Aquachlorobis(1,10-phenanthroline)nickel(II) chloride thioacetamide solvate

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The Ni atom in the cation of the title complex, $[NiCl(C_{12}H_8N_2)_2(H_2O)]Cl \cdot C_2H_5NS$, has a distorted octahedral coordination geometry defined by a Cl atom, the O atom of a water molecule, and four N atoms from two phen ligands. Molecules are linked into a three-dimensional framework primarily by O—H⋯Cl hydrogen bonds and π–π stacking interactions.

Comment

π–π Stacking between aromatic rings is related to electron-transfer processes in some biological systems (Deisenhofer & Michel, 1989). A series of metal complexes incorporating different aromatic ligands such as 1,10-phenanthroline (phen), benzimidazole and quinoline have been prepared and their crystal structures determined to provide useful information about π–π stacking (Wu et al., 2003; Pan & Xu, 2004; Li et al., 2005). In the present study, the structure of the title compound, (I), obtained from the reaction of nickel dichloride hexahydrate, thioacetamide and phen in an ethanol solution, is described.

(I)
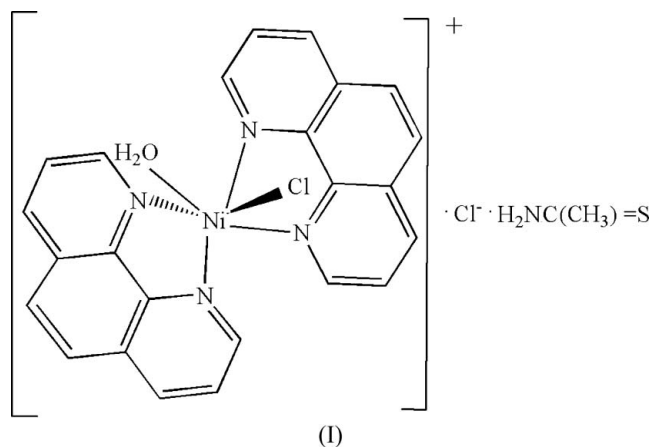
The asymmetric unit of (I) comprises an $[Ni(phen)_2(OH_2)Cl]^+$ cation, a Cl^- anion and a solvent thioacetamide molecule (Fig. 1). Four N atoms, derived from two phen ligands, a Cl anion and one O atom from the water molecule define a distorted octahedral geometry for the Ni centre. The phen ligands are nearly perpendicular to each other, making a dihedral angle of 86.56 (4)°. Distortions from the regular octahedral geometry may be related to the different bond distances and acute chelate angles (Table 1).

In the crystal structure, molecules are linked into a three-dimensional framework primarily by O—H⋯Cl hydrogen bonds (Fig. 2 and Table 2). There are π–π stacking interactions
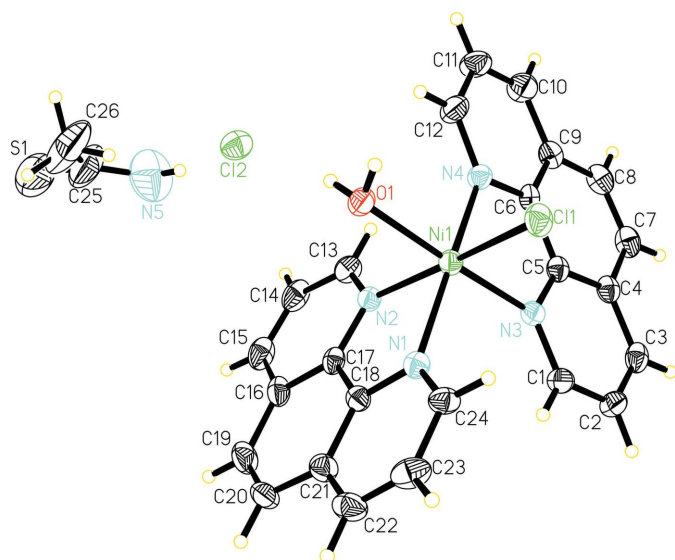
Figure 1
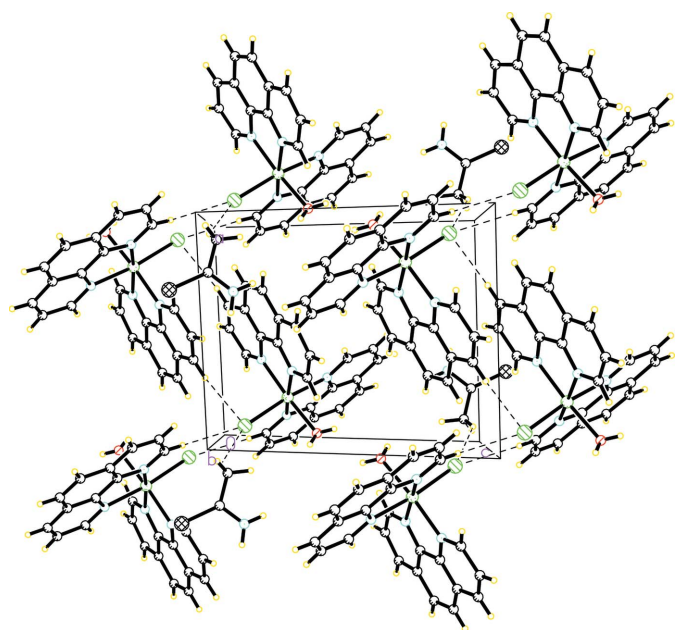The asymmetric unit of (I), showing 30% probability displacement ellipsoids.

Figure 2
Packing diagram for (I), showing hydrogen bonds as dashed lines.

between adjacent phen ligands with a centroid–centroid distance of 3.615 (6) Å (symmetry code: $1 - x, -y, 1 - z$).

Experimental

Nickel dichloride hexahydrate (4.75 g, 20 mmol), phen (3.96 g, 20 mmol) and thioacetamide (0.75 g, 10 mmol) were dissolved in an ethanol solution (25 ml). The mixture was heated for 6 h under reflux with stirring. It was then filtered to give a clear solution, into which diethyl ether vapour was allowed to condense in a closed vessel. After being allowed to stand for a few days at room temperature, some green single crystals suitable for X-ray crystallographic analysis precipitated.

Crystal data

[NiCl(C$_{12}$H$_8$N$_2$)$_2$(H$_2$O)]Cl·C$_2$H$_5$NS	$V = 1247.5$ (2) Å^3
$M_r = 583.16$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.553$ Mg m^{-3}
$a = 9.7697$ (5) Å	Mo $K\alpha$ radiation
$b = 11.2981$ (10) Å	$\mu = 1.11$ mm^{-1}
$c = 12.7974$ (15) Å	$T = 273$ (2) K
$\alpha = 64.193$ (4)°	Block, green
$\beta = 87.070$ (3)°	$0.16 \times 0.11 \times 0.06$ mm
$\gamma = 78.989$ (4)°	

Data collection

Bruker APEX-II area-detector diffractometer	8909 measured reflections
φ and ω scans	5336 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	2774 reflections with $I > 2\sigma(I)$
$T_{min} = 0.845$, $T_{max} = 0.933$	$R_{int} = 0.047$
	$\theta_{max} = 27.1°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.7997P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.98$	$\Delta\rho_{max} = 0.90$ e Å^{-3}
5336 reflections	$\Delta\rho_{min} = -0.47$ e Å^{-3}
334 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

Ni1—Cl1	2.3671 (13)	Ni1—N3	2.087 (4)
Ni1—N1	2.085 (3)	Ni1—N4	2.081 (4)
Ni1—N2	2.117 (3)		
Cl1—Ni1—O1	88.60 (11)	O1—Ni1—N4	95.25 (14)
Cl1—Ni1—N1	96.38 (10)	N1—Ni1—N2	79.07 (14)
Cl1—Ni1—N2	172.88 (11)	N1—Ni1—N3	93.59 (14)
Cl1—Ni1—N3	94.05 (10)	N1—Ni1—N4	169.18 (14)
Cl1—Ni1—N4	92.82 (10)	N2—Ni1—N3	91.71 (13)
O1—Ni1—N1	90.67 (13)	N2—Ni1—N4	92.29 (13)
O1—Ni1—N2	86.00 (15)	N3—Ni1—N4	80.05 (14)
O1—Ni1—N3	174.70 (14)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C24—H24⋯Cl1	0.93	2.86	3.431 (5)	121
O1—H1B⋯Cl2^i	0.82 (3)	2.41 (2)	3.189 (4)	158 (4)
N5—H5B⋯S1^{ii}	0.86	2.52	3.361 (8)	165
N5—H5A⋯Cl2	0.86	2.59	3.423 (6)	163
O1—H1A⋯Cl2	0.81 (3)	2.306 (15)	3.110 (4)	170 (4)

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

H atoms of the water molecule were refined without contraint (see Table 2 for distances). H atoms on C and N atoms were placed in calculated positions, with N—H = 0.86 Å and C—H = 0.93–0.96 Å, and refined in the riding-model approximation with U_{iso}(H) = $1.2U_{eq}$(N,C).

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *APEX2*; software used to prepare material for publication: *SHELXL97*.

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