Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis[5-bromo-1*H*-indole-3-carbaldehyde (2-nitrobenzoyl)hydrazonato-κ^2N,O]bis(pyridine-κN)nickel(II) pyridine disolvate

Hapipah M. Ali, Siti Nadiah Abdul Halim and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.111
Data-to-parameter ratio = 17.7

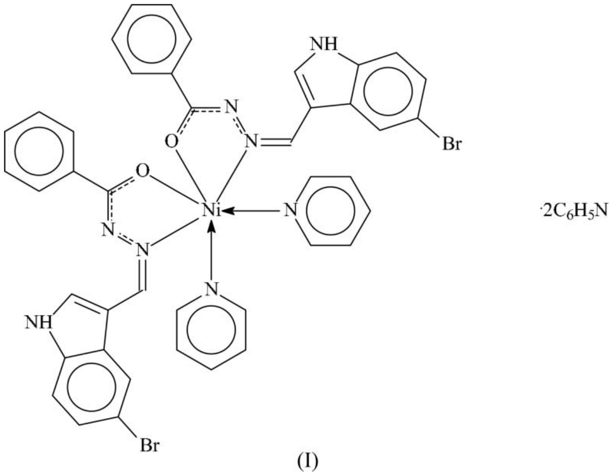
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Ni atom in the crystal structure of the title compound, $[\mathrm{Ni(C_5H_5N)_2(C_{16}H_{10}BrN_4O_3)_2}]\cdot 2\mathrm{C_5H_5N}$, lies on a special position of site symmetry 2 in an all-*cis* octahedral geometry.

Received 25 July 2005
Accepted 27 July 2005
Online 6 August 2005

Comment

A large number of transition metal derivatives of aromatic aldehyde-aroylhydrazones have been crystallographically authenticated; these have a hydroxy substituent in the aromatic aldehyde portion of the ligand that is able to bind covalently to the metal atom, *i.e.* these ligands function as monobasic *O*,*N*,*O*-terdentate chelates. Without the hydroxy subsituent, the hydrazone can bind as a monobasic bidentate ligand through the deprotonation of the nitrogen-bound H atom. However, there does not appear to be much interest in such metal complexes, as noted from the absence of such an entry in the Cambridge Structural Database (Version 5.26; Allen, 2002).



(I)

The Schiff base derived from 5-bromoindole-3-carbaldehyde and 2-nitrobenzhydrazine, whose structure is described in a previous study (Ali *et al.*, 2005), has been used to bind to nickel in the present study. Two deprotonated ligands chelate through their N and O atoms to the $\mathrm{Ni^{II}}$ atom; the compound crystallizes from pyridine as a bispyridine-coordinated complex along with two other uncoordinated pyridine molecules, (I) (Fig. 1). The NH group of the deprotonated ligand interacts with the unique uncoordinated pyridine molecule through a linear hydrogen bond [2.909 (3) Å].

Experimental

5-Bromoindole-3-carbaldehyde was reacted with 2-nitrobenzhydrazide to give the Schiff base (Ali *et al.*, 2005). Aqueous sodium hydroxide was added to an ethanol solution of the Schiff base (0.50 g, 1.30 mmol) to give a pH of 8.5. Nickel(II) acetate (0.16 g, 0.65 mmol) was added to the solution (approximately 25 ml) and the mixture was heated for 5 h. The solvent was removed to give the crude product, which was then purified by recrystallization from pyridine.

Crystal data

$[Ni(C_5H_5N)_2(C_{16}H_{10}BrN_4O_3)_2]\cdot 2C_5H_5N$
$M_r = 1147.49$
Monoclinic, $C2/c$
$a = 19.369$ (3) Å
$b = 15.515$ (2) Å
$c = 17.246$ (3) Å
$\beta = 93.72$ (1)°
$V = 5171.7$ (14) Å^3
$Z = 4$
$D_x = 1.474$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16510 reflections
$\theta = 3.1$–27.5°
$\mu = 1.98$ mm^{-1}
$T = 295$ (2) K
Block, red
0.37 × 0.25 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.334$, $T_{\max} = 0.729$
24781 measured reflections
5907 independent reflections
4302 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 27.5°$
$h = -25 \rightarrow 25$
$k = -20 \rightarrow 20$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.05$
5907 reflections
334 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.7544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.042 (1)	Ni1—N5	2.113 (2)
Ni1—N3	2.061 (2)		
O1—Ni1—O1$^{\rm i}$	170.7 (1)	N3—Ni1—N3$^{\rm i}$	90.6 (1)
O1—Ni1—N3	77.9 (1)	N3—Ni1—N5	91.8 (1)
O1—Ni1—N3$^{\rm i}$	95.4 (1)	N3—Ni1—N5$^{\rm i}$	170.3 (1)
O1—Ni1—N5	94.3 (1)	N5—Ni1—N5$^{\rm i}$	87.42 (6)
O1—Ni1—N5$^{\rm i}$	92.5 (1)		

Symmetry code: (i) $-x + 1, y, -z + \frac{3}{2}$.

The carbon-bound H atoms were positioned geometrically (C—H = 0.93 Å) and they were included in the refinement in the riding-model approximation, with $U_{\rm iso}$(H) values set at 1.2 times $U_{\rm eq}$ of the parent atoms. The amino H atom was located in a difference Fourier map, and was refined with a distance restraint of N—H = 0.85 (1) Å.

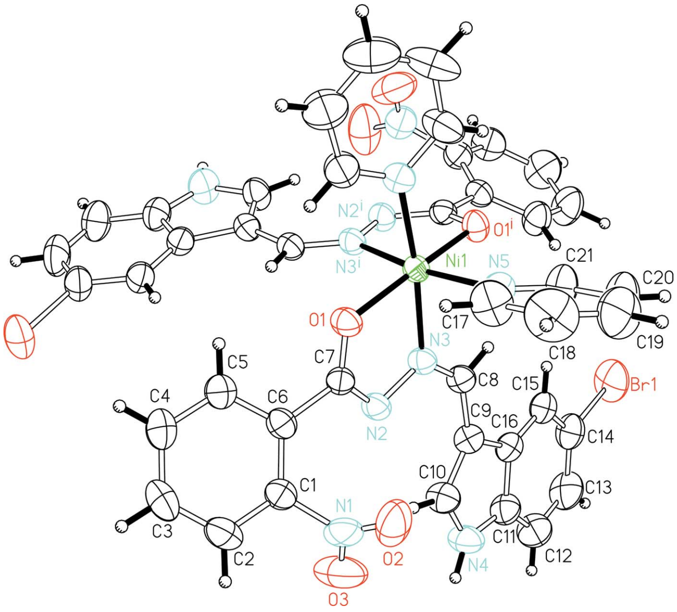


Figure 1
ORTEPII plot (Johnson, 1976) of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$]. The uncoordinated solvent molecules are not shown.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors thank the Ministry of Science, Technology and the Environment for supporting this study (grant No. IPRA 33–02-03–3055). We thank Professor Gao Shan of Heilongjiang University for the diffraction measurements.

References

Ali, H. M., Abdul Halim, S. N. & Ng, S. W. (2005). *Acta Cryst.* E**61**, o2308–o2309.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., 9009 New Trails Drive,] The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Acta Cryst. (2005). E**61**, m1682–m1683 Ali *et al.* • $[Ni(C_5H_5N)_2(C_{16}H_{10}BrN_4O_3)_2]\cdot 2C_5H_5N$ **m1683**