Bis[1,3-dihydroxy-2-hydroxymethyl-2-(2-oxido-benzylideneamino)propane-$\kappa^3 N,O,O'$]nickel(II) pyridine solvate

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The Ni^{II} atom in the title complex, [Ni(C₁₁H₁₄NO₄)₂]·C₅H₅N, is chelated by a terdentate Schiff base anion in a slightly disotorted octahedral geometry. One of the two coordinated hydroxyl groups forms a hydrogen bond to the pyridine solvent while other hydroxyl groups are engaged in inter-molecular hydrogen bonding, forming a two-dimensional layer.

Comment

The preceeding paper reports the crystal structure of the Ni complex based on the ligand derived by condensing 5-nitro-salicyaldehyde with tris(hydroxymethylamino)methane; the compound crystallizes as a pyridine solvate (Ali *et al.*, 2006). The title nickel analog, (I) (Fig. 1), is also a pyridine solvate, and shows an identical hydrogen-bonding scheme (Table 2), with neighboring molecules connected into a tightly held layer. The absence of the nitro substitutent in (I) has no significant effect on the bond distances or angles of the molecule.

(I)
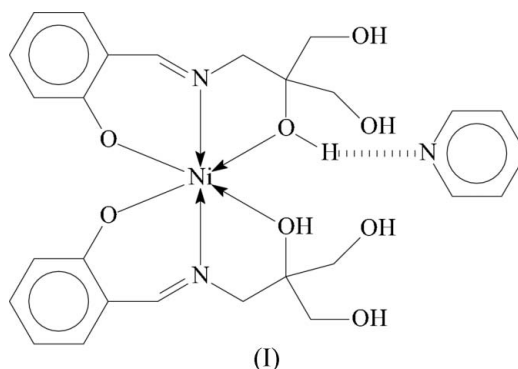
The crystal structure of the parent Schiff base, namely 2-{[tris(hydroxymethyl)methyl]aminomethylene}cyclohexa-3,5-dien-1(2*H*)-one, a water-soluble compound, has already been reported (Asgedom *et al.*, 1996; Chumakov *et al.*, 2000; Odabaşoğlu *et al.*, 2003; Tatar *et al.*, 2005; Zhang *et al.*, 2000). This Schiff base forms complexes with nickel (Dey *et al.*, 2002; Rustagi & Rao, 1975; Tsapkov *et al.*, 1994; 2004); one of these being the tris-pyridine adduct of the doubly deprotonated ligand (Dey *et al.*, 2002). The title complex is the only other structural study of a nickel derivative of this class of ligand.

Experimental

1,3-Dihydroxy-2-(2-hydroxybenzylideneamino)-2-(hydroxymethyl)-propane was synthesized from tris(hydroxymethyl)aminomethane and salicylaldehyde according a literature procedure (Odabasoglu *et*

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al., 2003). This compound (0.41 g, 1.82 mmol) was dissolved in ethanol (25 ml) and several drops of aqueous sodium hydroxide were added to raise the pH of the solution to about 8.5. Nickel(II) acetate (0.23 g, 0.92 mmol) was then added and the mixture heated for 5 h. The solvent was removed and the product recrystallized from pyridine.

Crystal data

[Ni(C$_{11}$H$_{14}$NO$_4$)$_2$]·C$_5$H$_5$N
$M_r = 586.27$
Monoclinic, $P2_1/n$
$a = 11.1164$ (2) Å
$b = 11.5298$ (2) Å
$c = 21.1733$ (3) Å
$\beta = 106.390$ (1)°
$V = 2603.50$ (7) Å^3

$Z = 4$
$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.80$ mm^{-1}
$T = 173$ (2) K
Prism, green
$0.35 \times 0.30 \times 0.20$ mm

Data collection

Bruker APEXII area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.650$, $T_{max} = 0.856$

38368 measured reflections
5978 independent reflections
4697 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.108$
$S = 1.12$
5978 reflections
376 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 1.3685P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.77$ e Å^{-3}
$\Delta\rho_{min} = -0.81$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.004 (2)	Ni1—O6	2.159 (2)
Ni1—O2	2.070 (2)	Ni1—N1	2.037 (2)
Ni1—O5	2.025 (2)	Ni1—N2	2.034 (2)
O1—Ni1—O2	169.66 (6)	O2—Ni1—N2	94.50 (7)
O1—Ni1—O5	92.51 (6)	O5—Ni1—N1	95.61 (7)
O1—Ni1—O6	90.15 (6)	O5—Ni1—N2	89.55 (7)
O1—Ni1—N1	91.94 (7)	O5—Ni1—O6	168.25 (6)
O1—Ni1—N2	93.15 (7)	O6—Ni1—N1	95.74 (7)
O2—Ni1—O5	94.49 (7)	O6—Ni1—N2	78.87 (7)
O2—Ni1—O6	84.52 (6)	N1—Ni1—N2	172.59 (7)
O2—Ni1—N1	79.82 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2o···N3	0.85 (1)	1.76 (1)	2.599 (2)	169 (3)
O3—H3o···O5^i	0.85 (1)	1.75 (1)	2.593 (2)	177 (3)
O4—H4o···O3^{ii}	0.85 (1)	1.82 (1)	2.651 (2)	169 (3)
O6—H6o···O7^{iii}	0.84 (1)	2.12 (2)	2.868 (2)	148 (3)
O7—H7o···O4^{iv}	0.85 (1)	1.90 (1)	2.734 (2)	166 (4)
O8—H8o···O1^{iii}	0.85 (1)	1.88 (1)	2.728 (2)	174 (3)

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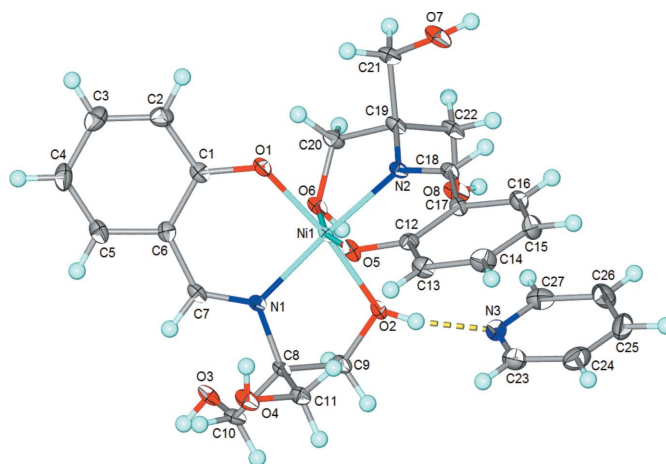

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 70% probability level and H atoms are shown as spheres of arbitrary radii. The dashed line denotes a hydrogen bond.

The carbon-bound H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$. The hydroxyl H atoms were located in a difference Fourier map, and were refined with a distance restraint [O—H = 0.85 (1) Å]; their displacement parameters were freely refined.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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