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Hexaaquanickel(II) bis(*p*-nitrobenzoate) dihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.053
wR factor = 0.160
Data-to-parameter ratio = 13.2

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

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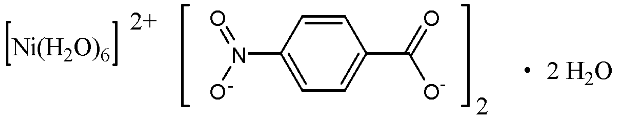
In the title complex, $[Ni(H_2O)_6]L_2\cdot 2H_2O$, where L is *p*-nitrobenzoate ($C_7H_4NO_4$), each Ni^{II} cation lies on an inversion center and is octahedrally coordinated by six water molecules. The L^- anions do not coordinate to the nickel, but act as counter-anions. The crystal structure is composed of alternating layers of $[Ni(H_2O)_6]^{2+}$ cations and L anions. The $[Ni(H_2O)_6]^{2+}$ cations, water molecules and L^- anions are connected through a complex pattern of hydrogen-bonding interactions, resulting in a three-dimensional network.

Comment

For many purposes, including catalysis, it is desirable to utilize transition metal complexes that contain anions which coordinate weakly or not at all (Batsanov *et al.*, 2001). We report here the structure of the nickel complex $[Ni(H_2O)_6]L_2\cdot 2H_2O$, where L is *p*-nitrobenzoate. The single-crystal X-ray structure analysis indicates that the crystal structure is built up of $[Ni(H_2O)_6]^{2+}$ cations, two uncoordinated L anions and two uncoordinated water molecules. Each Ni^{II} atom lies on an inversion center and is hexacoordinated by the six aqua ligands. The Ni—O distances range from 2.097 (3) to 2.137 (2) Å. The mean Ni—O distance of 2.117 (3) Å is longer than the values observed in other nickel compounds (Ma *et al.*, 2003). The three *trans* angles in the Ni octahedron are 177.40 (18), 173.82 (11) and 173.82 (11)°. The *cis* angles around the Ni^{II} atom deviate slightly from the ideal angle of 90° [85.02 (11)–93.97 (19)°]; thus, the Ni coordination center has a slightly distorted octahedral geometry. Selected bond lengths and angles are given in Table 1. The alternating layers of $[Ni(H_2O)_6]^{2+}$ cations and L anions in (I) are shown in Fig. 2. Selected hydrogen-bond parameters are listed in Table 1.



(I)

Experimental

All reagents and solvents were used as obtained without further purification. $Ni(OH)_2$ (0.3 mmol, 52 mg) and *p*-nitrobenzoic acid (0.6 mmol, 86 mg) were dissolved in ammonia (15 ml). The mixture was stirred for about 1 h to obtain a clear blue solution. After allowing the solution to stand in air for two weeks with ammonia gas escaping, large light-blue crystals were formed, The product was isolated, washed three times with water, and dried in a vacuum desiccator using P_4O_{10} (yield 56%). Elemental analysis found: C 31.36, H 4.66, N 5.30, Ni 10.84; calculated for $C_{14}H_{24}NiN_2O_{16}$: C 31.43, H 4.52, N 5.24, Ni 10.97%.

Crystal data

$[Ni(H_2O)_6](C_7H_4NO_4)_2 \cdot 2H_2O$
$M_r = 535.06$
Monoclinic, $C2/c$
$a = 29.030$ (6) Å
$b = 7.0550$ (14) Å
$c = 11.916$ (2) Å
$\beta = 112.85$ (3)°
$V = 2249.0$ (8) Å^3
$Z = 4$

$D_x = 1.580$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 60 reflections
$\theta = 2.5$–$24.4°$
$\mu = 0.94$ mm^{-1}
$T = 293$ (2) K
Block, light blue
$0.42 \times 0.27 \times 0.26$ mm

Data collection

Bruker SMART diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.744$, $T_{\max} = 0.782$
4473 measured reflections
1986 independent reflections

1818 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 25.0°$
$h = -34 \rightarrow 25$
$k = -8 \rightarrow 5$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.160$
$S = 1.08$
1986 reflections
150 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 4.0152P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.54$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O8$^{\rm i}$	0.85	2.23	2.777 (4)	122
O5—H5A···O7$^{\rm ii}$	0.85	2.62	3.255 (5)	132
O5—H5B···O2$^{\rm iii}$	0.85	1.88	2.680 (4)	156
O6—H6B···O3$^{\rm iv}$	0.85	2.46	3.221 (5)	149
O6—H6C···O4$^{\rm v}$	0.85	2.43	3.193 (5)	150
O7—H7B···O3$^{\rm v}$	0.85	2.58	3.417 (5)	168
O7—H7C···O8$^{\rm vi}$	0.85	2.31	3.092 (5)	154
O8—H8A···O2$^{\rm vii}$	0.85	2.16	2.790 (4)	131
O8—H8B···O1	0.85	2.14	2.710 (5)	125

Symmetry codes: (i) $x, y-1, z$; (ii) $x, -y, z-\frac{1}{2}$; (iii) $-x, y-1, \frac{1}{2}-z$; (iv) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (v) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (vi) $-x, y, \frac{1}{2}-z$; (vii) $x, 1-y, z-\frac{1}{2}$.

The H atoms bonded to C and O atoms were placed in calculated positions, with C—H = 0.96 Å and O—H = 0.85 Å. The $U_{\rm iso}$(H) values were fixed at 0.08 Å^2.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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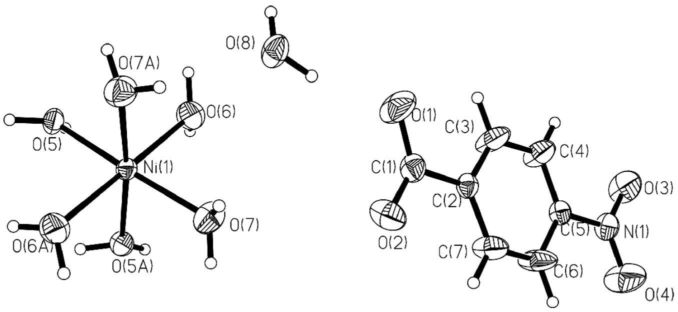

Figure 1
View of the asymmetric unit of (I), expanded to show the complete coordination of Ni$^{\rm II}$, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) $-x, y, \frac{1}{2}-z2$.]

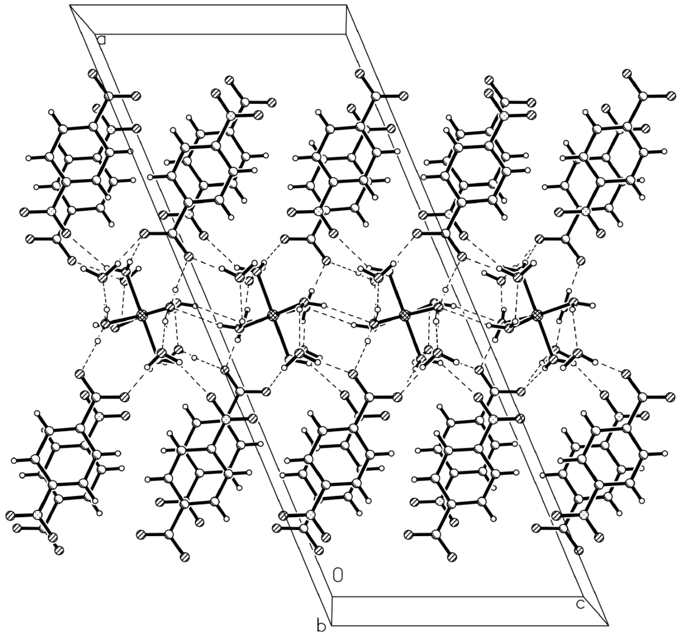

Figure 2
The packing of (I), viewed along the a axis.

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