metal-organic compounds

Tetraaquabis[3-(4-pyridyl)benzoato-κN]-nickel(II)

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The Ni^{II} atom in the title compound, [Ni(C$_{12}$H$_8$NO$_2$)$_2$(H$_2$O)$_4$], exists in an all-*trans* octahedral coordination environment. The 3-(4-pyridyl)benzoate ligand binds to Ni atom through the pyridyl N atom; the pyridine and benzene rings are oriented at a dihedral angle of 26.27 (10)°. Adjacent complexes are linked by O—H⋯O hydrogen bonds, forming a three-dimensional network. The metal atom lies on a special position of 2 site symmetry in the crystal structure.

Related literature

The 3-(pyridin-4-yl)benzoate unit is fairly rigid like the nicotinate unit, which also forms a similar zwitterionic nickel derivative; see: Batten & Harris (2001).
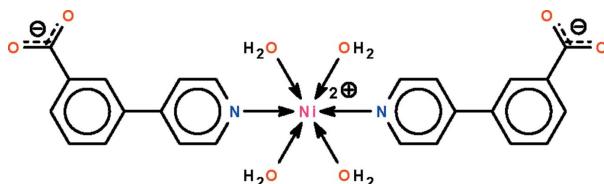
Experimental

Crystal data

[Ni(C$_{12}$H$_8$NO$_2$)$_2$(H$_2$O)$_4$]
$M_r = 527.16$
Monoclinic, $C2/c$
$a = 24.564$ (3) Å
$b = 7.0520$ (8) Å
$c = 13.781$ (2) Å
$\beta = 113.325$ (2)°

$V = 2192.1$ (4) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.94$ mm^{-1}
$T = 173$ K
$0.47 \times 0.31 \times 0.08$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{min} = 0.666$, $T_{max} = 0.929$

5587 measured reflections
2360 independent reflections
1977 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.090$
$S = 1.09$
2360 reflections
175 parameters
4 restraints

H atoms treated by a mixture of
 independent and constrained
 refinement
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1W	2.0627 (14)	Ni1—N1	2.0931 (16)
Ni1—O2W	2.0811 (14)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1w—H11⋯O1^i	0.84 (1)	1.88 (1)	2.682 (2)	160 (3)
O1w—H12⋯O2^{ii}	0.83 (1)	1.91 (1)	2.734 (2)	170 (2)
O2w—H21⋯O1^{iii}	0.84 (1)	1.93 (1)	2.732 (2)	159 (2)
O2w—H22⋯O2^{iv}	0.83 (1)	1.88 (1)	2.711 (2)	177 (3)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Batten, S. R. & Harris, A. R. (2001). *Acta Cryst.* E**57**, m9–m11.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS inc., Madison, Wisconsin, USA.
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Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

Tetraaquabis[3-(4-pyridyl)benzoato-*κN*]nickel(II)

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S1. Experimental

3-(Pyridin-4-yl)benzoic acid was purchased from a chemical supplier. The reagent (0.199 g, 1 mmol) and sodium hydroxide (0.040 g, 1 mmol) were mixed with nickel(II) nitrate hexahydrate (0.150 g, 0.5 mmol) in water (10 ml). The mixture was placed in a 15 ml Teflon-lined autoclave and heated at 423 K for 48 h. The autoclave was cooled over 12 h at a rate of 5 K an hour. Green crystals were isolated by hand (yield *ca* 60% based on Ni).

S2. Refinement

Carbon-bound hydrogen atoms were generated geometrically and were constrained to ride on their parent atoms [C–H = 0.95 Å; U_{iso}(H) =1.2U_{eq}(C)]. The water H-atoms were located in a difference Fourier map, and were refined with a distance restraint of O–H 0.84±0.01 Å; their temperature factors were refined.
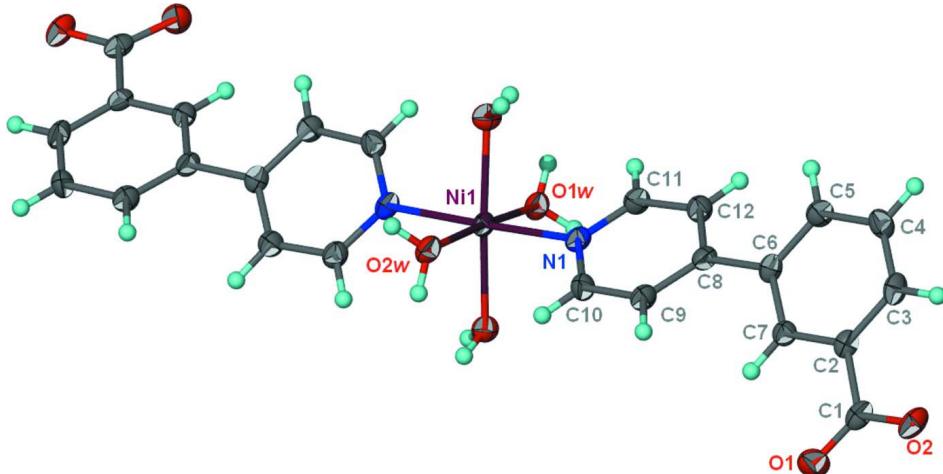
Figure 1

Thermal ellipsoid plot (Barbour, 2001) of Ni(H$_2$O)$_4$(C$_{12}$H$_2$NO$_2$)$_2$ at the 70% probability level; hydrogen atoms are drawn as sphere of arbitrary radius.

Tetraaquabis[3-(4-pyridyl)benzoato-*κN*]nickel(II)

Crystal data

[Ni(C$_{12}$H$_8$NO$_2$)$_2$(H$_2$O)$_4$]	c = 13.781 (2) Å
M_r = 527.16	β = 113.325 (2)°
Monoclinic, *C*2/*c*	V = 2192.1 (4) Å^3
Hall symbol: -C 2yc	Z = 4
a = 24.564 (3) Å	F(000) = 1096
b = 7.0520 (8) Å	D_x = 1.597 Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 996 reflections
$\theta = 3.0$–$26.9°$
$\mu = 0.94$ mm^{-1}

$T = 173$ K
Prism, green
$0.47 \times 0.31 \times 0.08$ mm

Data collection

Bruker APEXII
   diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan
   (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.666$, $T_{max} = 0.929$

5587 measured reflections
2360 independent reflections
1977 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.8°$
$h = -10 \rightarrow 31$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.090$
$S = 1.09$
2360 reflections
175 parameters
4 restraints
Primary atom site location: structure-invariant
   direct methods

Secondary atom site location: difference Fourier
   map
Hydrogen site location: inferred from
   neighbouring sites
H atoms treated by a mixture of independent
   and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.5684P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
Ni1	0.5000	0.30069 (5)	0.7500	0.01561 (13)
N1	0.58142 (7)	0.3092 (2)	0.73473 (13)	0.0179 (3)
O1	0.90563 (6)	0.2232 (2)	0.96391 (12)	0.0266 (3)
O2	0.96160 (6)	0.29413 (19)	0.87580 (12)	0.0242 (3)
O1W	0.46891 (6)	0.0848 (2)	0.64084 (11)	0.0199 (3)
O2W	0.53247 (6)	0.5047 (2)	0.86819 (11)	0.0208 (3)
C1	0.91223 (9)	0.2740 (3)	0.88189 (16)	0.0197 (4)
C2	0.85662 (8)	0.3156 (3)	0.78537 (16)	0.0172 (4)
C3	0.85969 (8)	0.3652 (3)	0.69003 (17)	0.0208 (4)
H3	0.8970	0.3732	0.6845	0.025*
C4	0.80768 (8)	0.4029 (3)	0.60302 (16)	0.0220 (4)
H4	0.8096	0.4366	0.5377	0.026*
C5	0.75295 (8)	0.3919 (3)	0.61016 (15)	0.0205 (4)
H5	0.7178	0.4188	0.5500	0.025*
C6	0.74932 (8)	0.3414 (3)	0.70535 (15)	0.0176 (4)
C7	0.80178 (8)	0.3043 (3)	0.79252 (16)	0.0173 (4)
H7	0.8000	0.2706	0.8580	0.021*
C8	0.69121 (8)	0.3288 (3)	0.71474 (16)	0.0173 (4)
C9	0.68658 (8)	0.3575 (3)	0.81138 (16)	0.0198 (4)
H9	0.7210	0.3856	0.8727	0.024*
C10	0.63215 (9)	0.3452 (3)	0.81801 (16)	0.0205 (4)

H10	0.6305	0.3632	0.8851	0.025*	
C11	0.58576 (9)	0.2824 (3)	0.64134 (16)	0.0199 (4)	
H11A	0.5505	0.2566	0.5811	0.024*	
C12	0.63867 (8)	0.2903 (3)	0.62852 (16)	0.0198 (4)	
H12A	0.6392	0.2694	0.5608	0.024*	
H11	0.4524 (11)	0.124 (4)	0.5786 (11)	0.048 (9)*	
H12	0.4935 (8)	0.004 (3)	0.6396 (17)	0.023 (6)*	
H21	0.5473 (10)	0.452 (3)	0.9275 (11)	0.030 (7)*	
H22	0.5096 (10)	0.591 (3)	0.869 (2)	0.043 (8)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01032 (18)	0.0186 (2)	0.0183 (2)	0.000	0.00608 (14)	0.000
N1	0.0125 (7)	0.0184 (8)	0.0222 (9)	−0.0001 (6)	0.0061 (7)	0.0022 (7)
O1	0.0189 (7)	0.0361 (9)	0.0215 (8)	−0.0012 (6)	0.0043 (6)	0.0023 (6)
O2	0.0118 (7)	0.0227 (8)	0.0362 (9)	0.0008 (5)	0.0077 (6)	0.0012 (6)
O1W	0.0155 (7)	0.0222 (8)	0.0216 (8)	0.0010 (6)	0.0071 (6)	−0.0019 (6)
O2W	0.0156 (7)	0.0225 (8)	0.0226 (8)	0.0028 (6)	0.0056 (6)	−0.0022 (6)
C1	0.0154 (9)	0.0154 (9)	0.0259 (11)	−0.0002 (7)	0.0056 (8)	−0.0042 (8)
C2	0.0149 (9)	0.0145 (9)	0.0214 (10)	−0.0026 (7)	0.0064 (8)	−0.0045 (8)
C3	0.0149 (9)	0.0215 (10)	0.0294 (11)	−0.0027 (8)	0.0122 (8)	−0.0026 (8)
C4	0.0212 (10)	0.0277 (11)	0.0202 (10)	−0.0032 (8)	0.0115 (8)	−0.0003 (9)
C5	0.0158 (9)	0.0234 (10)	0.0206 (10)	−0.0007 (8)	0.0054 (8)	−0.0002 (8)
C6	0.0135 (9)	0.0176 (9)	0.0223 (10)	−0.0020 (7)	0.0078 (8)	−0.0033 (7)
C7	0.0156 (9)	0.0179 (9)	0.0187 (9)	0.0002 (7)	0.0071 (8)	0.0002 (8)
C8	0.0149 (9)	0.0163 (9)	0.0205 (10)	0.0014 (7)	0.0069 (8)	0.0024 (7)
C9	0.0130 (9)	0.0242 (10)	0.0204 (10)	0.0011 (8)	0.0047 (8)	0.0004 (8)
C10	0.0175 (9)	0.0255 (10)	0.0198 (10)	0.0014 (8)	0.0090 (8)	0.0003 (8)
C11	0.0139 (9)	0.0237 (10)	0.0210 (10)	−0.0004 (8)	0.0057 (8)	0.0008 (8)
C12	0.0167 (10)	0.0230 (10)	0.0196 (10)	−0.0007 (8)	0.0071 (8)	−0.0007 (8)

Geometric parameters (Å, º)

Ni1—O1W	2.0627 (14)	C3—C4	1.388 (3)
Ni1—O1W^i	2.0627 (14)	C3—H3	0.9500
Ni1—O2W	2.0811 (14)	C4—C5	1.389 (3)
Ni1—O2W^i	2.0811 (14)	C4—H4	0.9500
Ni1—N1^i	2.0931 (16)	C5—C6	1.396 (3)
Ni1—N1	2.0931 (16)	C5—H5	0.9500
N1—C10	1.342 (3)	C6—C7	1.395 (3)
N1—C11	1.346 (2)	C6—C8	1.486 (3)
O1—C1	1.256 (3)	C7—H7	0.9500
O2—C1	1.256 (2)	C8—C12	1.392 (3)
O1W—H11	0.838 (10)	C8—C9	1.396 (3)
O1W—H12	0.834 (10)	C9—C10	1.378 (3)
O2W—H21	0.840 (10)	C9—H9	0.9500
O2W—H22	0.833 (10)	C10—H10	0.9500

C1—C2	1.511 (3)	C11—C12	1.380 (3)
C2—C3	1.390 (3)	C11—H11A	0.9500
C2—C7	1.391 (3)	C12—H12A	0.9500
O1W—Ni1—O1W^i	84.85 (8)	C4—C3—C2	119.26 (17)
O1W—Ni1—O2W	176.09 (6)	C4—C3—H3	120.4
O1W^i—Ni1—O2W	91.32 (6)	C2—C3—H3	120.4
O1W—Ni1—O2W^i	91.32 (6)	C3—C4—C5	120.92 (18)
O1W^i—Ni1—O2W^i	176.09 (6)	C3—C4—H4	119.5
O2W—Ni1—O2W^i	92.51 (8)	C5—C4—H4	119.5
O1W—Ni1—N1^i	90.11 (6)	C4—C5—C6	120.31 (18)
O1W^i—Ni1—N1^i	92.32 (6)	C4—C5—H5	119.8
O2W—Ni1—N1^i	89.24 (6)	C6—C5—H5	119.8
O2W^i—Ni1—N1^i	88.48 (6)	C7—C6—C5	118.45 (17)
O1W—Ni1—N1	92.32 (6)	C7—C6—C8	120.35 (17)
O1W^i—Ni1—N1	90.11 (6)	C5—C6—C8	121.19 (17)
O2W—Ni1—N1	88.48 (6)	C2—C7—C6	121.21 (18)
O2W^i—Ni1—N1	89.24 (6)	C2—C7—H7	119.4
N1^i—Ni1—N1	176.71 (9)	C6—C7—H7	119.4
C10—N1—C11	116.48 (16)	C12—C8—C9	116.40 (17)
C10—N1—Ni1	121.33 (13)	C12—C8—C6	122.37 (18)
C11—N1—Ni1	122.18 (13)	C9—C8—C6	121.23 (17)
Ni1—O1W—H11	113 (2)	C10—C9—C8	120.12 (18)
Ni1—O1W—H12	117.0 (16)	C10—C9—H9	119.9
H11—O1W—H12	105 (2)	C8—C9—H9	119.9
Ni1—O2W—H21	109.7 (18)	N1—C10—C9	123.49 (18)
Ni1—O2W—H22	117.8 (19)	N1—C10—H10	118.3
H21—O2W—H22	110 (2)	C9—C10—H10	118.3
O1—C1—O2	124.36 (19)	N1—C11—C12	123.49 (19)
O1—C1—C2	116.99 (17)	N1—C11—H11A	118.3
O2—C1—C2	118.65 (18)	C12—C11—H11A	118.3
C3—C2—C7	119.85 (18)	C11—C12—C8	120.03 (19)
C3—C2—C1	120.82 (17)	C11—C12—H12A	120.0
C7—C2—C1	119.32 (18)	C8—C12—H12A	120.0
O1W—Ni1—N1—C10	−144.22 (15)	C3—C2—C7—C6	0.1 (3)
O1W^i—Ni1—N1—C10	−59.36 (15)	C1—C2—C7—C6	−179.93 (17)
O2W—Ni1—N1—C10	31.96 (15)	C5—C6—C7—C2	−0.4 (3)
O2W^i—Ni1—N1—C10	124.49 (15)	C8—C6—C7—C2	−179.88 (17)
O1W—Ni1—N1—C11	36.93 (15)	C7—C6—C8—C12	−154.18 (19)
O1W^i—Ni1—N1—C11	121.79 (15)	C5—C6—C8—C12	26.4 (3)
O2W—Ni1—N1—C11	−146.89 (15)	C7—C6—C8—C9	26.6 (3)
O2W^i—Ni1—N1—C11	−54.36 (15)	C5—C6—C8—C9	−152.89 (19)
O1—C1—C2—C3	−177.38 (18)	C12—C8—C9—C10	0.6 (3)
O2—C1—C2—C3	3.5 (3)	C6—C8—C9—C10	179.95 (18)
O1—C1—C2—C7	2.7 (3)	C11—N1—C10—C9	0.8 (3)
O2—C1—C2—C7	−176.42 (17)	Ni1—N1—C10—C9	−178.14 (15)
C7—C2—C3—C4	0.0 (3)	C8—C9—C10—N1	−1.1 (3)

C1—C2—C3—C4	−179.89 (18)	C10—N1—C11—C12	0.0 (3)
C2—C3—C4—C5	0.1 (3)	Ni1—N1—C11—C12	178.89 (14)
C3—C4—C5—C6	−0.4 (3)	N1—C11—C12—C8	−0.4 (3)
C4—C5—C6—C7	0.5 (3)	C9—C8—C12—C11	0.1 (3)
C4—C5—C6—C8	179.99 (18)	C6—C8—C12—C11	−179.22 (18)

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1w—H11···O1[ii]	0.84 (1)	1.88 (1)	2.682 (2)	160 (3)
O1w—H12···O2[iii]	0.83 (1)	1.91 (1)	2.734 (2)	170 (2)
O2w—H21···O1[iv]	0.84 (1)	1.93 (1)	2.732 (2)	159 (2)
O2w—H22···O2[v]	0.83 (1)	1.88 (1)	2.711 (2)	177 (3)

Symmetry codes: (ii) *x*−1/2, −*y*+1/2, *z*−1/2; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2; (iv) −*x*+3/2, −*y*+1/2, −*z*+2; (v) *x*−1/2, *y*+1/2, *z*.