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{2′-[1-(5-Bromo-2-oxidophenyl)ethylidene]-benzohydrazidato(2−)}tris(pyridine)nickel(II)] pyridine solvate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.008 Å
R factor = 0.059
wR factor = 0.137
Data-to-parameter ratio = 16.0

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

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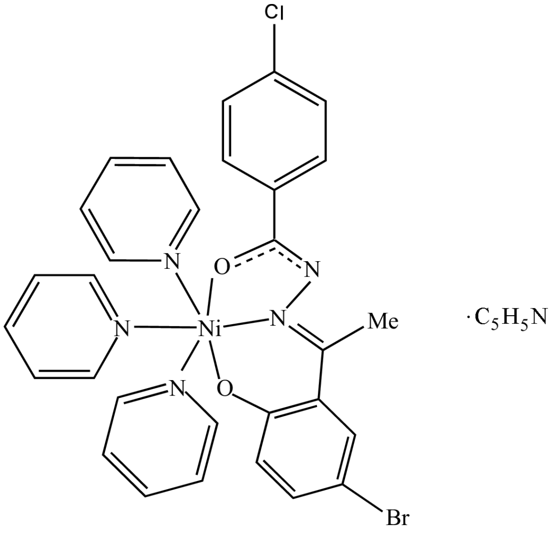
In the title compound, $[\mathrm{Ni(C_{15}H_{10}BrClN_2O_2)(C_5H_5N)_3}]\cdot\mathrm{C_5H_5N}$, the Ni atom is coordinated by three pyridine molecules and one oxygen-containing ligand in a tridentate manner *via* O, N and O atoms. The geometry of the Ni atom is distorted octahedral. There are no significant intermolecular interactions in the crystal structure.

Comment

The stable intermediate complex $[\mathrm{Ni(C_{15}H_{12}BrClN_2O_2)(NC_6H_5)_3}]$, (II), was previously obtained when reacting nickel acetate with the Schiff base ligand in the presence of triethylamine (Ali *et al.*, 2004). Recrystallization of this complex from pyridine gave the title compound, (I).



(I)

The Ni^{II} atom adopts an octahedral geometry different from the square-planar environment of (II). The molecular structure of (I) and selected bond lengths and angles are given in Fig. 1 and Table 1, respectively. The ligand is chelated to the Ni atom *via* O, N and O atoms in a tridentate manner. The axial bond lengths of 2.180 (3) and 2.186 (3) Å for Ni—N4 and Ni—N5, respectively, are longer than the equatorial bond lengths of 2.012 (3) Å for Ni—N2 and 2.095 (3) Å for Ni—N3. The results are comparable with those found in other octahedral nickel complexes, *viz*. $[\mathrm{Ni(C_{16}H_{24}N_2O)_2(N_3)_2}]$ (You *et al.*, 2004) and $[\mathrm{Ni(C_{15}H_{11}ClN_3O_3)_2}]\cdot\mathrm{C_5H_5N}$ (Ali *et al.*, 2004). The structural dimensions of the ligand are in normal ranges (Allen *et al.*, 1987; Orpen *et al.*, 1989). The chlorophenyl C7/C1–C6 and the bromophenyl C15/C10–C14 planes are inclined to each other at an angle of 4.6 (2)°.

There are several weak intramolecular C—H⋯N and C—H⋯O interactions (Table 2). However, no significant intermolecular interactions were found.

Experimental

The complex was synthesized by the template condensation of 5-bromo-2-hydroxyacetophenone (0.3 g, 2.2 mmol) and *p*-chlorobenzohydrazide (0.3 g, 2.2 mmol) with nickel acetate tetrahydrate (0.24 g, 1.1 mmol) by refluxing and stirring in ethanol for 5 h. The red–brown solid was filtered off and recrystallized from pyridine.

Crystal data

$[Ni(C_{15}H_{10}BrClN_2O_2)(C_5H_5N)_3]\cdot C_5H_5N$
$M_r = 740.72$
Monoclinic, $P2_1/n$
$a = 12.587$ (3) Å
$b = 21.037$ (5) Å
$c = 12.738$ (3) Å
$\beta = 90.907$ (5)°
$V = 3372.5$ (13) Å^3
$Z = 4$

$D_x = 1.459$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 826 reflections
$\theta = 1.9$–25.9°
$\mu = 1.88$ mm^{-1}
$T = 273$ (2) K
Block, red
0.25 × 0.19 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.638$, $T_{\max} = 0.713$
17985 measured reflections

6623 independent reflections
4038 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 25.9°$
$h = -15 \rightarrow 14$
$k = -25 \rightarrow 24$
$l = -12 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.137$
$S = 1.01$
6623 reflections
415 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.7524P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.59$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O2	1.973 (3)	Cl1—C3	1.741 (5)
Ni1—N2	2.012 (3)	O1—C7	1.290 (4)
Ni1—O1	2.030 (3)	O2—C15	1.298 (5)
Ni1—N3	2.095 (3)	N1—C7	1.307 (5)
Ni1—N4	2.180 (3)	N1—N2	1.394 (4)
Ni1—N5	2.186 (3)	N2—C8	1.295 (5)
Br2—C12	1.900 (4)		
O2—Ni1—N2	90.20 (12)	O1—Ni1—N4	87.35 (12)
O2—Ni1—O1	170.35 (11)	N3—Ni1—N4	88.13 (12)
N2—Ni1—O1	80.38 (12)	O2—Ni1—N5	92.93 (13)
O2—Ni1—N3	92.50 (13)	N2—Ni1—N5	93.37 (12)
N2—Ni1—N3	177.17 (14)	O1—Ni1—N5	89.70 (13)
O1—Ni1—N3	96.96 (13)	N3—Ni1—N5	85.62 (12)
O2—Ni1—N4	91.06 (13)	N4—Ni1—N5	172.72 (13)
N2—Ni1—N4	92.69 (12)		

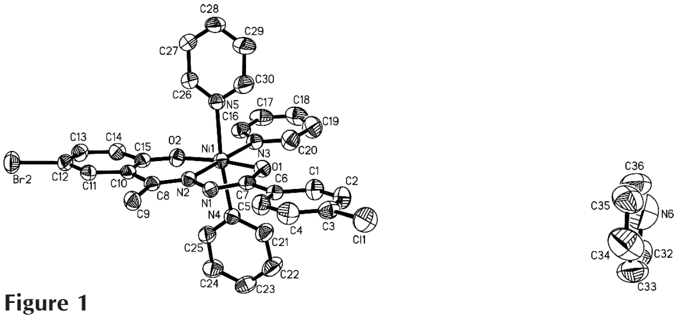


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids. H atoms have been omitted.

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C9—H9*A*⋯N1	0.96	2.23	2.697 (5)	109
C16—H16*A*⋯O2	0.93	2.42	3.002 (5)	120
C20—H20*A*⋯O1	0.93	2.56	3.154 (6)	122
C21—H21*A*⋯O1	0.93	2.49	3.014 (6)	116
C25—H25*A*⋯O2	0.93	2.47	3.044 (6)	120
C26—H26*A*⋯O2	0.93	2.53	3.102 (6)	120
C30—H30*A*⋯O1	0.93	2.45	3.037 (6)	121

After their location in a Fourier difference map, all H atoms were placed geometrically at ideal positions and allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and $U_{\rm iso}$(H) = 1.2 or 1.5$U_{\rm eq}$(C).

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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