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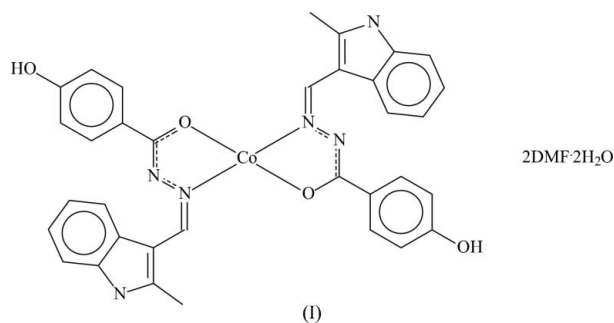
## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.038  
 $wR$  factor = 0.107  
Data-to-parameter ratio = 16.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Bis(2-methyl-1*H*-indole-3-carbaldehyde 4-hydroxy-  
benzoylhydrazonato- $\kappa^2N,O$ )nickel(II) *N,N*-dimethyl-  
formamide disolvate dihydrateThe title compound,  $[\text{Ni}(\text{C}_{17}\text{H}_{14}\text{N}_3\text{O}_2)_2] \cdot 2\text{C}_3\text{H}_7\text{NO} \cdot 2\text{H}_2\text{O}$ , contains  $\text{Ni}^{\text{II}}$  cations (site symmetry  $\bar{1}$ ) chelated by two deprotonated Schiff bases in a *trans*- $\text{NiN}_2\text{O}_2$  square-planar geometry. The complex molecule and the dimethylformamide (DMF) and water molecules interact by way of  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{N}-\text{H} \cdots \text{O}$  hydrogen bonds, resulting in a layered structure.

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## Comment

This report continues on from our previous study on nickel derivatives of indole-3-carbaldehyde arylhydrazone (Ali *et al.*, 2005). The title compound, (I), has the Ni atom (site symmetry  $\bar{1}$ ) in a square-planar geometry (Table 1 and Fig. 1). The dimethylformamide solvent used in recrystallization is incorporated into the crystal structure; however, it does not participate in bonding with the metal but interacts instead with the water molecule. Together, the constituent molecules interact by way of  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{N}-\text{H} \cdots \text{O}$  hydrogen bonds (Table 2), resulting in a sheet structure.

## Experimental

The Schiff base was synthesized from 2-methylindole-3-carbaldehyde (0.44 g, 2.7 mmol) and 4-hydroxybenzhydrazide (0.42 g, 2.7 mmol) in ethanol (100 ml). The reactants were heated under reflux for 2 h. The solvent was removed to give the organic compound. This compound (0.18 g, 0.6 mmol) and nickel(II) acetate dihydrate (0.08 g, 0.3 mmol) were heated in ethanol (100 ml) for 5 h. The complex was obtained as a red powder which was recrystallized from dimethylformamide, yielding crystals of (I).

## Crystal data

 $[\text{Ni}(\text{C}_{17}\text{H}_{14}\text{N}_3\text{O}_2)_2] \cdot 2\text{C}_3\text{H}_7\text{NO} \cdot 2\text{H}_2\text{O}$  $M_r = 825.56$ Triclinic,  $P\bar{1}$  $a = 7.2441$  (6) Å $b = 12.326$  (1) Å $c = 12.395$  (1) Å $\alpha = 76.336$  (1)° $\beta = 74.120$  (1)° $\gamma = 86.253$  (1)° $V = 1034.5$  (2) Å<sup>3</sup> $Z = 1$  $D_x = 1.325$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation $\mu = 0.53$  mm<sup>-1</sup> $T = 295$  (2) K

Irregular block, red

 $0.45 \times 0.35 \times 0.15$  mm

Data collection

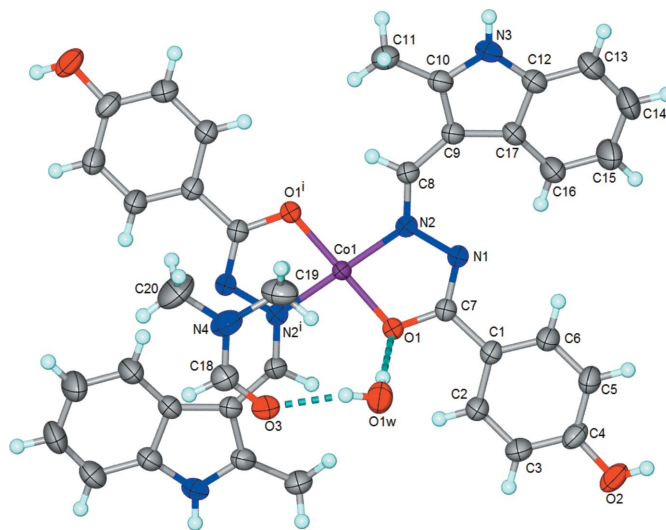
Bruker APEX-II CCD diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.801, T_{\max} = 0.925$

9651 measured reflections  
 4495 independent reflections  
 3822 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.017$   
 $\theta_{\text{max}} = 27.1^\circ$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.107$   
 $S = 1.02$   
 4495 reflections  
 278 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.185P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{Å}^{-3}$



**Figure 1**  
 The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). The dashed lines represent hydrogen bonds. [Symmetry code: (i)  $1 - x, 1 - y, 1 - z$ .]

**Table 1**

Selected geometric parameters (Å, °).

Ni1—O1	1.833 (1)	Ni1—N2	1.862 (1)
O1—Ni1—N2	83.52 (6)	O1—Ni1—N2 <sup>i</sup>	96.48 (6)

Symmetry code: (i)  $-x + 1, -y + 1, -z + 1$ .

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 <sup>o</sup> ···O1w <sup>ii</sup>	0.85 (1)	1.85 (1)	2.688 (2)	171 (3)
O1w—H1w1···O1	0.84 (1)	2.02 (1)	2.852 (2)	173 (3)
O1w—H1w2···O3	0.84 (1)	1.94 (1)	2.754 (2)	163 (3)
N3—H3 <sup>n</sup> ···O3 <sup>iii</sup>	0.86 (1)	2.00 (1)	2.859 (2)	173 (2)

Symmetry codes: (ii)  $-x + 2, -y + 1, -z$ ; (iii)  $x, y + 1, z$ .

The carbon-bound H atoms were placed at calculated positions ( $C-H = 0.93-0.96 \text{ Å}$ ) and refined as riding, with  $U_{\text{iso}}(H) = 1.2$  or 1.5 times  $U_{\text{eq}}(C)$ . The methyl group was rotated to best fit the electron density. The water, hydroxy and amino H atoms were located in a difference Fourier map and were refined with distance restraints [ $O-H = N-H = 0.85 (1) \text{ Å}$ ]; the  $U_{\text{iso}}$  values were freely refined.

Data collection: APEXII (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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References

Ali, H. M., Abdul Halim, S. N. & Ng, S. W. (2005). *Acta Cryst.* **E61**, m1682–m1683.  
 Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
 Bruker (2004). APEXII (Version 7.23A) and SAINT (Version 7.23A). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.