Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

cis-Diaquabis[2-(hydroxymethyl)pyridine]-nickel(II) dichloride

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Key indicators

Single-crystal X-ray study
T = 133 K
Mean σ(C–C) = 0.002 Å
R factor = 0.019
wR factor = 0.055
Data-to-parameter ratio = 21.5

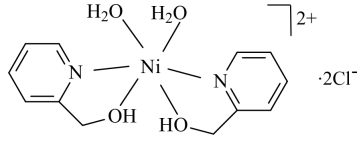
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 5 November 2003
Accepted 21 November 2003
Online 6 December 2003

The title complex, $[Ni(mpy)_2(H_2O)_2]Cl_2$ [mpy is 2-(hydroxymethyl)pyridine, C_6H_7NO], consists of two chloride anions and an $[Ni(mpy)_2(H_2O)_2]^{2+}$ cation, in which the Ni^{II} ion lies on a twofold axis and exhibits octahedral geometry, coordinated by two neutral mpy ligands and two water molecules. Mpy acts as an *N*,*O*-bidentate ligand, giving rise to a five-membered chelate ring. The $Ni-N_{mpy}$, $Ni-O_{mpy}$ and $Ni-O_{water}$ bond distances are 2.0629 (8), 2.0573 (7) and 2.0490 (7) Å, respectively. The H atoms of the water molecules and the hydroxy group of mpy form $O-H\cdots Cl$ hydrogen bonds, resulting in a three-dimensional network.

Comment

This work is a part of our study of the synthesis and structural characterization of metal complexes with 2-hydroxyalkylpyridines. We report here the crystal structure of the aqua complex of nickel(II) with 2-methanolpyridine (mpy), (I).



(I)

The structure of (I) consists of a complex cation, $[Ni(mpy)_2(H_2O)_2]^{2+}$ [mpy is 2-(hydroxymethyl)pyridine, C_6H_7NO], and two symmetry-equivalent Cl^- anions. In the cation, the nickel(II) ion lies on a twofold axis and is octahedrally coordinated by two neutral mpy ligands and two water molecules, forming a NiN_2O_6 core (Fig. 1). Each mpy ligand behaves as a bidentate donor *via* the pyridine N and hydroxy O atoms, forming a five-membered chelate ring with the nickel(II) ion. The coordination of two mpy and two water ligands results in a *cis* configuration.

The $Ni-N_{mpy}$, $Ni-O_{mpy}$ and $Ni-O_{water}$ bond distances are almost identical, and the $Ni-N_{mpy}$ and $Ni-O_{mpy}$ bond distances are comparable to those reported for $[Ni(sac)_2(mpy)_2]$ (sac is saccharinate; Yilmaz *et al.*, 2002). Some distortion from regular octahedral geometry is apparent (Table 1), especially the bite angle of the mpy ligand (O1—Ni—N1) and the *trans* N1—Ni—$N1^i$ angle (see Table 1 for symmetry code).

The mpy ligand is essentially planar [the deviations of atoms O1 (hydroxy) and C7 (methylene) from the least-squares pyridine ring plane are 0.007 (1) and 0.025 (1) Å, respectively]. The two mpy ligands containing atoms N1 and $N1^i$ attached to Ni are almost perpendicular to one another [the dihedral angle between the pyridine rings is 86.75 (2)°]. The

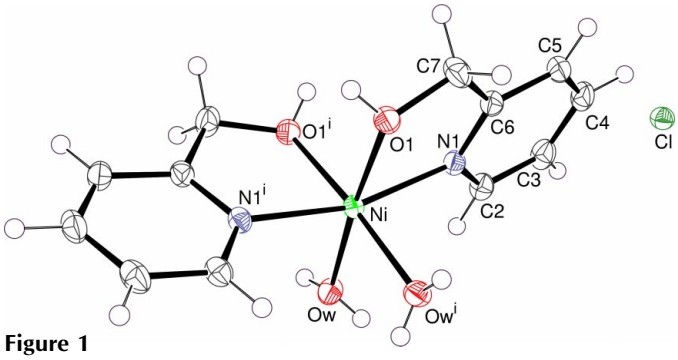


Figure 1
Molecular view of (I) (50% displacement ellipsoids). [Symmetry code: (i) $-x, y, \frac{1}{2}-z$.]

geometry of the mpy ligand is similar to that of the same species in $[Ni(sac)_2(mpy)_2]$ (Yilmaz *et al.*, 2002). The packing of (I) is shown in Fig. 2. The crystal structure exhibits three strong O—H···Cl intermolecular hydrogen bonds (Table 2) between the H atoms of the water molecules and the hydroxy group of mpy and the chloride ions. Additionally, one of the H atoms of the pyridine ring is also involved in a C—H···O interaction with the water O atom. The overall hydrogen-bond scheme can be described as a three-dimensional network formed by the mpy ligands, water molecules and Cl^- anions.

Experimental

The starting tetraaquabis(saccharinato)nickel(II) dihydrate, $[Ni(H_2O)_4(sac)_2]\cdot 2H_2O$, was prepared according to the method of Haider *et al.* (1985). A methanol solution (25 ml) of $[Ni(H_2O)_4(sac)_2]\cdot 2H_2O$ (0.53 g, 1 mmol) was mixed with mpy (0.22 g, 2 mmol) and HCl (0.07 g, 2 mmol) at 333 K. Crystals suitable for X-ray diffraction were grown by slow diffusion of ether into the reaction solution at room temperature.

Crystal data

$[Ni(C_6H_7NO)_2(H_2O)_2]Cl_2$
$M_r = 383.89$
Monoclinic, $C2/c$
$a = 20.0943\ (18)$ Å
$b = 7.6090\ (6)$ Å
$c = 12.1268\ (10)$ Å
$\beta = 121.708\ (3)^\circ$
$V = 1577.4\ (2)$ Å^3
$Z = 4$
$D_x = 1.617$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4680 reflections
$\theta = 2.9$–30.5°
$\mu = 1.58$ mm^{-1}
$T = 133\ (2)$ K
Prism, blue
0.33 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.643$, $T_{max} = 0.729$
12 845 measured reflections
2318 independent reflections
2177 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 30.0^\circ$
$h = -28 \rightarrow 28$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.055$
$S = 1.06$
2318 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.588P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

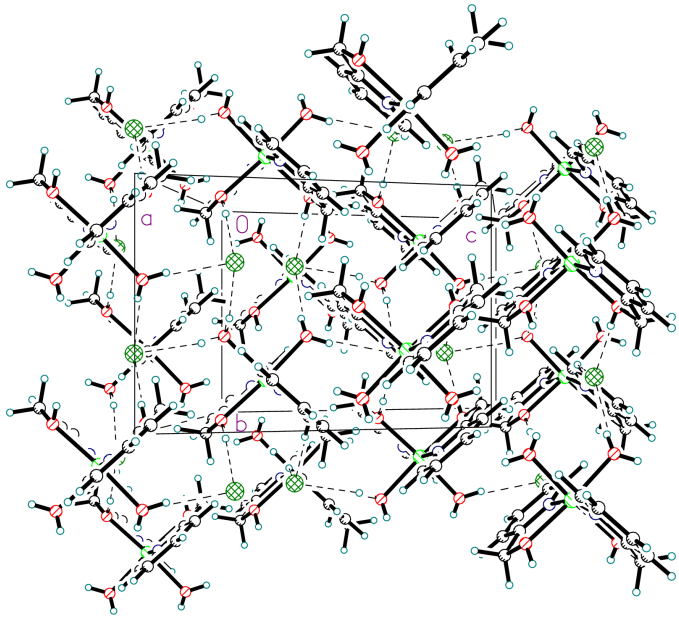


Figure 2
Packing diagram for (I).

Table 1
Selected geometric parameters (Å, °).

Ni—O*W*	2.0490 (7)	Ni—N1	2.0629 (8)
Ni—O1	2.0573 (7)		
OW—Ni—*OW*^{i}	87.55 (4)	*OW*^{i}—Ni—N1	92.31 (3)
OW—Ni—O1	175.60 (3)	O1—Ni—N1	79.23 (3)
OW^{i}—Ni—O1	88.56 (3)	O1^{i}—Ni—N1	90.33 (3)
O1—Ni—O1^{i}	95.40 (4)	N1—Ni—N1^{i}	164.54 (5)
OW—Ni—N1	98.86 (3)		

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···*OW*^{ii}	0.95	2.64	3.4931 (13)	149
O1—H1···Cl^{iii}	0.776 (17)	2.236 (17)	3.0097 (8)	176 (17)
OW—H*W*1···Cl^{iv}	0.819 (17)	2.315 (18)	3.1284 (8)	172 (15)
OW—H*W*2···Cl^{v}	0.746 (18)	2.345 (18)	3.0831 (8)	170 (17)

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

H atoms bonded to C atoms were included as riding [C—H = 0.95 or 0.99 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$], while the hydroxy H atoms were refined freely [O—H = 0.746 (18)–0.819 (17) Å].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

VTY thanks DAAD for financial support given for the research visit to the Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Germany.

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