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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
Disorder in solvent or counterion
R factor = 0.054
wR factor = 0.155
Data-to-parameter ratio = 15.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

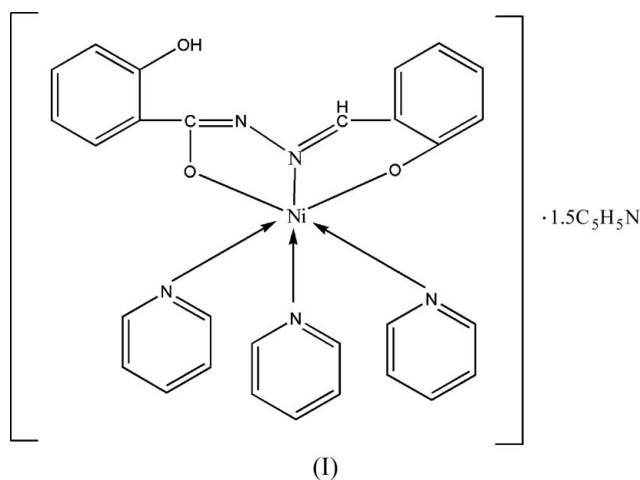
Tris(pyridine- κN)[salicylaldehyde (2-hydroxybenzoyl)hydrazonato- $\kappa^2\text{N},\text{O}$]nickel(II) pyridine sesquisolvate

In the title compound, $[\text{Ni}(\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_3)(\text{C}_5\text{H}_5\text{N})_3] \cdot 1.5\text{C}_5\text{H}_5\text{N}$, the Ni^{II} ion is coordinated by one N and two O atoms from a Schiff base ligand and by the N atoms of three pyridine molecules to form a distorted octahedral geometry.

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Comment

Previously we have reported the crystal structure of a salicylaldehyde salicylhydrazone Zn^{II} pyridine complex (Hu *et al.*, 2005). Now we report the crystal structure of the title compound, (I).



The asymmetric unit of (I) contains a $[\text{Ni}(\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_3)-(\text{C}_5\text{H}_5\text{N})_3]$ unit, a pyridine molecule at a general position and another pyridine molecule disordered over a crystallographic inversion centre (Fig. 1). The Ni^{II} ion is coordinated by one N and two O atoms from a Schiff base ligand, and by three N atoms of three pyridine molecules. This NiN_4O_2 coordination forms a distorted octahedral geometry (Table 1). The crystal packing is stabilized by $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 2).

Experimental

To a ethanol solution (100 ml) of salicylhydrazine (6 g), one molar equivalent of salicylaldehyde in ethanol (50 ml) was added slowly with continuous stirring and the salicylaldehyde salicylhydrazone was precipitated at once. Salicylaldehyde salicylhydrazone (1 mmol), $\text{Ni}(\text{OAc})_2$ (1 mmol), DMF (30 ml), and pyridine (10 ml) were refluxed for 1 h. The hot solution was filtered and allowed to stand at room temperature for 21 d, and red crystals of compound (I) were obtained.

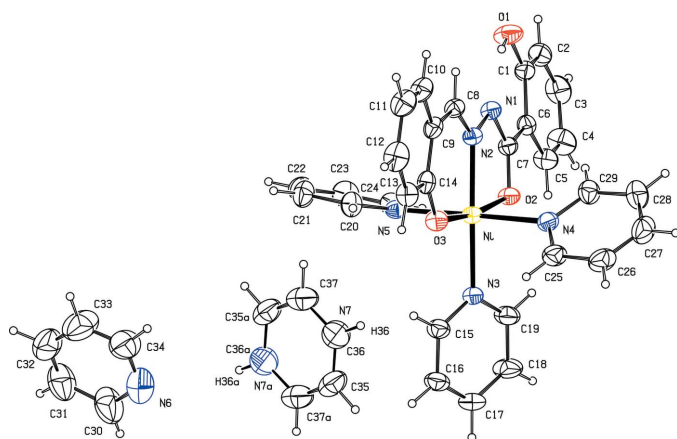


Figure 1

The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level. Atoms N7 and C36 are disordered and occupy the same site with an occupancy of 0.50, together with the H atom attached to C36. Atoms labeled with the suffix a are generated by the symmetry operation $(1 - x, 1 - y, 1 - z)$.

Crystal data

$[\text{Ni}(\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_3)(\text{C}_5\text{H}_5\text{N})_3] \cdot$
 $1.5\text{C}_5\text{H}_5\text{N}$
 $M_r = 668.90$
 Monoclinic, $P2_1/n$
 $a = 12.5579$ (10) Å
 $b = 20.3993$ (17) Å
 $c = 13.2756$ (11) Å
 $\beta = 93.493$ (1)°
 $V = 3394.5$ (5) Å³
 $Z = 4$

$D_x = 1.309$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 6390
 reflections
 $\theta = 2.3\text{--}24.7^\circ$
 $\mu = 0.62$ mm⁻¹
 $T = 293$ (2) K
 Block, red
 $0.40 \times 0.30 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2000)
 $T_{\min} = 0.791$, $T_{\max} = 0.887$
 18260 measured reflections

6652 independent reflections
 5272 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 26.0^\circ$
 $h = -15 \rightarrow 15$
 $k = -19 \rightarrow 25$
 $l = -16 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.155$
 $S = 1.02$
 6652 reflections
 425 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 1.8492P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.38$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni—O3	1.993 (2)	Ni—N3	2.083 (2)
Ni—N2	1.995 (2)	Ni—N4	2.160 (3)
Ni—O2	2.0475 (19)	Ni—N5	2.176 (2)
O3—Ni—N2	91.70 (9)	O2—Ni—N4	89.83 (10)
O3—Ni—O2	170.75 (8)	N3—Ni—N4	90.08 (10)
N2—Ni—O2	79.10 (9)	O3—Ni—N5	90.64 (9)
O3—Ni—N3	96.05 (9)	N2—Ni—N5	87.96 (9)
N2—Ni—N3	171.75 (9)	O2—Ni—N5	89.90 (9)
O2—Ni—N3	93.19 (9)	N3—Ni—N5	89.11 (9)
O3—Ni—N4	89.76 (10)	N4—Ni—N5	179.13 (9)
N2—Ni—N4	92.80 (9)		

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H1 \cdots N1	0.82	1.82	2.549 (3)	147
C16—H16 \cdots O1 ¹	0.93	2.58	3.356 (5)	141
C20—H20 \cdots O3	0.93	2.43	3.022 (4)	121
C24—H24 \cdots O2	0.93	2.47	3.030 (4)	119
C19—H19 \cdots O2	0.93	2.52	3.059 (4)	118

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

Atoms N7, C36 and H36 are disordered across a crystallographic inversion centre and hence the occupancy factor for each disordered component was fixed at 0.50. The H atoms were placed in idealized positions and allowed to ride on their parent atoms, with O—H = 0.82 Å, C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C/O})$. The highest peak in the difference map lies close to the Ni atom (0.82 Å).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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