

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Tetraaquabis[3-(4-pyridyl)benzoato- κ N]-nickel(II)Qiang-Xin Wang,^a Ming-Hua Zeng^a and Seik Weng Ng^{b*}^aSchool of Chemistry & Chemical Engineering, Guangxi Normal University, 541004 Guilin 541004, People's Republic of China, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

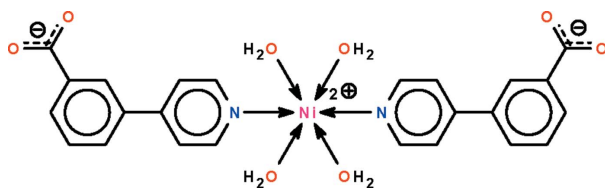
Received 26 November 2009; accepted 27 November 2009

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.090; data-to-parameter ratio = 13.5.

The Ni^{II} atom in the title compound, $[\text{Ni}(\text{C}_{12}\text{H}_8\text{NO}_2)_2(\text{H}_2\text{O})_4]$, 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

 $[\text{Ni}(\text{C}_{12}\text{H}_8\text{NO}_2)_2(\text{H}_2\text{O})_4]$ $M_r = 527.16$ 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) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.94$ mm⁻¹ $T = 173$ K

0.47 × 0.31 × 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 $I > 2\sigma(I)$ $R_{\text{int}} = 0.023$

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

 $\Delta\rho_{\text{max}} = 0.38$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.27$ 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, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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).

This work was supported by the Guangxi Graduate Education Innovation Program (2009106020703M44) and the University of Malaya.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2699).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Batten, S. R. & Harris, A. R. (2001). *Acta Cryst.* **E57**, m9–m11.
 Bruker (2004). APEX2 and SAINT. Bruker AXS inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2009). publCIF. In preparation.

supplementary materials

Acta Cryst. (2009). E65, m1708 [doi:10.1107/S1600536809051204]

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

Q.-X. Wang, M.-H. Zeng and S. W. Ng

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).

Refinement

Carbon-bound hydrogen atoms were generated geometrically and were constrained to ride on their parent atoms [C–H = 0.95 Å; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$]. 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.

Figures

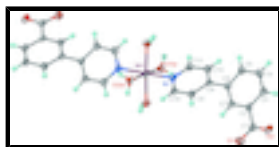


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $\text{Ni}(\text{H}_2\text{O})_4(\text{C}_{12}\text{H}_8\text{NO}_2)_2$ at the 70% probability level; hydrogen atoms are drawn as sphere of arbitrary radius.

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

Crystal data

$[\text{Ni}(\text{C}_{12}\text{H}_8\text{NO}_2)_2(\text{H}_2\text{O})_4]$

$M_r = 527.16$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 24.564$ (3) Å

$b = 7.0520$ (8) Å

$c = 13.781$ (2) Å

$\beta = 113.325$ (2)°

$V = 2192.1$ (4) Å³

$Z = 4$

$F(000) = 1096$

$D_x = 1.597$ 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

2360 independent reflections

supplementary materials

Radiation source: fine-focus sealed tube
graphite
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.666$, $T_{\max} = 0.929$
5587 measured reflections

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

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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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 (\AA , $^\circ$)

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)

supplementary materials

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 codes: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1w—H11 \cdots O1 ⁱⁱ	0.84 (1)	1.88 (1)	2.682 (2)	160 (3)
O1w—H12 \cdots O2 ⁱⁱⁱ	0.83 (1)	1.91 (1)	2.734 (2)	170 (2)
O2w—H21 \cdots O1 ^{iv}	0.84 (1)	1.93 (1)	2.732 (2)	159 (2)
O2w—H22 \cdots 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) $x-1/2, y+1/2, z$.

Fig. 1

