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(5-Bromo-2-oxidoacetophenone 4-chlorobenzoylhydrazido)(triethylamine)nickel(II)

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Key indicators

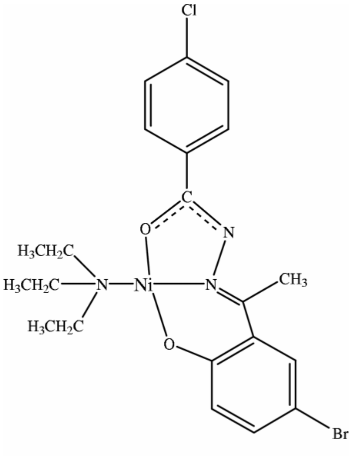
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.083
Data-to-parameter ratio = 16.9

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

In the title compound, $[Ni(C_{15}H_{12}BrClN_2O_2)(C_6H_{15}N)]$, the Ni^{II} atom exists in square-planar geometry, coordinated by a molecule of triethylamine through the N atom and by a tridentate Schiff base ligand *via* N and O atoms. No significant intermolecular interactions are found in the crystal structure.

Comment

Triethylamine is normally used for the deprotonation of Schiff base ligands before coordination to metals. The title compound, (I), is the first example of coordination of triethylamine to a metal atom prior to complexation.



(I)

Compound (I) was obtained by reacting an ethanolic solution of nickel acetate with the Schiff base ligand in the presence of triethylamine. The geometry of the Ni atom is square planar, with *cis* angles in the range 88.15 (7)–94.99 (7)°. The ligand is chelated to the Ni atom *via* O1, N1 and O2 in a tridentate manner (Fig. 1 and Table 1). One molecule of triethylamine is coordinated to the Ni atom, with a bond length of 2.0095 (16) Å. Atoms O1—Ni1—O2 make an angle of 178.96 (7)°. The chelating fragment O1/O2/N1/N3 is perfectly planar. The bond lengths Ni1—N1 and Ni1—O1 of 1.8460 (16) and 1.8128 (15) Å, respectively, are comparable to those in another Ni^{II} square-planar complex (Bian *et al.*, 2004), with an Ni1—N1 bond length of 1.859 (4) Å and an Ni1—O1 bond length of 1.831 (3) Å. The structural dimensions of the ligand are in the normal ranges (Allen *et al.*, 1987; Orpen *et al.*, 1989). The chlorophenyl and bromophenolate rings are planar and make a dihedral angle of 3.16 (12)° with each other. There is a short intramolecular C—H···N contact (Table 2).

However, significant intermolecular interactions are not found in the crystal structure of (I).

Experimental

The title complex was synthesized by condensation of 5-bromo-2-hydroxyacetophenone *p*-chlorobenzoylhydrazone (0.3 g, 2.2 mmol) and nickel acetate dihydrate (0.24 g, 1.1 mmol) in the presence of a few drops of triethylamine, followed by refluxing and stirring in ethanol for 5 h. Dark red crystals were obtained after standing at room temperature for a few days.

Crystal data

$[Ni(C_{15}H_{12}BrClN_2O_2)(C_6H_{15}N)]$
$M_r = 525.51$
Triclinic, $P\bar{1}$
$a = 9.846$ (2) Å
$b = 10.410$ (2) Å
$c = 12.253$ (3) Å
$\alpha = 79.99$ (4)°
$\beta = 85.74$ (4)°
$\gamma = 62.49$ (3)°
$V = 1097.0$ (4) Å^3
$Z = 2$
$D_x = 1.591$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 927 reflections
$\theta = 1.7$–26.5°
$\mu = 2.85$ mm^{-1}
$T = 273$ (2) K
Block, dark red
0.50 × 0.37 × 0.36 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.277$, $T_{max} = 0.359$
11547 measured reflections
4503 independent reflections
3916 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 26.5°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.083$
$S = 1.05$
4503 reflections
266 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.286P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.8128 (15)	O1—C1	1.311 (3)
Ni1—O2	1.8402 (15)	O2—C9	1.297 (2)
Ni1—N1	1.8460 (16)	N1—C7	1.307 (3)
Ni1—N3	2.0095 (16)	N1—N2	1.397 (2)
Br1—C4	1.902 (2)	N2—C9	1.303 (3)
Cl1—C13	1.739 (2)		
O1—Ni1—O2	178.96 (7)	O1—Ni1—N3	88.15 (7)
O1—Ni1—N1	94.99 (7)	O2—Ni1—N3	92.65 (7)
O2—Ni1—N1	84.19 (7)	N1—Ni1—N3	176.74 (7)

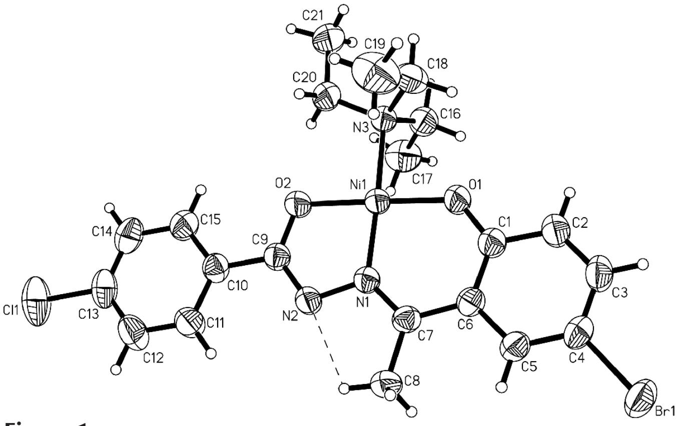


Figure 1
The molecular structure of (I), drawn with 50% probability displacement ellipsoids. The dashed line indicates the short intramolecular C—H···N contact.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*C*···N2	0.96	2.19	2.672 (3)	110

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_{iso}(H) = 1.2 or 1.5 times U_{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* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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