metal-organic papers

Aquadichloro(2,9-dimethyl-1,10-phenanthroline-κ^2N,N')nickel(II)

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In the title compound, [NiCl₂(C₁₄H₁₂N₂)(H₂O)], there are two molecules in the asymmetric unit. Each Ni atom is five-coordinate in a geometry between trigonal–bipyramidal and tetragonal–pyrimidal. Molecules are linked into a three-dimensional framework by O—H···Cl and C—H···Cl hydrogen bonds. The packing is further stabilized by π–π interactions between the phenanthroline ring systems.

Comment

We have recently reported the structure of aquadichloro(2,9-dimethyl-1,10-phenanthroline-κ^2N,N)copper(II) (Ding *et al.*, 2006). In our ongoing studies, the title compound, (I), was obtained by the reaction of 2,9-dimethyl-1,10-phenanthroline and NiCl₂.

(I)
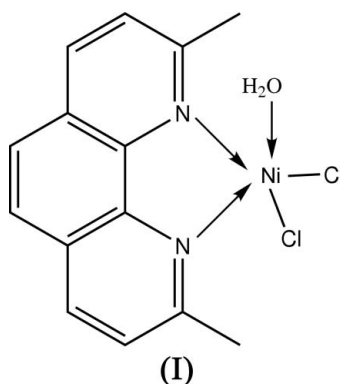
The asymmetric unit of (I) contains two crystallographically independent molecules (Fig. 1). The corresponding bond lengths and angles of these two molecules agree at a level of two standard deviations (Table 1). Each Niᴵᴵ atom is five-coordinated by two N atoms from one 9,10-dimethyl-phennathroline ligand, one O atom from a water molecule and two Cl atoms. This NiON₂Cl₂ unit adopts a geometry between trigonal–bipyramidal and tetragonal–bipyramidal.

In each molecule there is an intramolecular hydrogen bond (C14—H14A···O1 and C15—H15A···O2), forming a six-membered ring. In the crystal structure, molecules are linked into a three-dimensional framework by O—H···Cl and C—H···Cl hydrogen bonds (Table 2 and Fig. 2). The packing is further stabilized by π–π stacking interactions involving the phenanthroline ring systems with a $Cg3\cdots Cg7^{iv}$ distance of 3.637 Å [$Cg3$ and $Cg7$ are the centroids of the N1/C2–C5/C13 and C5–C8/C12/C13 rings, respectively; symmetry code: (iv) $3 - x, 2 - y, 1 - z$].

Experimental

To a solution of 2,9-dimethyl-1,10-phenanthroline (0.21 g, 1 mmol) in ethanol (10 ml) a solution of NiCl₂ (0.13 g, 1 mmol) in distilled water (10 ml) was added. The mixture was stirred and refluxed for 7 h. The hot solution was then filtered into a flask containing ethanol–water (1:1 *v/v*). Green crystals appeared over a period of one week by slow evaporation at room temperature.

Crystal data

[NiCl$_2$(C$_{14}$H$_{12}$N$_2$)(H$_2$O)]	$V = 1518.7$ (5) Å^3
$M_r = 711.76$	$Z = 4$
Triclinic, $P\bar{1}$	$D_x = 1.556$ Mg m^{-3}
$a = 7.5631$ (13) Å	Mo $K\alpha$ radiation
$b = 11.501$ (2) Å	$\mu = 1.62$ mm^{-1}
$c = 18.924$ (4) Å	$T = 293$ (2) K
$\alpha = 106.692$ (3)°	Plate, green
$\beta = 93.545$ (4)°	$0.30 \times 0.16 \times 0.06$ mm
$\gamma = 103.467$ (2)°	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	8714 measured reflections
	5826 independent reflections
ω scans	4892 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.017$
$T_{min} = 0.641$, $T_{max} = 0.909$	$\theta_{max} = 26.1°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0726P)^2 + 0.9988P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.04$	$\Delta\rho_{max} = 1.04$ e Å^{-3}
5826 reflections	$\Delta\rho_{min} = -0.49$ e Å^{-3}
361 parameters	
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

Ni1—O1	2.015 (3)	Ni2—O2	2.011 (3)
Ni1—N1	2.049 (3)	Ni2—N4	2.049 (3)
Ni1—N2	2.059 (3)	Ni2—N3	2.053 (3)
Ni1—Cl1	2.3431 (10)	Ni2—Cl4	2.3435 (10)
Ni1—Cl2	2.3548 (10)	Ni2—Cl3	2.3515 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1W1···Cl3^i	0.85	2.40	3.107 (4)	140
O1—H1W1···Cl4	0.85	2.80	3.106 (3)	103
O2—H1W2···Cl1	0.86	2.34	3.141 (3)	157
O1—H2W1···Cl4	0.86	2.79	3.106 (3)	104
O2—H2W2···Cl2^{ii}	0.86	2.31	3.152 (3)	166
C14—H14A···O1	0.96	2.40	3.315 (6)	159
C15—H15A···O2	0.96	2.28	3.223 (6)	168
C17—H17A···Cl4^{iii}	0.93	2.83	3.634 (6)	145

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

C-bound H atoms were refined using a riding model with C—H bond distances of 0.93–0.96 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). The H atoms of the water molecules were located in a difference Fourier map and refined as riding at the distances
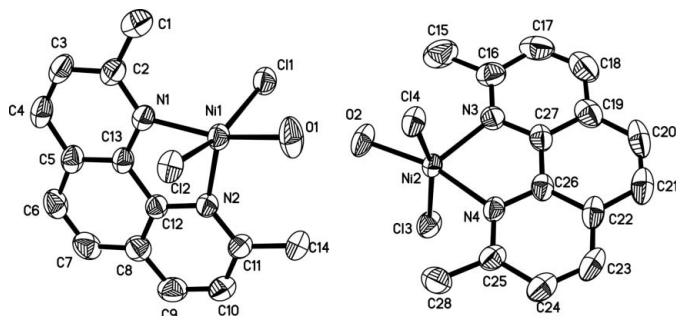

Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme. H atoms have been omitted for clarity.
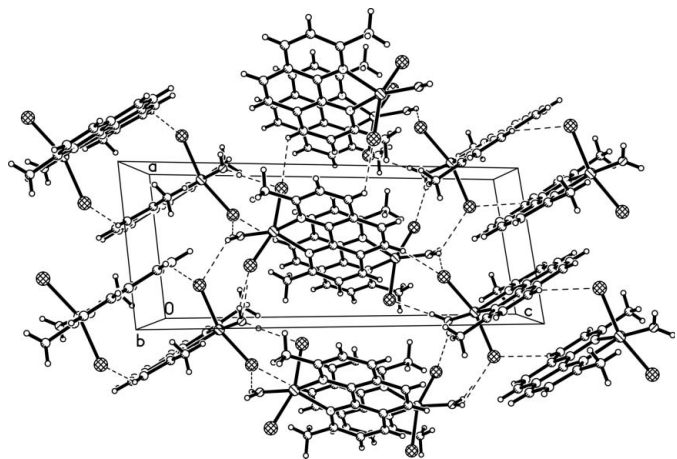

Figure 2
The crystal packing viewed down the *b* axis, showing the π–π interactions. Hydrogen bonds are indicated by dashed lines.

shown in Table 2, with $U_{iso}(H) = 1.2U_{eq}(O)$. The maximum electron-density peak in the final difference map lies 0.89 Å from atom Cl4.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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