Crystal structure of (N^1E,N^2E)-N^1,N^2-bis(5-hydroxymethyl-2-methyl-3-oxopyridinio-4-ylmethylene)ethane-1,2-diamine-nickel(II) dihydrochloride, $[Ni(C_{18}H_{22}N_4O_4)]Cl_2$

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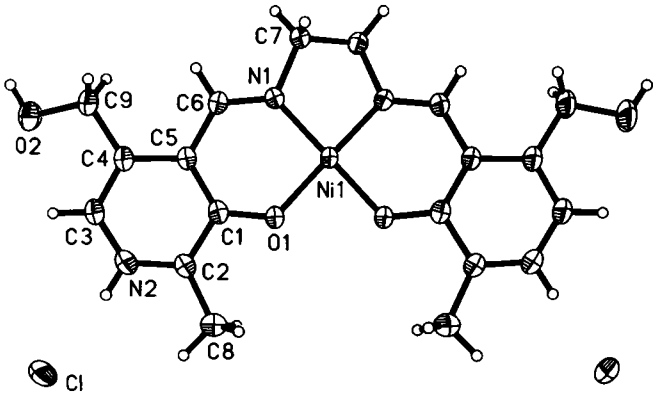

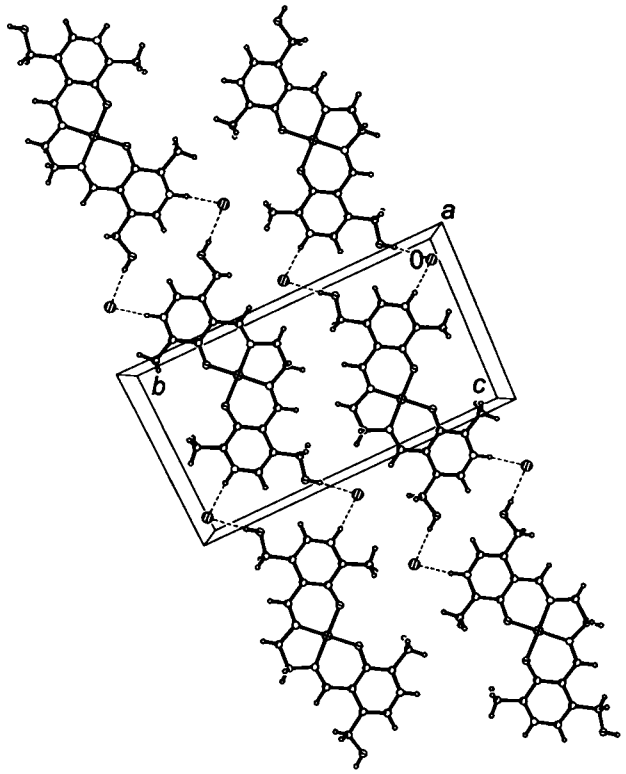

Abstract

$C_{18}H_{22}Cl_2N_4NiO_4$, monoclinic, $C12/c1$ (no. 15), a = 10.4852(3) Å, b = 18.6442(4) Å, c = 10.8653(3) Å, β = 111.458(1)°, V = 1976.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.033, $wR_{ref}(F^2)$ = 0.076, T = 296 K.

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Source of material

All reagents used were of analytical grade from commercial sources. Pyridoxal hydrochloride (0.2 mmol, 40.7 mg) and 1,2-diaminoethane (0.1 mmol, 6.0 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min to give a clear yellow solution. The solution was added a methanol solution (10 ml) of $NiCl_2 \cdot 6H_2O$ (0.1 mmol, 23.8 mg) with stirring. After keeping the resulting solution at room temperature in air for 7 d, red needle-like crystals were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed three times with methanol and dried in a vacuum desiccator over anhydrous $CaCl_2$ (yield 85.6 %).

Elemental analysis – found: C 44.1 %, H 4.6 %, N 11.6 %; calc. for $C_{18}H_{22}N_4O_4Cl_2Ni$: C 44.3 %, H 4.5 %, N 11.5 %.

Discussion

Transition metal complexes containing Schiff base ligands have been of great interest for many years [1]. These complexes play an important role in the coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures. Some of nickel(II) complexes with Schiff base ligands have pharmacological and catalytic properties [2]. In order to further develop the coordination chemistry of such nickel complexes, a new Schiff base compound by nickel(II) chloride and a Schiff base ligand from 1,2-diaminoethane and pyridoxal hydrochloride is reported here.

The Ni atom is coordinated by two O atoms and two N atoms from the bis-Schiff base ligands (figure, top). The Ni atom lies on the center of this NiO_2N_2 polyhedron with a slightly distorted square-planar environment. The dihedral angles of the C7/N1/Ni/N1A/C7A ring with the C1/O1/Ni/N1/C6/C5 and C1A/O1A/Ni/N1A/C6A/C5A rings are 1.6(2)° and 8.0(3)°, respectively. Whereas the C1/O1/Ni/N1/C6/C5 ring is almost coplanar with the C1/C2/N2/C3/C4/C5 ring (dihedral angle of 0.3(2)°), and that is also uniform between the C1A/O1A/Ni/N1A/C6A/C5A ring and C1A/C2A/N2A/C3A/C4A/C5A ring. The Ni—O bond length of 1.839(2) Å and the Ni—N bond distance of 1.843(2) Å are similar to the values observed in other Schiff base nickel(II) complexes [3-4]. As expected, the molecule adopts two *trans* configuration about the C6=N1 and C6A=N1A bonds. Their bond length, 1.293(3) Å, conforms to the expected value for a normal C=N π bond [5]. As shown in the figure, bottom, each complex cation is linked with two Cl^- anions through the N–H···Cl hydrogen bonds, and also linked with other two Cl^- anions through intermolecular O–H···Cl hydrogen bonds in the crystal structure, forming a one-dimensional chain structure. The chains are stacked to each other to form a layered structure by C–H···Cl and π-π intermolecular forces.

Table 1. Data collection and handling.

Crystal:	red needle, size 0.10 × 0.12 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.86 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8874, 1770
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1438
$N(param)_{refined}$:	132
Programs:	SHELXS-97 [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(3A)	8*f*	0.7084	0.2258	0.3676	0.044
H(7A)	8*f*	0.6441	0.4459	–0.1884	0.043
H(7B)	8*f*	0.5380	0.4729	–0.1269	0.043
H(8A)	8*f*	0.5802	0.0537	0.0764	0.067
H(8B)	8*f*	0.6178	0.0818	–0.0420	0.067
H(8C)	8*f*	0.4673	0.0891	–0.0462	0.067
H(9A)	8*f*	0.7759	0.3812	0.2814	0.061
H(9B)	8*f*	0.6203	0.3932	0.2579	0.061
H(6A)	8*f*	0.6212	0.4053	0.0566	0.080
H(2A)	8*f*	0.7402	0.3791	0.4716	0.080
H(2B)	8*f*	0.6586	0.1227	0.2526	0.080

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	4*e*	½	0.29257(2)	–¼	0.0413(3)	0.0231(2)	0.0156(2)	0	0.0052(2)	0
Cl	8*f*	0.74374(8)	0.03340(3)	0.38102(6)	0.0867(5)	0.0344(4)	0.0351(4)	0.0088(3)	0.0246(3)	0.0108(3)
O(1)	8*f*	0.5404(2)	0.21998(8)	–0.1271(1)	0.057(1)	0.0252(8)	0.0169(8)	0.0002(7)	0.0069(7)	0.0008(6)
O(2)	8*f*	0.7222(3)	0.3437(1)	0.4238(2)	0.180(3)	0.048(1)	0.023(1)	–0.022(1)	0.029(1)	–0.0054(9)
N(1)	8*f*	0.5552(2)	0.36449(9)	–0.1252(2)	0.042(1)	0.023(1)	0.019(1)	–0.0004(8)	0.0072(8)	0.0017(7)
N(2)	8*f*	0.6431(2)	0.1659(1)	0.2053(2)	0.048(1)	0.030(1)	0.026(1)	0.0028(9)	0.0124(9)	0.0064(8)
C(1)	8*f*	0.5810(2)	0.2268(1)	0.0010(2)	0.034(1)	0.031(1)	0.022(1)	0.002(1)	0.008(1)	0.0018(9)
C(2)	8*f*	0.5962(2)	0.1617(1)	0.0746(2)	0.040(1)	0.029(1)	0.023(1)	0.003(1)	0.009(1)	0.0048(9)
C(3)	8*f*	0.6755(3)	0.2273(1)	0.2757(2)	0.052(2)	0.037(1)	0.017(1)	–0.003(1)	0.008(1)	0.0025(9)
C(4)	8*f*	0.6599(2)	0.2913(1)	0.2119(2)	0.043(1)	0.033(1)	0.022(1)	–0.002(1)	0.009(1)	0.0014(9)
C(5)	8*f*	0.6121(2)	0.2916(1)	0.0714(2)	0.037(1)	0.027(1)	0.020(1)	–0.001(1)	0.0070(9)	0.0014(9)
C(6)	8*f*	0.5988(2)	0.3587(1)	0.0021(2)	0.045(1)	0.030(1)	0.022(1)	–0.003(1)	0.008(1)	–0.0014(9)
C(7)	8*f*	0.5558(2)	0.4363(1)	–0.1820(2)	0.058(2)	0.023(1)	0.021(1)	–0.004(1)	0.008(1)	0.0002(9)
C(8)	8*f*	0.5623(3)	0.0903(1)	0.0100(2)	0.065(2)	0.029(1)	0.036(1)	0.004(1)	0.012(1)	0.002(1)
C(9)	8*f*	0.6957(3)	0.3595(1)	0.2905(2)	0.086(2)	0.041(2)	0.016(1)	–0.012(1)	0.009(1)	–0.002(1)

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