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{4-Chloro-2′-[1-(5-chloro-2-oxidophenyl)-ethylidene-κ^3*O*,*N*$^{2'}$,*O*′]benzhydrazido}-tris(pyridine-κ*N*)nickel(II) pyridine solvate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.006 Å
R factor = 0.061
wR factor = 0.141
Data-to-parameter ratio = 17.4

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

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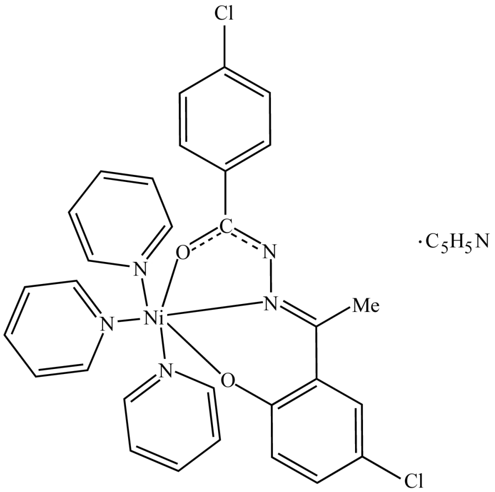
The crystal structure of the title compound, $[Ni(C_{15}H_{10}Cl_2N_2O_2)(C_5H_5N)_3]\cdot C_5H_5N$, is built of molecules of a mononuclear nickel complex and uncoordinated solvent pyridine molecules. The Ni^{II} atom in the complex is coordinated by two O and one N atoms of the meridional tridentate ligand, *viz*. the twice deprotonated hydrazone formed from 2-hydroxy-5-chloroacetophenone and *p*-chlorobenzhydrazide. The remaining three coordination sites of the Ni octahedron are occupied by three coordinated pyridine ligands.

Comment

The title compound, (I), has a structure similar to $[Ni(C_{15}H_{10}ClN_3O_3)(C_5H_5N)_3]\cdot C_5H_5N$, reported by Ali *et al.* (2004).



(I)

The Ni atom has a distorted octahedral coordination environment formed by the two O and one N atom of the meridional tridentate ligand and the N atoms of the three pyridine groups (Fig. 1). The tridentate benzohydrazide ligand coordinates to the Ni atom through its central hydrazone N atom and the O atoms of two deprotonated peripheral phenol groups [Ni1—O1 = 2.033 (2) Å and Ni1—O2 = 1.972 (2) Å]. The Ni1—N2 bond involving the hydrazone N atom [Ni1—N2 = 2.009 (2) Å], as well as the somewhat longer Ni1—N3 bond with the pyridine ligand in the *trans* position to the hydrazone [Ni1—N3 = 2.092 (2) Å and N2—Ni1—N3 = 176.86 (10)°] are nevertheless substantially shorter than the other two Ni—N_{py} bonds which are in *trans* positions to each other [Ni1—N4 = 2.183 (2) Å, Ni1—N5 = 2.174 (2) Å and N4—Ni1—N5 = 172.50 (9)°]. The same pattern in the Ni—N bond lengths, with

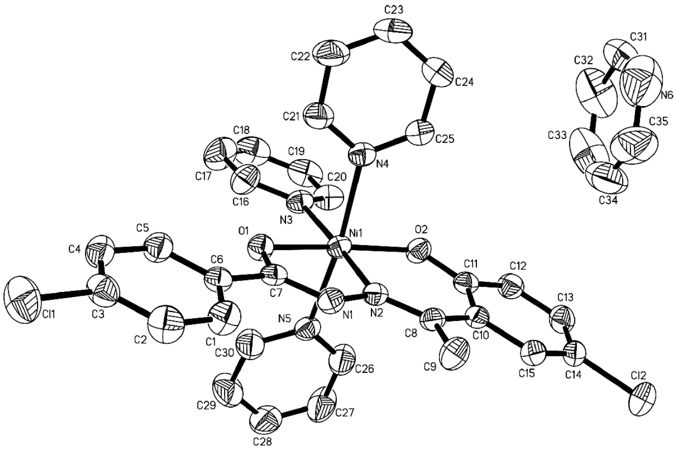


Figure 1
The molecular structure of the title compound, with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

remarkably similar bond-length values, was observed in the above-mentioned analogous complex (Ali *et al.*, 2004). The smaller but notable difference in the Ni—O bonds, with the bond involving the O atom of the benzhydrazide component [Ni1—O1 = 2.033 (2) Å] being somewhat longer than the bond involving the O atom of the acetophenone component [Ni1—O2 = 1.972 (2) Å], is also observed in both complexes. The potential delocalization of electron density over the NCO fragment in the benzhydrazide moiety (see scheme) may well be the reason for the differences in the Ni—O bonds.

The bond lengths and angles of the Schiff base ligand are in normal ranges (Allen *et al.*, 1987; Orpen *et al.*, 1989). The chelate fragment, Ni1/O1/C7/N1/N2/C8/C10/C11/O2, is essentially planar; atom C10 exhibits the maximum deviation from the mean plane of 0.167 (3) Å. The benzene groups (C1–C6 and C10–C15) form dihedral angles of 4.65 (13) and 7.41 (11)°, respectively, with the mean plane of the tridentate ligand.

Experimental

2-Hydroxyacetophenone (0.24 g, 1.8 mmol, purchased from Avocado) was dissolved in a 250 ml round-bottomed flask containing ethanol (30 ml). *p*-Chlorobenzhydrazide (0.3 g, 1.8 mmol, Avocado) was added to the solution, followed by nickel acetate tetrahydrate (0.22 g, 0.9 mmol). The solution was refluxed with stirring for 5 h. The red solid obtained was filtered off and and the resulting solid recrystallized from pyridine.

Crystal data

$[Ni(C_{15}H_{10}Cl_2N_2O_2)(C_5H_5N)_3]\cdot C_5H_5N$
$M_r = 696.26$
Monoclinic, $P2_1/n$
$a = 12.6009$ (17) Å
$b = 20.845$ (3) Å
$c = 12.6417$ (17) Å
$\beta = 90.418$ (3)°
$V = 3320.4$ (8) Å^3
$Z = 4$
$D_x = 1.393$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3704 reflections
$\theta = 1.8$–27.0°
$\mu = 0.79$ mm^{-1}
$T = 273$ (2) K
Block, yellow
0.27 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.816$, $T_{\max} = 0.912$
19309 measured reflections
7232 independent reflections
4961 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 27.0°$
$h = -16 \rightarrow 14$
$k = -26 \rightarrow 17$
$l = -16 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.141$
$S = 1.09$
7232 reflections
415 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.4914P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.033 (2)	N1—N2	1.393 (3)
Ni1—O2	1.972 (2)	N1—C7	1.309 (4)
Ni1—N2	2.009 (2)	N2—C8	1.299 (4)
Ni1—N3	2.092 (2)	O1—C7	1.283 (3)
Ni1—N4	2.183 (2)	O2—C11	1.296 (3)
Ni1—N5	2.174 (2)		
O1—Ni1—N3	96.73 (9)	N2—Ni1—O1	80.19 (9)
O1—Ni1—N4	87.38 (9)	N2—Ni1—N3	176.86 (10)
O1—Ni1—N5	89.96 (9)	N2—Ni1—N5	93.92 (9)
O2—Ni1—N2	90.31 (9)	N2—Ni1—N4	92.53 (9)
O2—Ni1—N3	92.79 (10)	N3—Ni1—N5	85.43 (9)
O2—Ni1—O1	170.27 (8)	N3—Ni1—N4	87.91 (9)
O2—Ni1—N4	90.99 (9)	N5—Ni1—N4	172.50 (9)
O2—Ni1—N5	92.79 (10)		

After their location in a difference map, all H atoms were positioned geometrically and allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$(C) for methyl H atoms. The N atom of the pyridine solvent molecule was identified on the basis of the absence of an attached H atom in the Fourier map.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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