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Dichloro(1,10-phenanthroline)(benzene-1,2-diamine)nickel(II) methanol solvate

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Key indicators

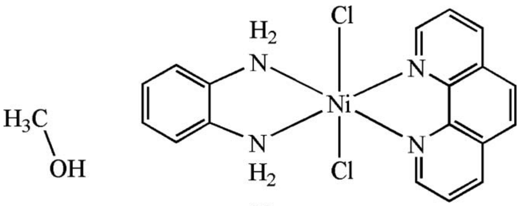
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.041
wR factor = 0.119
Data-to-parameter ratio = 13.6

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

The complex molecule of the title compound, $[NiCl_2(C_6H_8N_2)(C_{12}H_8N_2)]\cdot CH_4O$, has twofold rotation symmetry. The complex has a distorted octahedral $NiCl_2N_4$ coordination. Hydrogen-bonding and π–π stacking interactions help to stabilize the crystal structure.

Comment

The use of diamine complexes as hydrogenation catalysts or precatalysts has been reported previously (Noyori *et al.*, 2001; Haack *et al.*, 1997; Ohkuma *et al.*, 2002; Hedberg *et al.*, 2005; Ito *et al.*, 2001). In order to investigate the application of diamine complexes of Ni^{II} as hydrogenation catalysts, we have prepared the title complex, (I), and report here its structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule has a twofold rotation symmetry; the Ni^{II} ion and midpoints of the C3—C3A and C9—C9A bonds [symmetry code: (A) $2 - x, y, \frac{1}{2} - z$] are located on a twofold axis. The Ni^{II} ion is coordinated by two Cl^- anions and four N atoms in a distorted octahedral geometry (Table 1). The parallel phenanthroline rings of neighboring complexes are partially overlapped with a face-to-face separation of 3.463 (3) Å, indicating the existence of π–π stacking. Hydrogen-bonding and π–π stacking interactions (Table 2) help to stabilize the crystal structure (Fig. 2).

Experimental

$NiCl_2\cdot 6H_2O$ (1 mmol), 1,10-phenanthroline (1 mmol) and 1,2-phenylenediamine (1 mmol) were dissolved in methanol (12 ml). The solution was transferred into a 23 ml Teflon-lined autoclave and heated at 433 K for 80 h. The autoclave was then cooled over a period of 30 h to give purple single crystals of (I).

Crystal data

$[NiCl_2(C_6H_8N_2)(C_{12}H_8N_2)]\cdot CH_4O$
M_r = 450.00
Monoclinic, $C2/c$
a = 13.898 (4) Å
b = 18.246 (5) Å
c = 10.015 (3) Å
β = 126.313 (3)°
V = 2046.4 (10) Å^3
Z = 4
D_x = 1.461 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.23 mm^{-1}
T = 298 (2) K
Block, purple
0.26 × 0.22 × 0.17 mm

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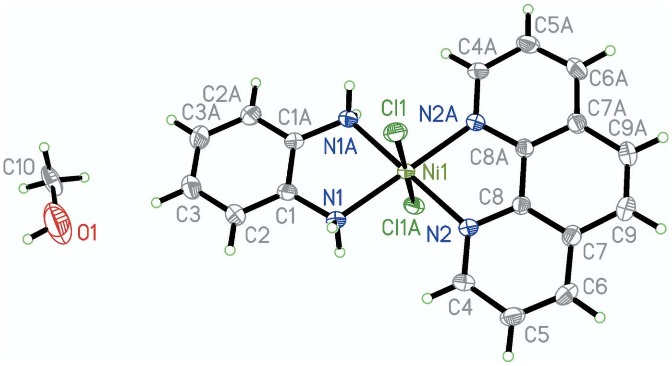

Figure 1
The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (A) $2 - x, y, \frac{1}{2} - z$].

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.741$, $T_{\max} = 0.819$
5191 measured reflections
1794 independent reflections
1204 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.034$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.119$
$S = 1.07$
1794 reflections
132 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 1.4849P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—N1	2.080 (3)	Ni1—Cl1	2.4671 (12)
Ni1—N2	2.077 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1$^{\text{i}}$	0.90	2.49	3.267 (3)	144
O1—H1···Cl1$^{\text{ii}}$	0.82	2.36	3.168 (10)	171

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

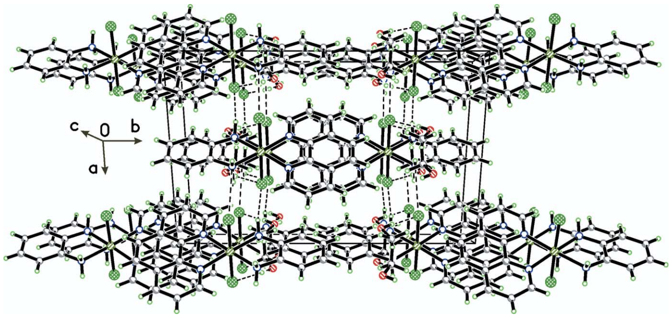

Figure 2
A packing diagram, with dashed lines indicating hydrogen bonds.

The methanol solvent molecule is disordered over a twofold axis and its occupancy was fixed at 0.5 for the unique location. H atoms of the methanol molecule were located in a difference Fourier map and refined as riding with O—H = 0.82, C—H = 0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O,C})$. Other H atoms were placed in calculated positions, with N—H = 0.90 Å and C—H = 0.93 Å, and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N,C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* (Version 5.01), *SAINT* (Versions 5.01) and *SADABS* (Version 2.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Haack, K.-J., Hashiguchi, S., Fujii, A., Ikariya, T. & Noyori, R. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 285–288.
Hedberg, C., Källström, K., Arvidsson, P. I., Brandt, P. & Andersson, P. G. (2005). *J. Am. Chem. Soc.* **127**, 15083–15090.
Ito, M., Hirakawa, M., Murata, K. & Ikaraya, T. (2001). *Organometallics*, **20**, 379–381.
Noyori, R., Yamakawa, M. & Hashiguchi, S. (2001). *J. Org. Chem.* **66**, 7931–7944.
Ohkuma, T., Koizumo, M., Muniz, K., Hilt, G., Kabuto, C. & Noyori, R. (2002). *J. Am. Chem. Soc.* **124**, 6508–6509.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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