metal-organic papers

[1-(5-Chloro-2-oxidophenyl)ethanone 4-nitro-benzoylhydrazonato(2−)]tris(pyridine)nickel(II) pyridine solvate

Hapipah Ali,[a] Nur Ashikin Khamis,[a] W. Jefri Basirun[a] and Bohari M. Yamin[b]*

[a]Department of Chemistry, Universiti Malaya, Kuala Lumpur, Malaysia, and [b]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail:
bohari@pkrisc.cc.ukm.my

The double chelate ring in the title complex, [Ni($C_{15}H_{11}Cl$-N_3O_3)$_2$]·C_5H_5N, is essentially planar, with the *O*,*N*,*O*-donor atoms and a pyridine ligand N atom occupying the equatorial positions in a distorted octahedral environment about the Ni atom. The N—Ni—N axial bond angle is 173.16 (8)°.

Comment

The title complex, (I), was obtained on recrystallization of [Ni($C_{15}H_{11}N_3O_3Cl$)$_2$] from pyridine. Elemental analysis indicated the presence of four pyridine molecules and the loss of one of the ligands. The molecular structure of (I) is shown in Fig. 1 and selected bond distances and angles are given in Table 1.

(I)
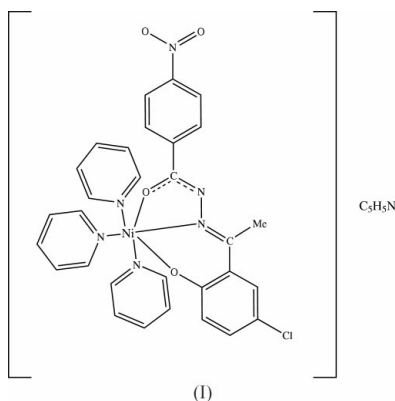
It can be seen that the Ni atom is chelated by the ligand in a tridentate manner *via* atoms O1, N1 and O2. The coordination geometry of atom Ni1 is distorted octahedral. Atoms N4 and N5 occupy the axial positions, with an angle of 173.16 (8)°. The equatorial atoms, O1, N1, O2 and N6, make *cis* angles at the Ni atom in the range 80.17 (8)–95.52 (8)°. The axial Ni—N4 and Ni—N5 bond lengths are both 2.176 (2) Å, slightly longer than the equatorial Ni—N6 and Ni—N1 bond distances of 2.096 (2) and 2.012 (2) Å, respectively. These distances are comparable
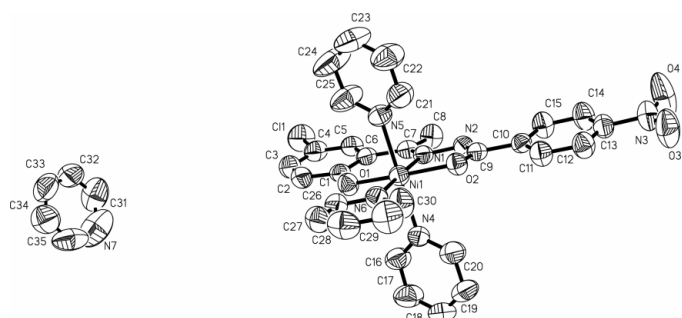
Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.
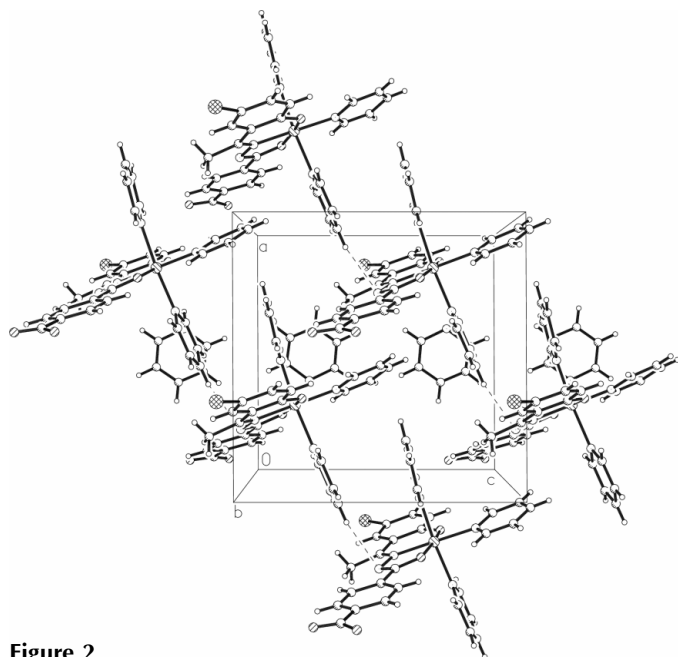
Figure 2
A packing diagram for (I), viewed down the *b* axis. Dashed lines denote C—H···N hydrogen bonds.

with those observed in the octahedral nickel complex $[Ni(C_{16}H_{24}N_{20})_2(N_3)_2]$ (You *et al.*, 2004). One pyridine molecule is present as an uncoordinated solvent molecule.

The structural dimensions of the tridentate ligand are typical for a Schiff base and are in the normal ranges (Allen *et al.*, 1987; Orpen *et al.*, 1989); they are in agreement with other octahedral nickel complexes, such as $[Ni(C_7H_7N_4O_2)]$ (Zhou *et al.*, 2004). The chelate double-ring fragment Ni1/O1/C6/C7/C8/N1/N2/C9/O2/N6 is essentially planar, with a maximum deviation from the mean plane of 0.083 (2) Å for atom N6. The dihedral angle between the C1–C6 and C10–C15 benzene rings is only 3.35 (13)°.

In the crystal structure of (I), symmetry-related molecules are linked by an intermolecular C18—H18*A*···N2[i] interaction (symmetry code as in Table 2), forming polymeric chains lying in the *ac* face, as shown in Fig. 2.

Experimental

The complex $[Ni(C_{15}H_{11}N_3O_3Cl)_2]$ was synthesized by the template condensation of 2-hydroxy-5-chloroacetophenone-4-nitrobenzhydrazide (0.3 g, 1.0 mmol) with nickel acetate dihydrate (0.21 g, 0.5 mmol), by refluxing and stirring in ethanol for 5 h. A pale-yellow solid was obtained and filtered off. The title complex, (I), was obtained by recrystallization of this product from pyridine.

Crystal data

$[Ni(C_{15}H_{11}ClN_3O_3)_2]\cdot C_5H_5N$	$D_x = 1.387$ Mg m^{-3}
$M_r = 706.82$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4938
$a = 12.585$ (2) Å	reflections
$b = 21.175$ (3) Å	$\theta = 1.8–27.5°$
$c = 12.704$ (2) Å	$\mu = 0.70$ mm^{-1}
$\beta = 90.211$ (3)°	$T = 273$ (2) K
$V = 3385.7$ (9) Å^3	Block, dark orange
$Z = 4$	$0.42 \times 0.23 \times 0.14$ mm

Data collection

Bruker SMART APEX area-detector diffractometer	7728 independent reflections
ω scans	5161 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.036$
(*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.5°$
$T_{min} = 0.757$, $T_{max} = 0.908$	$h = -16 \to 13$
26 253 measured reflections	$k = -27 \to 27$
	$l = -16 \to 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$+ 0.3467P]$
$wR(F^2) = 0.139$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
7728 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
433 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.9787 (18)	O2—C9	1.283 (3)
Ni1—N1	2.012 (2)	O3—N3	1.208 (3)
Ni1—O2	2.0357 (17)	O4—N3	1.200 (4)
Ni1—N6	2.096 (2)	N1—C7	1.302 (3)
Ni1—N4	2.176 (2)	N1—N2	1.398 (3)
Ni1—N5	2.176 (2)	N2—C9	1.316 (3)
O1—C1	1.297 (3)		
O1—Ni1—N1	90.39 (8)	O2—Ni1—N4	87.80 (8)
O1—Ni1—O2	170.49 (7)	N6—Ni1—N4	88.26 (9)
N1—Ni1—O2	80.17 (8)	O1—Ni1—N5	92.16 (8)
O1—Ni1—N6	93.93 (8)	N1—Ni1—N5	93.79 (8)
N1—Ni1—N6	175.66 (8)	O2—Ni1—N5	89.68 (8)
O2—Ni1—N6	95.52 (8)	N6—Ni1—N5	85.65 (9)
O1—Ni1—N4	91.37 (8)	N4—Ni1—N5	173.16 (8)
N1—Ni1—N4	92.04 (9)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18A···N2[i]	0.93	2.58	3.500 (4)	169

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

After their location in Fourier difference maps, all H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2 or $1.5U_{eq}$(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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