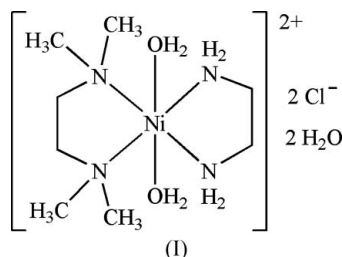


Diaqua(ethylenediamine)(*N,N,N',N'*-tetramethylethylenediamine)nickel(II) dichloride dihydrate**Zi-Lu Chen,* Yu-Zhen Zhang and Fu-Pei Liang**College of Chemistry and Chemical Engineering,
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Guilin 541004, People's Republic of ChinaCorrespondence e-mail:
chenziluczl@yahoo.co.uk**Key indicators**Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
 R factor = 0.038
 wR factor = 0.112
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $[\text{Ni}(\text{C}_2\text{H}_8\text{N}_2)(\text{C}_6\text{H}_{16}\text{N}_2)(\text{H}_2\text{O})_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$, the Ni^{II} ion, located on a twofold axis, is coordinated by four N atoms of the ethylenediamine and *N,N,N',N'*-tetramethylethylenediamine ligands and two water molecules in a distorted octahedral geometry. Hydrogen bonds between ethylenediamine, water molecules and chloride ions help to construct a three-dimensional supramolecular architecture.

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As the development of complexes bearing NH functionalities as hydrogenation catalysts is rapidly increasing (Hedberg *et al.*, 2005; Wu *et al.*, 2003; Sandoval *et al.*, 2003; Noyori *et al.*, 2001; Ohkuma *et al.*, 2002), we have paid much attention to the preparation of related complexes using 3d metals such as nickel, cobalt and manganese. We report here the synthesis and X-ray structure of the title complex, (I).



The asymmetric unit of (I) consists of one-half of the Ni^{II} complex cation, with the other half generated by twofold symmetry, a chloride anion and an uncoordinated water molecule. The Ni^{II} ion, located on a twofold axis, is coordinated by two N atoms of one ethylenediamine ligand, two N atoms of one *N,N,N',N'*-tetramethylethylenediamine ligand and two water molecules in a distorted octahedral geometry (Table 1). Each chloride ion is linked to two uncoordinated water molecules and one coordinated water molecule through $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds (Table 2). In addition, $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds involving the amino groups of the ethylenediamine ligands, and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the water molecules help to construct a three-dimensional supramolecular architecture (Fig. 2).

Experimental

A methanol solution (10 ml) of *N,N,N',N'*-tetramethylethylenediamine (0.12 ml, 1 mmol) and ethylenediamine (0.07 ml, 1 mmol) was added dropwise to a methanol solution (5 ml) of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.2370 g, 1 mmol) with stirring. The resulting solution was stirred at room temperature for a further 12 h and then filtered. Diffusion of diethyl ether into the filtrate gave blue crystals of (I).

Crystal data

[Ni(C₂H₈N₂)(C₆H₁₆N₂)(H₂O)₂]
Cl₂·2H₂O
M_r = 377.99
Orthorhombic, *Pbcn*
a = 15.005 (4) Å
b = 9.591 (3) Å
c = 12.505 (3) Å
V = 1799.6 (9) Å³
Z = 4
D_x = 1.395 Mg m⁻³
Mo Kα radiation
μ = 1.39 mm⁻¹
T = 298 (2) K
Block, blue
0.35 × 0.33 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1998)
T_{min} = 0.629, T_{max} = 0.744
8632 measured reflections
1593 independent reflections
1303 reflections with I > 2σ(I)
R_{int} = 0.028
θ_{max} = 25.0°

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.038
wR(F²) = 0.112
S = 1.09
1593 reflections
91 parameters
H-atom parameters constrained
w = 1/[σ²(F_o²) + (0.0503P)² + 3.0865P]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.77 e Å⁻³
Δρ_{min} = -0.89 e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0173 (16)

Table 1

Selected geometric parameters (Å, °).

Ni1—N1	2.099 (3)	Ni1—N2	2.166 (3)
Ni1—O1	2.145 (3)		
N1—Ni1—N1 ⁱ	82.8 (2)	O1—Ni1—N2 ⁱ	90.93 (11)
N1—Ni1—O1	90.06 (12)	N1—Ni1—N2	96.68 (14)
N1—Ni1—O1 ⁱ	87.85 (12)	O1—Ni1—N2	91.15 (11)
O1—Ni1—O1 ⁱ	177.21 (15)	N2 ⁱ —Ni1—N2	83.90 (19)
N1—Ni1—N2 ⁱ	178.84 (13)		

Symmetry code: (i) -x + 1, y, -z + ½.

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H7...Cl1	0.85	2.29	3.132 (4)	168
O2—H8...Cl1 ⁱⁱ	0.85	2.35	3.188 (4)	168
O1—H6...Cl1 ⁱⁱⁱ	0.85	2.29	3.139 (3)	173
O1—H5...O2 ^{iv}	0.85	1.86	2.707 (4)	177
N1—H1B...Cl1 ⁱⁱ	0.90	2.59	3.378 (4)	147
N1—H1A...O2 ^{iv}	0.90	2.44	3.291 (6)	158

Symmetry codes: (ii) -x + ½, y + ½, z; (iii) x - ½, y + ½, -z + ½; (iv) -x + ½, -y + ½, z + ½.

H atoms of water molecules were located in a difference Fourier map and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(O). H atoms on N and C atoms were placed in calculated positions, with N—H = 0.90 Å and C—H = 0.97 Å for the ethylene group and 0.96 Å for the methyl group, and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,N) for other atoms.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.



Figure 1

A view of the structure of (I), showing the atom-numbering scheme and 30% displacement ellipsoids. Dashed lines indicate hydrogen bonds. [Symmetry code: (i) 1-x, y, 3/2-z.]

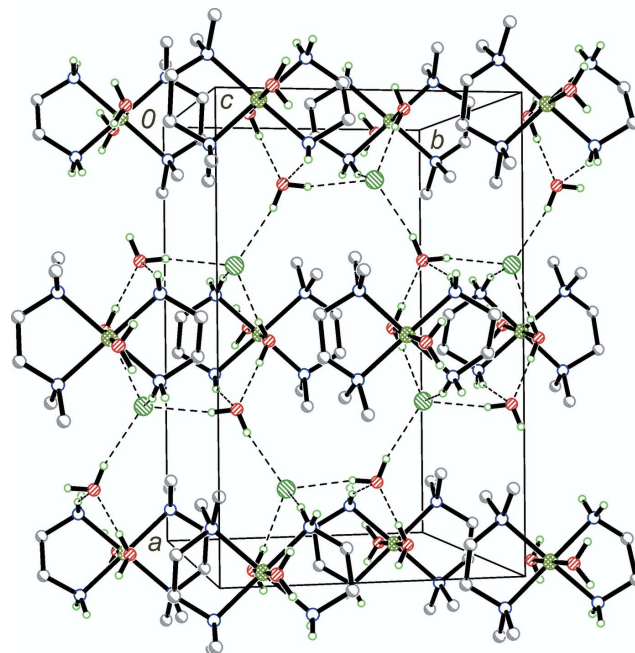


Figure 2

A view of the three-dimensional supramolecular network of (I), showing hydrogen bonds between ethylenediamine, water molecules and chloride ions, indicated by dashed lines. H atoms not involved in the interactions shown have been omitted for clarity.

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