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Dioxidobis{2-[(*E*)-*p*-tolyliminomethyl]-phenolato}molybdenum(VI)

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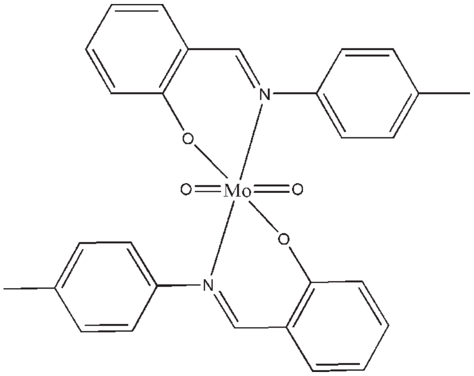
Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.019 Å; *R* factor = 0.079; *wR* factor = 0.168; data-to-parameter ratio = 16.2.

The asymmetric unit of the title compound, [Mo($C_{14}H_{12}NO)_2O_2$], comprises half of the complex with the full molecule generated by the application of twofold symmetry. The Mo^{VI} atom is surrounded by two oxide O atoms and the two sets of *N*,*O*-donor atoms of the bidentate Schiff base ligands. The resulting N_2O_4 donor set defines a distorted octahedral coordination geometry. Intermolecular C—H···O contacts link molecules into chains along the *b* axis. The crystal structure is further stabilized by intermolecular π–π interactions [ring centroid–centroid distance = 3.724 (6) Å].

Related literature

For related structures with MoO_2 units and for the synthesis, see: Arnaiz *et al.* (2000); Holm *et al.* (1996); Syamal & Maurya (1989).



Experimental

Crystal data

[Mo($C_{14}H_{12}NO)_2O_2$]	V = 2505.4 (18) Å^3
M_r = 548.43	Z = 4
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 26.375 (8) Å	μ = 0.56 mm^{-1}
b = 6.8095 (8) Å	T = 296 K
c = 15.648 (10) Å	0.21 × 0.11 × 0.08 mm
β = 116.94 (2)°	

Data collection

Stoe IPDS II diffractometer	4305 measured reflections
Absorption correction: multi-scan [*MULABS* in *PLATON* (Spek, 2009; Blessing, 1995)]	2006 independent reflections
T_{min} = 0.892, T_{max} = 0.957	761 reflections with $I > 2\sigma(I)$
	R_{int} = 0.144

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079	124 parameters
$wR(F^2)$ = 0.168	H-atom parameters constrained
S = 0.83	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
2006 reflections	$\Delta\rho_{min}$ = −0.81 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*A*···O2^{i}	0.93	2.34	3.237 (18)	163
C13—H13*A*···O2^{ii}	0.93	2.42	3.164 (17)	136

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

This work was supported by grants from the University of Kerman and the University of Tehran. RK thanks the Islamic Azad University for the X-ray diffractometer facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2696).

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supplementary materials

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Comment

Numerous chemical reactions are catalyzed by complexes containing the dioxomolybdenum(VI) unit, MoO_2 (Arnaiz *et al.* 2000). Moreover, Schiff base compounds containing molybdenum play a significant role in the chemistry of molybdoenzymes (Holm *et al.* 1996; Syamal & Maurya, 1989).

The asymmetric unit of the title compound, Fig. 1, comprises half of the complex. The Mo atom is located on a crystallographic 2-fold axis. The Mo^{VI} atom is surrounded by two oxo-O atoms and the N_2O_2 donor atoms of two bidentate Schiff base ligands to define a distorted octahedral coordination geometry. Intermolecular C—H···O contacts link molecules into chains along the *b* axis, Table 1 and Fig. 2. The crystal structure is further stabilized by intermolecular π–π interactions with the ring centroid(C1– C6) to centroid$(C8–C13)^i$ distance being 3.724 (6) Å for *i*: -*x*, *y*, 1/2-*z*.

Experimental

The title complex was prepared by adding $MoO_2(acac)_2$ and the ligand (molar ratio 1:1) to dry methanol (30 ml), followed by refluxing for 1 h. Small, light-yellow crystals were filtered off and recrystallized from acetonitrile. The quality of the crystal was not optimal and it was weakly diffracting. Although recrystallization was attempted repeatedly, better crystals were not obtained.

Refinement

All H atoms were positioned geometrically (C–H = 0.93–0.96 Å) and constrained to refine with the parent atoms with U_{iso} (H) = 1.2–1.5 U_{eq} (C).

Figures

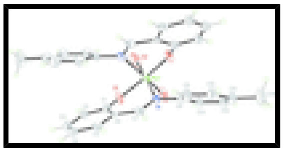

Fig. 1. The molecular structure of the title complex showing the atom-labelling scheme and displacement ellipsoids at the 35% probability level. Symmetry code for the unlabeled atoms: -*x*, *y*, 1/2 - *z*.

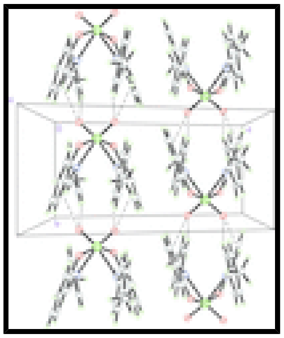

Fig. 2. A view of the crystal packing of the title compound down the *a* axis showing connections of molecules through C—H···O interactions (shown as dashed lines) along the *b* axis.

Dioxidobis{2-[(*E*)-*p*-tolyliminomethyl]phenolato}molybdenum(VI)

Crystal data

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$M_r = 548.43$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 26.375$ (8) Å

$b = 6.8095$ (8) Å

$c = 15.648$ (10) Å

$\beta = 116.94$ (2)°

$V = 2505.4$ (18) Å^3

$Z = 4$

$F(000) = 1120$

$D_x = 1.454$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2560 reflections

$\theta = 1.7$–29.6°

$\mu = 0.56$ mm^{-1}

$T = 296$ K

Block, colourless

0.21 × 0.11 × 0.08 mm

Data collection

Stoe IPDS II diffractometer

Radiation source: fine-focus sealed tube

graphite

Detector resolution: 0.15 mm pixels mm^{-1}

φ and ω scans

Absorption correction: multi-scan (*MULABS* in *PLATON*; Blessing, 1995)

$T_{min} = 0.892$, $T_{max} = 0.957$

4305 measured reflections

2006 independent reflections

761 reflections with $I > 2\sigma(I)$

$R_{int} = 0.144$

$\theta_{max} = 25.5$°, $\theta_{min} = 2.6$°

$h = -31 \rightarrow 22$

$k = -7 \rightarrow 8$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.079$

$wR(F^2) = 0.168$

$S = 0.83$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
2006 reflections	$(\Delta/\sigma)_{max} < 0.001$
124 parameters	$\Delta\rho_{max} = 0.55$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.81$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.0000	−0.2892 (2)	0.2500	0.0502 (7)
O1	−0.0423 (3)	−0.2223 (11)	0.3209 (4)	0.053 (2)
O2	−0.0486 (4)	−0.4409 (11)	0.1680 (6)	0.079 (3)
N1	0.0559 (5)	−0.0212 (13)	0.3428 (5)	0.037 (3)
C1	−0.0533 (6)	−0.061 (2)	0.3573 (8)	0.0479 (18)
C2	−0.1029 (5)	−0.0625 (17)	0.3693 (7)	0.0479 (18)
H2A	−0.1260	−0.1733	0.3541	0.057*
C3	−0.1165 (5)	0.1058 (16)	0.4047 (7)	0.0479 (18)
H3A	−0.1493	0.1052	0.4127	0.057*
C4	−0.0846 (5)	0.2697 (19)	0.4278 (6)	0.0479 (18)
H4A	−0.0957	0.3798	0.4503	0.057*
C5	−0.0345 (5)	0.2726 (19)	0.4176 (6)	0.054 (4)
H5A	−0.0118	0.3845	0.4333	0.065*
C6	−0.0187 (5)	0.1007 (18)	0.3827 (6)	0.037 (3)
C7	0.0360 (5)	0.1081 (16)	0.3804 (6)	0.041 (3)
H7A	0.0589	0.2167	0.4087	0.049*
C8	0.1129 (6)	0.013 (2)	0.3551 (7)	0.044 (3)
C9	0.1308 (6)	0.198 (2)	0.3415 (7)	0.063 (2)
H9A	0.1063	0.3049	0.3253	0.075*
C10	0.1856 (6)	0.219 (2)	0.3524 (7)	0.067 (4)
H10A	0.1972	0.3429	0.3429	0.081*
C11	0.2243 (6)	0.063 (2)	0.3771 (8)	0.063 (2)
C12	0.2048 (6)	−0.115 (2)	0.3891 (7)	0.063 (2)
H12A	0.2294	−0.2217	0.4054	0.075*
C13	0.1500 (5)	−0.144 (2)	0.3781 (7)	0.063 (2)
H13A	0.1384	−0.2690	0.3861	0.075*
C14	0.2838 (6)	0.095 (3)	0.3862 (11)	0.120 (6)
H14A	0.3085	−0.0083	0.4239	0.181*

H14B	0.2984	0.2193	0.4169	0.181*
H14C	0.2821	0.0955	0.3236	0.181*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.0830 (15)	0.0203 (8)	0.0809 (12)	0.000	0.0666 (11)	0.000
O1	0.056 (6)	0.046 (5)	0.080 (5)	0.015 (5)	0.050 (5)	0.018 (5)
O2	0.110 (10)	0.039 (5)	0.138 (8)	−0.036 (6)	0.099 (7)	−0.043 (5)
N1	0.045 (9)	0.033 (6)	0.037 (6)	0.027 (6)	0.022 (6)	0.014 (4)
C1	0.043 (5)	0.055 (4)	0.049 (4)	0.005 (4)	0.024 (4)	0.007 (3)
C2	0.043 (5)	0.055 (4)	0.049 (4)	0.005 (4)	0.024 (4)	0.007 (3)
C3	0.043 (5)	0.055 (4)	0.049 (4)	0.005 (4)	0.024 (4)	0.007 (3)
C4	0.043 (5)	0.055 (4)	0.049 (4)	0.005 (4)	0.024 (4)	0.007 (3)
C5	0.064 (10)	0.060 (9)	0.034 (6)	0.006 (9)	0.018 (6)	−0.006 (6)
C6	0.031 (9)	0.064 (9)	0.017 (6)	0.010 (7)	0.012 (6)	0.002 (6)
C7	0.053 (11)	0.037 (7)	0.029 (6)	0.010 (7)	0.015 (7)	0.018 (5)
C8	0.029 (9)	0.075 (10)	0.034 (7)	0.010 (8)	0.020 (6)	0.004 (6)
C9	0.053 (6)	0.079 (5)	0.062 (4)	0.031 (6)	0.031 (4)	0.015 (4)
C10	0.072 (11)	0.063 (9)	0.073 (8)	−0.010 (10)	0.038 (8)	−0.020 (8)
C11	0.053 (6)	0.079 (5)	0.062 (4)	0.031 (6)	0.031 (4)	0.015 (4)
C12	0.053 (6)	0.079 (5)	0.062 (4)	0.031 (6)	0.031 (4)	0.015 (4)
C13	0.053 (6)	0.079 (5)	0.062 (4)	0.031 (6)	0.031 (4)	0.015 (4)
C14	0.070 (14)	0.165 (18)	0.150 (14)	−0.014 (12)	0.071 (11)	−0.031 (12)

Geometric parameters (Å, °)

Mo1—O2	1.694 (9)	C5—H5A	0.9300
Mo1—O2^{i}	1.694 (9)	C6—C7	1.459 (14)
Mo1—O1^{i}	1.950 (6)	C7—H7A	0.9300
Mo1—O1	1.950 (6)	C8—C13	1.385 (15)
Mo1—N1	2.382 (9)	C8—C9	1.394 (17)
Mo1—N1^{i}	2.382 (9)	C9—C10	1.384 (15)
O1—C1	1.330 (12)	C9—H9A	0.9300
N1—C7	1.293 (12)	C10—C11	1.400 (16)
N1—C8	1.446 (14)	C10—H10A	0.9300
C1—C6	1.367 (15)	C11—C12	1.363 (17)
C1—C2	1.405 (16)	C11—C14	1.528 (18)
C2—C3	1.388 (14)	C12—C13	1.391 (16)
C2—H2A	0.9300	C12—H12A	0.9300
C3—C4	1.344 (14)	C13—H13A	0.9300
C3—H3A	0.9300	C14—H14A	0.9600
C4—C5	1.402 (14)	C14—H14B	0.9600
C4—H4A	0.9300	C14—H14C	0.9600
C5—C6	1.432 (14)		
O2—Mo1—O2^{i}	104.8 (6)	C4—C5—H5A	120.5
O2—Mo1—O1^{i}	98.2 (3)	C6—C5—H5A	120.5

O2^{i}—Mo1—O1^{i}	98.2 (4)	C1—C6—C5	119.7 (12)
O2—Mo1—O1	98.2 (4)	C1—C6—C7	123.9 (11)
O2^{i}—Mo1—O1	98.2 (3)	C5—C6—C7	116.3 (12)
O1^{i}—Mo1—O1	153.0 (4)	N1—C7—C6	126.5 (11)
O2—Mo1—N1	167.6 (4)	N1—C7—H7A	116.7
O2^{i}—Mo1—N1	87.6 (3)	C6—C7—H7A	116.7
O1^{i}—Mo1—N1	79.2 (3)	C13—C8—C9	119.4 (13)
O1—Mo1—N1	80.2 (3)	C13—C8—N1	118.7 (12)
O2—Mo1—N1^{i}	87.6 (3)	C9—C8—N1	121.9 (12)
O2^{i}—Mo1—N1^{i}	167.6 (4)	C10—C9—C8	118.9 (14)
O1^{i}—Mo1—N1^{i}	80.2 (3)	C10—C9—H9A	120.5
O1—Mo1—N1^{i}	79.2 (3)	C8—C9—H9A	120.5
N1—Mo1—N1^{i}	80.0 (4)	C9—C10—C11	122.9 (14)
C1—O1—Mo1	136.7 (7)	C9—C10—H10A	118.6
C7—N1—C8	116.2 (10)	C11—C10—H10A	118.6
C7—N1—Mo1	122.2 (9)	C12—C11—C10	116.2 (14)
C8—N1—Mo1	121.5 (7)	C12—C11—C14	123.3 (15)
O1—C1—C6	122.9 (12)	C10—C11—C14	120.5 (14)
O1—C1—C2	116.5 (12)	C11—C12—C13	123.1 (14)
C6—C1—C2	120.6 (12)	C11—C12—H12A	118.4
C3—C2—C1	118.0 (12)	C13—C12—H12A	118.4
C3—C2—H2A	121.0	C8—C13—C12	119.5 (13)
C1—C2—H2A	121.0	C8—C13—H13A	120.2
C4—C3—C2	123.4 (12)	C12—C13—H13A	120.2
C4—C3—H3A	118.3	C11—C14—H14A	109.5
C2—C3—H3A	118.3	C11—C14—H14B	109.5
C3—C4—C5	119.2 (12)	H14A—C14—H14B	109.5
C3—C4—H4A	120.4	C11—C14—H14C	109.5
C5—C4—H4A	120.4	H14A—C14—H14C	109.5
C4—C5—C6	119.0 (12)	H14B—C14—H14C	109.5
O2—Mo1—O1—C1	−136.1 (11)	C2—C1—C6—C5	−3.0 (15)
O2^{i}—Mo1—O1—C1	117.6 (11)	O1—C1—C6—C7	−5.2 (16)
O1^{i}—Mo1—O1—C1	−9.2 (10)	C2—C1—C6—C7	174.4 (9)
N1—Mo1—O1—C1	31.5 (11)	C4—C5—C6—C1	1.9 (14)
N1^{i}—Mo1—O1—C1	−50.1 (11)	C4—C5—C6—C7	−175.7 (8)
O2—Mo1—N1—C7	62 (2)	C8—N1—C7—C6	−176.2 (9)
O2^{i}—Mo1—N1—C7	−119.5 (8)	Mo1—N1—C7—C6	7.8 (13)
O1^{i}—Mo1—N1—C7	141.7 (8)	C1—C6—C7—N1	10.1 (16)
O1—Mo1—N1—C7	−20.8 (7)	C5—C6—C7—N1	−172.5 (9)
N1^{i}—Mo1—N1—C7	59.9 (7)	C7—N1—C8—C13	137.6 (10)
O2—Mo1—N1—C8	−113.4 (19)	Mo1—N1—C8—C13	−46.3 (11)
O2^{i}—Mo1—N1—C8	64.6 (8)	C7—N1—C8—C9	−44.5 (13)
O1^{i}—Mo1—N1—C8	−34.2 (7)	Mo1—N1—C8—C9	131.6 (9)
O1—Mo1—N1—C8	163.4 (8)	C13—C8—C9—C10	−0.7 (15)
N1^{i}—Mo1—N1—C8	−116.0 (8)	N1—C8—C9—C10	−178.7 (8)

Mo1—O1—C1—C6	−25.2 (17)	C8—C9—C10—C11	−0.3 (16)
Mo1—O1—C1—C2	155.2 (7)	C9—C10—C11—C12	0.8 (16)
O1—C1—C2—C3	−178.3 (9)	C9—C10—C11—C14	178.8 (10)
C6—C1—C2—C3	2.1 (16)	C10—C11—C12—C13	−0.2 (17)
C1—C2—C3—C4	0.0 (16)	C14—C11—C12—C13	−178.1 (11)
C2—C3—C4—C5	−1.0 (15)	C9—C8—C13—C12	1.3 (15)
C3—C4—C5—C6	0.1 (14)	N1—C8—C13—C12	179.3 (9)
O1—C1—C6—C5	177.4 (9)	C11—C12—C13—C8	−0.9 (16)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O2^{ii}	0.93	2.34	3.237 (18)	163
C13—H13A···O2^{iii}	0.93	2.42	3.164 (17)	136

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

Fig. 1

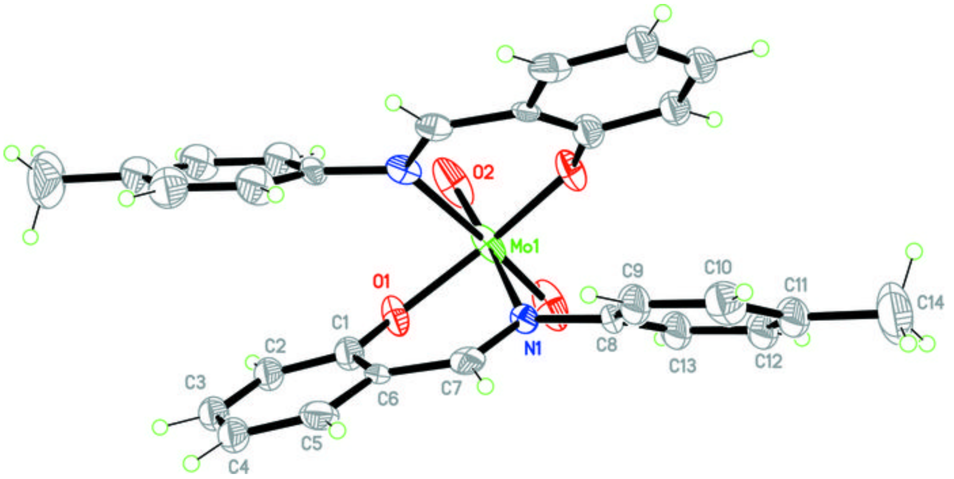

Fig. 2