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Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.032

wR factor = 0.097

Data-to-parameter ratio = 14.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Bis[1,3-dihydroxy-2-hydroxymethyl-2-(5-nitro-
2-oxidobenzylideneamino)propane- $\kappa^3\text{N},\text{O},\text{O}'$]-
nickel(II) pyridine solvate**

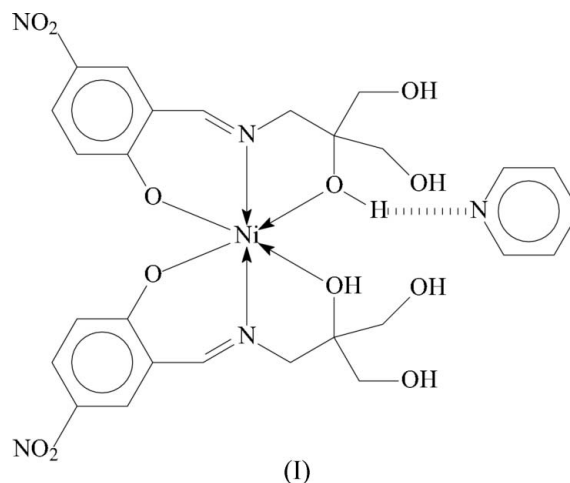
The Ni^{II} atom in the title complex, $[\text{Ni}(\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_6)_2] \cdot \text{C}_5\text{H}_5\text{N}$, is chelated by a terdentate Schiff base anion in a slightly octahedral geometry. One of the coordinated hydroxyl groups forms a hydrogen bond with the pyridine solvent molecule. In the crystal structure, other hydroxyl groups are involved in intermolecular hydrogen bonding, forming a two-dimensional layer.

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Comment

The preceding paper reports the crystal structure of the zinc derivative of the Schiff base derived by condensing 5-nitrosalicylaldehyde with tris(hydroxymethyl)amino methane; the compound crystallizes as a pyridine solvate (Ali *et al.*, 2006). The title Ni analog, (I), (Fig. 1), is isostructural, and in the crystal structure, an identical hydrogen-bonding motif (Table 2) links neighboring molecules into a tightly held two-dimensional layer.

**Experimental**

1,3-Dihydroxy-2-hydroxymethyl-2-(2-hydroxy-5-nitrobenzylideneamino)propane was synthesized from tris(hydroxymethyl)amino methane and 5-nitrosalicylaldehyde according a literature procedure (Chumakov *et al.*, 2003, 2005). This ligand (0.30 g, 1.11 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 (0.30 g, 0.57 mmol) was then added and the mixture heated for 5 h. The solvent was removed and the product recrystallized from pyridine.

Crystal data

[Ni(C₁₁H₁₃N₂O₆)₂].C₅H₅N
M_r = 676.28
 Monoclinic, *P*2₁/*c*
a = 11.2465 (1) Å
b = 11.4857 (2) Å
c = 21.9861 (3) Å
 β = 101.105 (1)°
V = 2786.85 (7) Å³

Z = 4
D_x = 1.612 Mg m⁻³
 Mo *K*α radiation
 μ = 0.77 mm⁻¹
T = 173 (2) K
 Prism, green
 0.35 × 0.31 × 0.26 mm

Data collection

Bruker APEXII area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.675, *T_{max}* = 0.824

39058 measured reflections
 6373 independent reflections
 6017 reflections with *I* > 2σ(*I*)
R_{int} = 0.027
 θ_{max} = 27.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.032
wR(*F*²) = 0.097
S = 1.08
 6373 reflections
 430 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 2.8761P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.72 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{min} = -0.81 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1—O1	2.021 (1)	Ni1—O10	2.123 (1)
Ni1—O4	2.080 (1)	Ni1—N2	2.031 (1)
Ni1—O7	2.040 (1)	Ni1—N4	2.024 (1)
O1—Ni1—O4	170.81 (5)	O4—Ni1—N4	92.59 (5)
O1—Ni1—O7	90.63 (5)	O7—Ni1—O10	167.14 (5)
O1—Ni1—O10	87.40 (5)	O7—Ni1—N2	96.15 (5)
O1—Ni1—N2	91.79 (5)	O7—Ni1—N4	88.23 (5)
O1—Ni1—N4	96.11 (5)	N2—Ni1—N4	170.93 (6)
O4—Ni1—O7	92.59 (5)	O10—Ni1—N2	96.60 (5)
O4—Ni1—O10	91.32 (5)	O10—Ni1—N4	79.36 (5)
O4—Ni1—N2	79.32 (5)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4o...N5	0.85 (1)	1.80 (1)	2.653 (2)	175 (3)
O5—H5o...O6 ⁱ	0.85 (1)	1.79 (1)	2.606 (2)	162 (3)
O6—H6o...O7 ⁱⁱ	0.85 (1)	1.77 (1)	2.611 (2)	176 (3)
O10—H10o...O12 ⁱⁱⁱ	0.84 (1)	1.98 (2)	2.757 (2)	153 (3)
O11—H11o...O1 ⁱⁱⁱ	0.85 (1)	1.94 (1)	2.783 (2)	178 (3)
O12—H12o...O5 ^{iv}	0.85 (1)	1.93 (2)	2.739 (2)	159 (3)

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

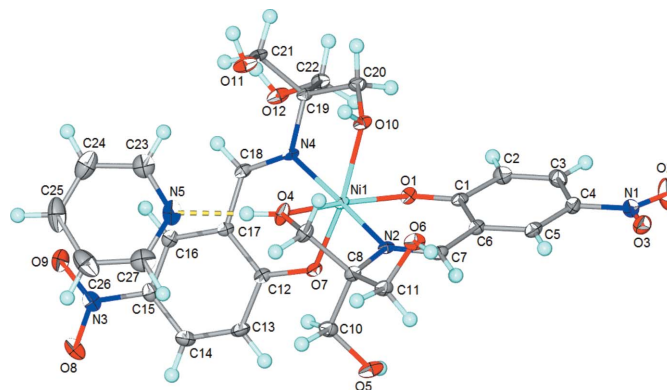


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 70% probability level and H atoms 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) = 1.2*U_{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] and isotropic displacement parameters.

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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