^{i}	0.95	2.43	3.328 (5)	159
C11—H11···O4^{ii}	0.95	2.38	3.297 (3)	161
C12—H12A···O4	0.99	2.47	2.974 (4)	111
C27—H27A···O3^{ii}	0.98	2.55	3.490 (4)	161
C28—H28A···O2^{iii}	0.99	2.40	3.213 (5)	139
C28—H28A···O4^{iii}	0.99	2.53	3.429 (6)	151

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

Fig. 1


C27
C13
C14
C12
C26
O4
N2
N1
C15
Mo1
C11
C16
C18
O3
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C25
C17
C10
C8
C9
O1
C24
C19
C1
C22
C20
C7
C23
C2
C21
C4
C6
C3
C5

Fig. 2