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Bis[(imidazol-4-ylmethyl)(2-pyridylmethyl)-amine-κ^3N,N',N'']nickel(II) dichloride dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.085
Data-to-parameter ratio = 15.8

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

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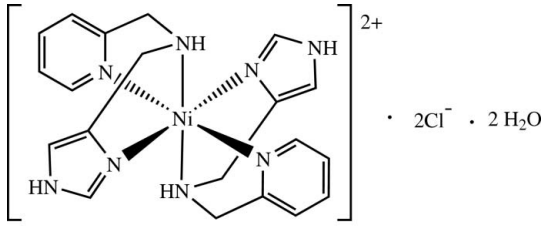
The crystal structure of the title compound, $[Ni(C_{20}H_{24}N_8)]Cl_2\cdot 2H_2O$, consists of Ni^{II} complex cations, Cl^- anions and uncoordinated water molecules. The Ni^{II} ion is located on an inversion center and is chelated by two (imidazol-4-ylmethyl)(2-pyridylmethyl)amine (IPA) ligands in a distorted octahedral coordination. The tridentate IPA ligand displays the facial configuration. Hydrogen bonding stabilizes the crystal structure.

Comment

Imidazole is an important ligand in mimicking the histidine binding mode in metalloproteins (Kaim & Schwederski, 1994). In the past decade, mixed ligands of pyridyl and imidazolyl groups have attracted much attention (Ohtsu *et al.*, 2001). In the course of studying model compounds for histine-coordinated metalloproteins, we have synthesized an amine compound incorporating both pyridyl and imidazolyl groups, *viz*. (imidazol-4-ylmethyl)(2-pyridylmethyl)amine (IPA). IPA reacts readily with various metal salts to give the corresponding complexes. We report here the structure of the title IPA complex of Ni^{II}, (I).



(I)

The crystal structure of (I) consists of discrete Ni^{II} complex cations, Cl^- anions and uncoordinated water molecules (Fig. 1). The Ni^{II} ion is located on an inversion center and is chelated by two IPA ligands in a distorted octahedral coordination (Table 1). The three independent Ni—N bond distances are significantly different. The Ni—N2 bond is much longer than the Ni—N1 and Ni—N3 bonds.

The tridentate IPA ligand displays the facial coordination configuration. The two chelating five-membered rings have an envelope conformation, with atoms N2 and C7 lying at the flap positions and deviating from the mean planes formed by the other four atoms by 0.462 (3) (for the N1-containing ring) and 0.382 (3) Å (for the N3-containing ring), respectively. The most relevant example of an Ni^{II} complex with a multidentate ligand containing both pyridyl and imidazolyl groups is $[Ni(HL)(hfac)(H_2O)][hfac]$ (HL is imidazolylmethylidene-aminoethylpyridine and hfac is hexafluoroacetylacetonate)

(Colacio *et al.*, 2000). In that complex, the chelating tridentate ligand displays a meridional coordination.

Extensive hydrogen bonding occurs in the crystal structure of (I) (Table 2), providing stability.

Experimental

IPA was prepared by a condensation reaction, mixing equimolar imidazole-4-carbaldehyde and 2-aminomethylpyridine in ethanol at room temperature followed by hydrogenation with excess sodium borohydride. Crystals of (I) were obtained by a diffusion reaction between layers of reactants. An aqueous solution (2 ml) of $NiCl_2$ (20 mg) was layered with THF (2 ml) and then another layer of a methanol solution (2 ml) of excess IPA (30 mg). After a few days, the boundaries between the layers disappeared. Single crystals of (I) were produced after one week.

Crystal data

$[Ni(C_{10}H_{12}N_4)_2]Cl_2\cdot 2H_2O$
$M_r = 542.12$
Monoclinic, $P2_1/c$
$a = 8.7738\ (18)$ Å
$b = 19.066\ (4)$ Å
$c = 7.9456\ (16)$ Å
$\beta = 114.09\ (3)^\circ$
$V = 1213.4\ (5)$ Å^3
$Z = 2$
$D_x = 1.484$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.05$ mm^{-1}
$T = 298\ (2)$ K
Prism, purple
0.58 × 0.42 × 0.36 mm

Data collection

Rigaku AFC-7S diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.657$, $T_{max} = 0.689$
2533 measured reflections
2381 independent reflections
1928 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.0^\circ$
3 standard reflections
every 150 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.085$
$S = 1.01$
2381 reflections
151 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.5253P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—N1	2.0960 (18)	Ni—N3	2.0657 (19)
Ni—N2	2.140 (2)		
N1—Ni—N2	80.29 (7)	N2—Ni—N3	82.66 (7)
N1—Ni—N3	86.03 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1W···Cl^{i}	0.91	2.29	3.194 (2)	170
O1—H2W···Cl^{ii}	0.92	2.33	3.213 (3)	159
N2—H2N···Cl	0.91	2.37	3.262 (2)	167
N4—H4N···O1	0.86	1.96	2.814 (4)	170

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

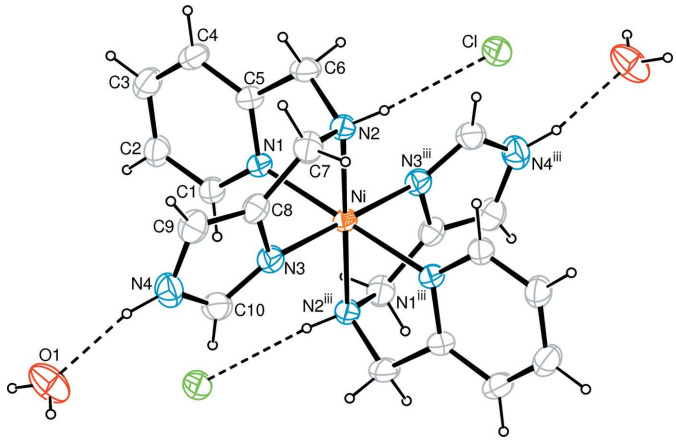


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (iii) $1-x, 1-y, 1-z$]. Dashed lines indicate hydrogen bonds.

Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Other H atoms were placed in calculated positions, with N—H = 0.86 (imidazole) or 0.91 Å (amine), C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(N,C)$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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