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cis-Diaquabis(ethylenediamine)nickel(II) benzene-1,4-dicarboxylate monohydrate

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The title compound, *cis*-$[Ni(C_2H_8N_2)_2(H_2O)_2](C_8H_4O_4)\cdot H_2O$, was obtained by a reaction of nickel chloride hexahydrate, terephthalic acid, ethylenediamine and NaOH (molar ratio 2:1:4:2). The compound contains a six-coordinate Ni^{II} cation, with four ethylenediamine N atoms and two water O atoms attached to the Ni atom, one benzene-1,4-dicarboxylate anion, and one water molecule of solvation. A C_2 axis passes through the Ni atom, the anion and the water O atom. Intermolecular hydrogen-bonding interactions are present, linking the nickel complex cations, organic anions and uncoordinated water molecules in the crystal structure.

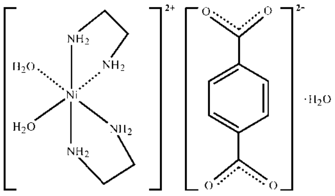
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.097
Data-to-parameter ratio = 20.6

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

Comment

The importance of functional materials with extended structures has attracted a lot of attention recently (Yaghi *et al.*, 1998; Eddaoudi *et al.*, 2001). Both hydrogen bonds and π–π interactions are known as tools in the preparation of this type of material *via* a variety of metal ions and organic di- or polylinkers. In this context, we report here the synthesis and crystal structure of the title nickel complex, (I), in a hydrogen-bonded framework with weak π–π interactions.



(I)

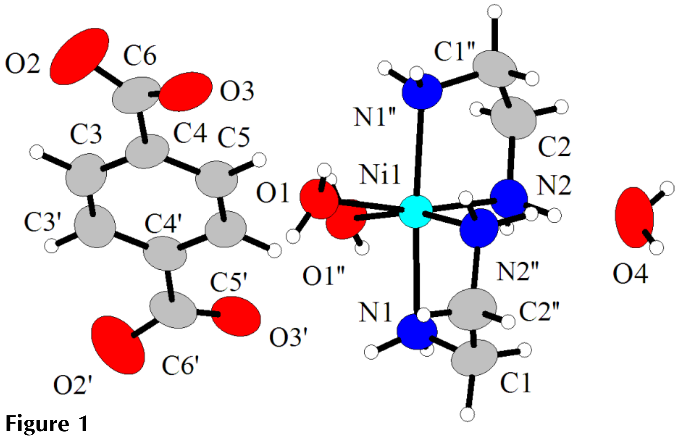


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii [symmetry codes: (′) $-x, y, \frac{1}{2} - z$; (″) $1 - x, y, \frac{3}{2} - z$].

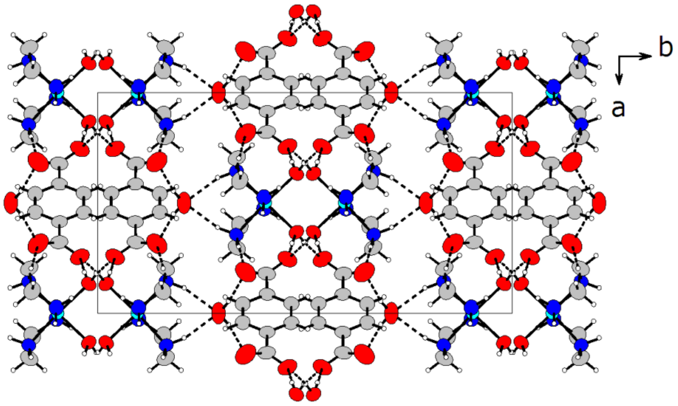


Figure 2
The crystal packing diagram of (I), with hydrogen bonds shown as dashed lines, viewed along the *c* axis.

The title compound, (I), contains a six-coordinate Ni^{II} cation, with four ethylenediamine N atoms and two water O atoms in a *cis* orientation attached to the Ni atom, one 1,4-benzenedicarboxylate anion, and one water molecule of solvation (Fig. 1). A crystallographically imposed C_2 axis is present, passing through the uncoordinated water atom O4, Ni1, and the mid-points of the C3—C3′ and C5—C5′ bonds. The most interesting feature of the crystal structure is the intermolecular hydrogen bonding between cations as the hydrogen-bond donors and anions as the hydrogen-bond acceptors, *via* hydrogen-bond connectors, *viz*. the uncoordinated water molecules (Fig. 2). All the hydrogen-bonding interactions appear normal (Steed & Atwood, 2000), based on H···A distances of 1.87–2.14 Å, *D*···*A* distances of 2.651 (2)–3.003 (2) Å and bond angles of 160–171° (Table 2).

Experimental

Terephthalic acid (0.351 g, 2.092 mmol), nickel chloride hexahydrate (1.101 g, 4.631 mmol), imidazole (0.607 g, 8.827 mmol) and NaOH (0.229 g, 5.317 mmol) were dissolved by stirring in 10 ml of water. After three months, the blue single crystals of (I) which had formed were collected and dried in air (yield 50%).

Crystal data

$[Ni(C_2H_8N_2)_2(H_2O)_2](C_8H_4O_4)\cdot H_2O$
$M_r = 397.08$
Monoclinic, $C2/c$
$a = 12.489\ (1)$ Å
$b = 20.494\ (2)$ Å
$c = 8.4365\ (6)$ Å
$\beta = 122.191\ (1)°$
$V = 1827.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3931 reflections
$\theta = 2.5–25°$
$\mu = 1.10$ mm^{-1}
$T = 295\ (2)$ K
Column, blue
0.60 × 0.40 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1995)
$T_{min} = 0.620$, $T_{max} = 0.718$
6464 measured reflections
2269 independent reflections
2024 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 28.3°$
$h = -13 \rightarrow 16$
$k = -27 \rightarrow 22$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.097$
$S = 1.07$
2269 reflections
110 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.0602P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	2.0965 (11)	Ni1—O1	2.1148 (10)
Ni1—N1	2.1076 (10)		
N2—Ni1—N1	94.69 (4)	N1—Ni1—O1	97.31 (4)
N2—Ni1—O1	167.68 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O3	0.82	1.87	2.651 (2)	160
O1—H1*B*···O3^{i}	0.83	1.88	2.677 (2)	161
N2—H2*A*···O4	0.90	2.14	3.003 (2)	161
N2—H2*B*···O2^{ii}	0.90	2.03	2.919 (3)	171
O4—H4*A*···O2^{iii}	0.87	1.87	2.733 (3)	170

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

H atoms were located in difference Fourier maps and refined as riding on their parent C, N and O atoms (C—H = 0.93 and 0.97 Å, N—H = 0.90 Å and O—H = 0.82 and 0.83 Å; $U_{iso} = 1.2U_{eq}$ of the parent atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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