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Bis{2-methoxy-6-[3-(methylamino)-propyliminomethyl]phenolato}nickel(II)

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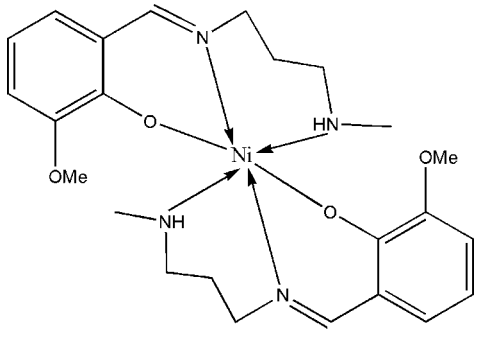
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.035; wR factor = 0.095; data-to-parameter ratio = 13.7.

The title complex, $[Ni(C_{12}H_{17}N_2O_2)_2]$, possesses a crystallographically imposed center of symmetry occupied by the Ni^{II} ion. Each 2-methoxy-6-[3-(methylamino)propyliminomethyl]-phenolate ligand coordinates the Ni atom in a tridentate mode [Ni—O = 2.0356 (18) Å, and Ni—N = 2.048 (2) and 2.184 (2) Å], resulting in a distorted octahedral coordination geometry.

Related literature

For related crystal structures, see: Zhu *et al.* (2004); Liu *et al.* (2006); Zhang (2006); Tang (2006); Diao (2007); Diao *et al.* (2007).



Experimental

Crystal data

$[Ni(C_{12}H_{17}N_2O_2)_2]$
M_r = 501.26
Orthorhombic, *Pbca*
a = 8.762 (3) Å
b = 15.297 (5) Å
c = 17.247 (5) Å
V = 2311.5 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.88 mm^{-1}
T = 298 (2) K
0.23 × 0.21 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.824, T_{max} = 0.858
11114 measured reflections
2150 independent reflections
1361 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.095
S = 1.02
2150 reflections
157 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2312).

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supplementary materials

Acta Cryst. (2007). E**63**, m2654 [**doi:10.1107/S1600536807048155**]

Bis{2-methoxy-6-[3-(methylamino)propyliminomethyl]phenolato}nickel(II)

C.-B. Tang

Comment

In continuation of our study of Ni complexes with Shiff base ligands (Tang, 2006), we report here the crystal structure of the title compound, NiL_2, where HL is a Shiff base 2-methoxy-6-[(3-methylaminopropylimino)methyl]phenol.

The title complex [Ni($C_{12}H_{17}N_2O_2$)$_2$] is a centrosymmetric mononuclear nickel(II) complex (Fig. 1). The Ni atom, lying on the inversion centre, is six-coordinated by two phenolic oxygen atoms, two imine N atoms and two amine N atoms from two Schiff base ligands, forming an octahedral coordination geometry. The coordinative bond lengths and angles are within normal ranges and comparable with those observed in similar nickel(II) complexes (Zhu *et al.*, 2004; Liu *et al.*, 2006; Zhang, 2006; Diao, 2007; Diao, Li *et al.*, 2007).

Experimental

3-Methoxy-2-hydroxybenzaldehyde (0.2 mmol, 30.5 mg) and *N*-methyl-1,3-diaminopropane (0.2 mmol, 17.6 mg) were dissolved in a methanol solution (10 ml). To the mixture was added an aqueous solution (1 ml) of nickel(II) chloride hexahydrate (0.1 mmol, 23.8 mg). The final mixture was stirred at room temperature for 30 min, resulting in a green solution. The solution was allowed to stand in air for three days, yielding green block-shaped crystals of the title complex.

Refinement

H2 attached to N2 was located from a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90 (1) Å. C-bound H atoms were geometrically positioned, with C–H = 0.93–0.97 Å, and refined as riding, with U_{iso}(H) set to 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

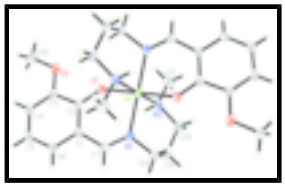

Fig. 1. The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms are related with the labelled ones by symmetry code (−*x*, 1 − *y*, 1 − *z*).

Bis{2-methoxy-6-[3-(methylamino)propyliminomethyl]phenolato}nickel(II)

Crystal data

[Ni($C_{12}H_{17}N_2O_2$)$_2$]	D_x = 1.440 Mg m^{-3}
M_r = 501.26	Mo *K*α radiation

	λ = 0.71073 Å
Orthorhombic, *Pbca*	Cell parameters from 1621 reflections
a = 8.762 (3) Å	θ = 2.3–24.5º
b = 15.297 (5) Å	μ = 0.88 mm^{-1}
c = 17.247 (5) Å	T = 298 (2) K
V = 2311.5 (12) Å^3	Block, green
Z = 4	0.23 × 0.21 × 0.18 mm
F_{000} = 1064	

Data collection

Bruker SMART CCD area-detector diffractometer	2150 independent reflections
Radiation source: fine-focus sealed tube	1361 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.058
T = 298(2) K	θ_{max} = 25.5º
ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→8
T_{min} = 0.824, T_{max} = 0.858	k = −16→18
11114 measured reflections	l = −20→20

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 atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.095	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.8022P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
2150 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
157 parameters	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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}
Ni1	0.0000	0.5000	0.5000	0.02728 (16)
O1	0.1095 (2)	0.46563 (12)	0.40048 (10)	0.0328 (5)
O2	0.2997 (2)	0.38059 (13)	0.30828 (12)	0.0461 (6)
N1	0.0197 (3)	0.62692 (15)	0.46346 (14)	0.0321 (6)
N2	−0.2128 (3)	0.50609 (15)	0.43420 (14)	0.0345 (6)
C1	0.2275 (3)	0.60365 (18)	0.37150 (15)	0.0305 (7)
C2	0.2130 (3)	0.51145 (18)	0.36524 (15)	0.0289 (6)
C3	0.3187 (3)	0.46964 (19)	0.31390 (16)	0.0332 (7)
C4	0.4273 (4)	0.5157 (2)	0.27416 (16)	0.0416 (8)
H4	0.4941	0.4863	0.2413	0.050*
C5	0.4391 (4)	0.6059 (2)	0.28230 (18)	0.0452 (8)
H5	0.5142	0.6366	0.2555	0.054*
C6	0.3404 (3)	0.6492 (2)	0.32953 (16)	0.0394 (8)
H6	0.3474	0.7096	0.3342	0.047*
C7	0.1176 (3)	0.65523 (19)	0.41434 (16)	0.0345 (7)
H7	0.1180	0.7152	0.4052	0.041*
C8	−0.1012 (3)	0.6874 (2)	0.48796 (17)	0.0419 (8)
H8A	−0.0690	0.7473	0.4792	0.050*
H8B	−0.1214	0.6802	0.5429	0.050*
C9	−0.2454 (3)	0.6683 (2)	0.44154 (19)	0.0470 (8)
H9A	−0.3209	0.7126	0.4538	0.056*
H9B	−0.2216	0.6736	0.3868	0.056*
C10	−0.3154 (3)	0.5793 (2)	0.45555 (18)	0.0413 (8)
H10A	−0.3419	0.5742	0.5100	0.050*
H10B	−0.4089	0.5746	0.4257	0.050*
C11	−0.2999 (4)	0.4240 (2)	0.42889 (19)	0.0492 (9)
H11A	−0.3447	0.4110	0.4784	0.074*
H11B	−0.2328	0.3773	0.4140	0.074*
H11C	−0.3791	0.4302	0.3908	0.074*
C12	0.3999 (4)	0.3351 (2)	0.25727 (19)	0.0553 (9)
H12A	0.3926	0.3598	0.2062	0.083*
H12B	0.3718	0.2745	0.2555	0.083*
H12C	0.5028	0.3404	0.2757	0.083*
H2	−0.174 (3)	0.5177 (15)	0.3878 (8)	0.022 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0236 (3)	0.0302 (3)	0.0281 (3)	0.0015 (2)	0.0041 (2)	−0.0008 (2)
O1	0.0306 (11)	0.0354 (11)	0.0325 (11)	−0.0025 (9)	0.0083 (10)	−0.0001 (9)
O2	0.0472 (14)	0.0403 (13)	0.0508 (13)	0.0030 (10)	0.0197 (11)	−0.0089 (11)
N1	0.0326 (15)	0.0325 (13)	0.0314 (12)	0.0035 (11)	0.0053 (12)	−0.0016 (11)

N2	0.0309 (14)	0.0408 (14)	0.0318 (13)	−0.0003 (12)	0.0030 (12)	−0.0002 (12)
C1	0.0284 (16)	0.0387 (17)	0.0244 (14)	−0.0011 (13)	−0.0005 (13)	0.0024 (13)
C2	0.0241 (15)	0.0426 (18)	0.0201 (13)	0.0000 (14)	−0.0029 (12)	0.0025 (12)
C3	0.0324 (17)	0.0400 (17)	0.0271 (15)	0.0022 (14)	0.0011 (14)	−0.0014 (13)
C4	0.0363 (18)	0.058 (2)	0.0307 (16)	0.0054 (16)	0.0105 (15)	0.0014 (15)
C5	0.041 (2)	0.051 (2)	0.0430 (18)	−0.0102 (17)	0.0111 (16)	0.0094 (16)
C6	0.0387 (18)	0.0422 (18)	0.0372 (17)	−0.0031 (15)	0.0054 (15)	0.0069 (15)
C7	0.0384 (18)	0.0318 (16)	0.0332 (15)	0.0019 (14)	−0.0047 (15)	0.0018 (13)
C8	0.043 (2)	0.0331 (17)	0.0495 (19)	0.0055 (15)	0.0078 (17)	−0.0031 (14)
C9	0.0404 (19)	0.046 (2)	0.055 (2)	0.0172 (16)	0.0023 (17)	0.0016 (17)
C10	0.0263 (17)	0.055 (2)	0.0428 (18)	0.0085 (15)	−0.0007 (15)	0.0007 (16)
C11	0.045 (2)	0.052 (2)	0.051 (2)	−0.0074 (17)	−0.0110 (17)	−0.0031 (16)
C12	0.063 (2)	0.052 (2)	0.052 (2)	0.0125 (18)	0.019 (2)	−0.0094 (17)

Geometric parameters (Å, °)

Ni1—O1^{i}	2.0356 (18)	C4—H4	0.9300
Ni1—O1	2.0356 (18)	C5—C6	1.360 (4)
Ni1—N1	2.048 (2)	C5—H5	0.9300
Ni1—N1^{i}	2.048 (2)	C6—H6	0.9300
Ni1—N2^{i}	2.184 (2)	C7—H7	0.9300
Ni1—N2	2.184 (2)	C8—C9	1.524 (4)
O1—C2	1.297 (3)	C8—H8A	0.9700
O2—C3	1.376 (3)	C8—H8B	0.9700
O2—C12	1.424 (3)	C9—C10	1.513 (4)
N1—C7	1.281 (3)	C9—H9A	0.9700
N1—C8	1.469 (3)	C9—H9B	0.9700
N2—C11	1.473 (4)	C10—H10A	0.9700
N2—C10	1.483 (4)	C10—H10B	0.9700
N2—H2	0.886 (10)	C11—H11A	0.9600
C1—C6	1.410 (4)	C11—H11B	0.9600
C1—C2	1.420 (4)	C11—H11C	0.9600
C1—C7	1.448 (4)	C12—H12A	0.9600
C2—C3	1.432 (4)	C12—H12B	0.9600
C3—C4	1.368 (4)	C12—H12C	0.9600
C4—C5	1.392 (4)		
O1^{i}—Ni1—O1	180.0	C6—C5—H5	120.1
O1^{i}—Ni1—N1	93.12 (8)	C4—C5—H5	120.1
O1—Ni1—N1	86.88 (8)	C5—C6—C1	120.9 (3)
O1^{i}—Ni1—N1^{i}	86.88 (8)	C5—C6—H6	119.6
O1—Ni1—N1^{i}	93.12 (8)	C1—C6—H6	119.6
N1—Ni1—N1^{i}	180.0	N1—C7—C1	126.8 (3)
O1^{i}—Ni1—N2^{i}	88.59 (8)	N1—C7—H7	116.6
O1—Ni1—N2^{i}	91.41 (8)	C1—C7—H7	116.6
N1—Ni1—N2^{i}	97.37 (9)	N1—C8—C9	109.0 (2)
N1^{i}—Ni1—N2^{i}	82.63 (9)	N1—C8—H8A	109.9

$O1^{i}$—Ni1—N2	91.41 (8)	C9—C8—H8A	109.9
O1—Ni1—N2	88.59 (8)	N1—C8—H8B	109.9
N1—Ni1—N2	82.63 (9)	C9—C8—H8B	109.9
$N1^{i}$—Ni1—N2	97.37 (9)	H8A—C8—H8B	108.3
$N2^{i}$—Ni1—N2	180.0	C10—C9—C8	115.1 (3)
C2—O1—Ni1	125.81 (17)	C10—C9—H9A	108.5
C3—O2—C12	116.9 (2)	C8—C9—H9A	108.5
C7—N1—C8	117.4 (2)	C10—C9—H9B	108.5
C7—N1—Ni1	125.5 (2)	C8—C9—H9B	108.5
C8—N1—Ni1	116.60 (19)	H9A—C9—H9B	107.5
C11—N2—C10	110.2 (2)	N2—C10—C9	113.2 (2)
C11—N2—Ni1	116.00 (19)	N2—C10—H10A	108.9
C10—N2—Ni1	114.88 (18)	C9—C10—H10A	108.9
C11—N2—H2	108.1 (16)	N2—C10—H10B	108.9
C10—N2—H2	107.6 (16)	C9—C10—H10B	108.9
Ni1—N2—H2	98.9 (17)	H10A—C10—H10B	107.7
C6—C1—C2	121.0 (3)	N2—C11—H11A	109.5
C6—C1—C7	117.3 (3)	N2—C11—H11B	109.5
C2—C1—C7	121.3 (3)	H11A—C11—H11B	109.5
O1—C2—C1	124.3 (2)	N2—C11—H11C	109.5
O1—C2—C3	120.0 (3)	H11A—C11—H11C	109.5
C1—C2—C3	115.6 (2)	H11B—C11—H11C	109.5
C4—C3—O2	124.0 (3)	O2—C12—H12A	109.5
C4—C3—C2	122.0 (3)	O2—C12—H12B	109.5
O2—C3—C2	114.0 (2)	H12A—C12—H12B	109.5
C3—C4—C5	120.8 (3)	O2—C12—H12C	109.5
C3—C4—H4	119.6	H12A—C12—H12C	109.5
C5—C4—H4	119.6	H12B—C12—H12C	109.5
C6—C5—C4	119.7 (3)		

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

Fig. 1