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Tris(1,2-ethanediamine-κ^2N,N')nickel(II) dichloride dihydrate

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Key indicators

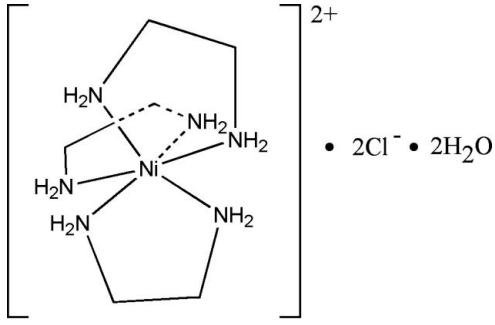
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.011 Å
R factor = 0.034
wR factor = 0.099
Data-to-parameter ratio = 8.7

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

The title compound, $[Ni(C_2H_8N_2)_3]Cl_2\cdot 2H_2O$ or $[Ni(en)_3]Cl_2\cdot 2H_2O$, where en is 1,2-ethanediamine, contains three $[Ni(en)_3]^{2+}$ cations, six Cl^- anions and six water molecules in the asymmetric unit. The crystal structure is supported by O—H···Cl, N—H···Cl, O—H···O and N—H···O hydrogen bonds.

Comment

There are numerous examples of tris(1,2-ethanediamine-*N*,*N*′)nickel(II) $[Ni(en)_3]^{2+}$ complexes (James *et al.*, 1998; Chesnut *et al.*, 1999; Xiang *et al.*, 2001; Wrzeszcz *et al.*, 2002). We present here the structure of the title compound, (I).



(I)

As shown in Fig. 1, there are three crystallographically independent $[Ni(en)_3]^{2+}$ cations, six Cl^- anions and six water molecules in the asymmetric unit. In the $[Ni(en)_3]^{2+}$ complex cation, three bidentate en ligands are coordinated to an Ni^{II} ion, forming a six-coordinated nickel(II) complex with slightly distorted octahedral coordination geometry. Selected bond distances and angles are given in Table 1 for one cation and these show no unusual values; those for the other two cations are very similar.

In the crystal structure (Fig. 2), $[Ni(en)_3]^{2+}$ cations are linked to Cl^- anions and water molecules *via* N—H···Cl and N—H···O hydrogen bonds, respectively, and the water molecules are linked to the Cl^- anions *via* O—H···Cl hydrogen bonds (Table 2).

Experimental

$NiCl_2\cdot 6H_2O$, $H_2NCH_2CH_2NH_2$ and H_2O, in a molar ratio of 1:3:150, were mixed and dissolved in sufficient ethanol by heating to 373 K, to give a clear solution. After the reaction system had been cooled slowly to room temperature, crystals of (I) formed, and these were collected and washed with distilled water.

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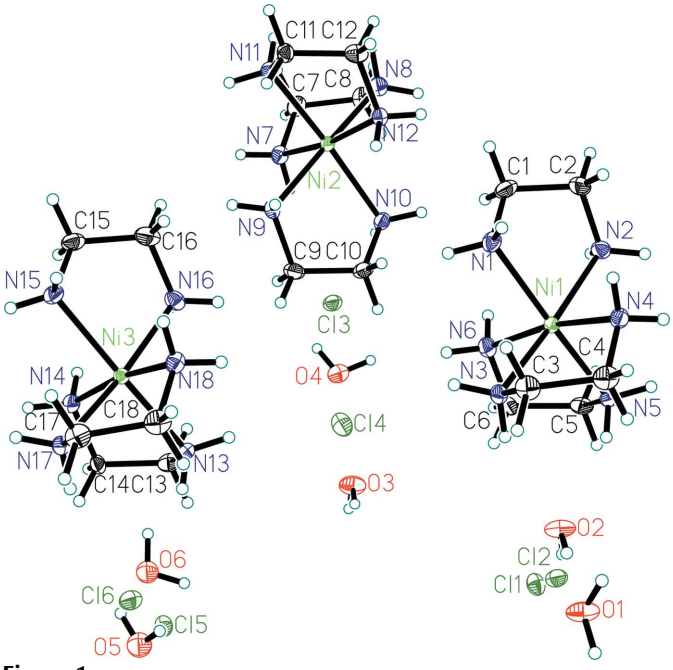

Figure 1
The asymmetric unit of (I), with atom labels, showing 35% probability displacement ellipsoids.

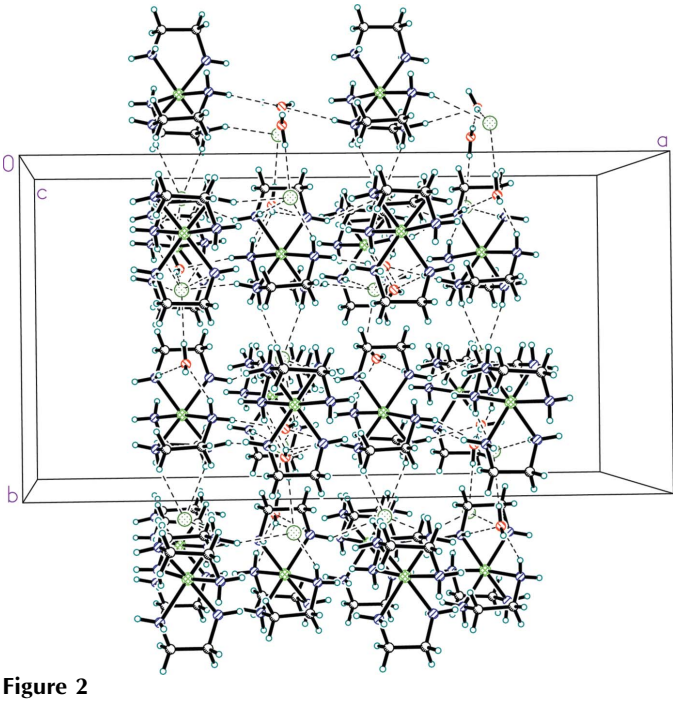

Figure 2
A packing diagram viewed down along the *c* axis. Hydrogen bonds are drawn as dashed lines.

Crystal data

$[Ni(C_2H_8N_2)_3]Cl_2·2H_2O$
M_r = 345.93
Monoclinic, *Cc*
a = 26.186 (2) Å
b = 13.8917 (9) Å
c = 12.8986 (8) Å
β = 93.197 (2)°
V = 4684.8 (6) Å^3
Z = 12
D_x = 1.471 Mg m^{-3}
Mo *K*α radiation
μ = 1.59 mm^{-1}
T = 293 (2) K
Block, green
0.30 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
12308 measured reflections
5282 independent reflections
4247 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.099
S = 1.05
4247 reflections
486 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 4.1952P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Absolute structure: Flack (1983), with 4239 Friedel pairs
Flack parameter: 0.00 (2)

Table 1
Selected geometric parameters (Å, °).

Ni1—N5	2.122 (6)	Ni1—N2	2.131 (7)
Ni1—N4	2.123 (6)	Ni1—N3	2.146 (6)
Ni1—N1	2.130 (6)	Ni1—N6	2.149 (6)
N5—Ni1—N4	92.6 (3)	N1—Ni1—N3	93.0 (2)
N5—Ni1—N1	170.9 (2)	N2—Ni1—N3	172.6 (2)
N4—Ni1—N1	94.2 (2)	N5—Ni1—N6	81.9 (2)
N5—Ni1—N2	92.3 (2)	N4—Ni1—N6	170.6 (3)
N4—Ni1—N2	93.4 (2)	N1—Ni1—N6	92.1 (3)
N1—Ni1—N2	81.3 (2)	N2—Ni1—N6	94.5 (3)
N5—Ni1—N3	93.9 (2)	N3—Ni1—N6	90.4 (2)
N4—Ni1—N3	82.3 (2)		
N13—C13—C14—N14	56.1 (7)	N15—C15—C16—N16	57.9 (10)
N3—C3—C4—N4	56.2 (11)	N17—C17—C18—N18	55.5 (10)
N11—C11—C12—N12	−56.2 (7)	N1—C1—C2—N2	59.3 (10)
N7—C7—C8—N8	−58.1 (10)	N5—C5—C6—N6	55.6 (8)
N9—C9—C10—N10	−56.3 (10)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*E*···O1	0.85 (4)	1.97 (2)	2.815 (9)	179 (5)
O2—H2*F*···Cl2	0.85 (4)	2.45 (2)	3.236 (8)	155 (3)
O4—H4*E*···Cl3	0.85 (4)	2.64 (4)	3.163 (8)	121 (4)
O5—H5*E*···Cl6	0.85 (4)	2.56 (2)	3.231 (9)	136 (3)
O5—H5*F*···O6	0.85 (4)	2.17 (2)	2.749 (4)	125 (4)
N3—H*B*3···O2	0.90	2.23	3.086 (5)	158
N5—H*A*5···O2	0.90	2.21	3.064 (4)	158
N6—H*A*6···Cl4	0.90	2.65	3.443 (3)	147
N13—H*A*13···Cl4	0.90	2.64	3.440 (6)	149
N13—H*B*13···Cl6	0.90	2.54	3.401 (5)	160
N17—H*B*17···Cl6	0.90	2.50	3.371 (4)	161

Water H atoms were located in a difference Fourier map and refined with the O—H distances restrained to 0.85 (2) Å. Other H atoms were treated as riding atoms, at distances of N—H = 0.90 Å and C—H = 0.97 Å, with U_{iso}(H) = 1.2U_{eq}(C,N). The structure shows a high degree of pseudo-symmetry, but could not be successfully refined in space group *C*2/*c*.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine

structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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