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Diaquabis(5-*n*-butylpyridine-2-carboxylato-κ^2N,O)nickel(II) dihydrate

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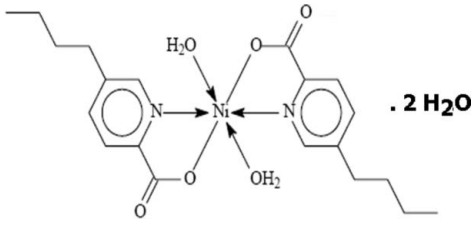
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.041; wR factor = 0.105; data-to-parameter ratio = 11.5.

The metal atom of the title compound, $[Ni(C_{10}H_{13}NO_2)_2(H_2O)_2]\cdot 2H_2O$, lies on an inversion center; it is *N*,*O*-chelated by the alkyl-substituted 2-pyridylcarboxylate group, and two water molecules complete the octahedral environment. The coordinated and solvent water molecules engage in hydrogen bonding with the acceptor O atom of the carboxylate group to furnish a two-dimensional network motif. Three atoms of the butyl group are disordered, with refined site occupancies of 0.681 (8):0.319 (8).

Related literature

See Fan *et al.* (2007); He *et al.* (2007); Okabe, Muranishi & Wada (2002) for the copper(II), cobalt(II) and iron(III) derivatives, respectively, and Okabe, Wada & Muranishi (2002) for the cadmium derivative.



Experimental

Crystal data

$[Ni(C_{10}H_{13}NO_2)_2(H_2O)_2]\cdot 2H_2O$
M_r = 487.19
Triclinic, $P\overline{1}$
a = 5.2217 (10) Å
b = 9.9614 (17) Å
c = 11.5070 (19) Å
α = 79.378 (3)°
β = 79.191 (3)°
γ = 80.152 (3)°
V = 572.05 (17) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.90 mm^{-1}
T = 293 (2) K
0.16 × 0.15 × 0.09 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.870, T_{max} = 0.924
3673 measured reflections
2022 independent reflections
1747 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 1.06
2022 reflections
176 parameters
48 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1	2.0398 (18)	Ni1—O1*W*	2.113 (2)
Ni1—N1	2.053 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O2*W*	0.85 (3)	2.04 (2)	2.784 (3)	147 (3)
O1*W*—H1*W*2···O1^{i}	0.85 (3)	1.92 (2)	2.760 (3)	179 (4)
O2*W*—H2*W*2···O2^{i}	0.85 (3)	1.94 (2)	2.792 (3)	177 (4)
O2*W*—H2*W*1···O2^{ii}	0.85 (3)	2.08 (2)	2.893 (3)	160 (3)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y, -z+1$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2115).

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supplementary materials

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Diaquabis(5-*n*-butylpyridine-2-carboxylato-κ^2*N*,*O*)nickel(II) dihydrate

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Comment

5-Butyl-pyridyl-2-carboxylic acid (fusaric acid), unlike the parent unsubstituted carboxylic acid that affords a large number of *N*,*O*-chelated metal derivates, forms only three metal carboxylates that have been characterized by crystallography (Fan *et al.*, 2007; He *et al.*, 2007). The aquacadmium derivative (Okabe, Wada & Muranishi, 2002) has two chelating carboxylate groups. The sixth coordination site is taken up by a carboxyl oxygen atom from an adjacent molecule; the water and carboxyl oxygen atoms are *cis* to each other. The copper derivative has two coordinated methanol molecules whereas the iron(III) derivative is a tris-chelated compound; (Okabe, Muranishi & Wada, 2002). The metal atom in the title nickel(II) derivative (I) (Fig. 1) lies on an inversion center, and presents two coordinated water molecules in the octahedral environment, the remaining sites being occupied by two chelating alky-substituted 2-pyridylcarboxylate groups (Table 1); the coordinated water and the lattice water molecules engage in hydrogen bonding (Table 2) with the carboxyl group to furnish a two-dimensional network motif parallel to (001) (Fig. 2).

Experimental

Nickel chloride (0.26 g, 20 mmol) and fusaric acid (0.36 g, 20 mmol) were dissolved in a small volume of hot water; aqueous sodium hydroxide (30 mmol) was added to a pH of 7. Crystals suitable for *x*-ray diffraction appeared from the solution after a few days.

Refinement

The butyl group is disordered over two positions in the β-, γ- and δ-carbon atoms, and was refined with a distance restraint of C—C 1.54 (1) Å; the vibration of the disordered atoms was restrained to be nearly isotropic. The occupation refined to a 681 (8):319 (8) ratio. The water H-atoms were located in a difference Fourier map, and were refined with distance restraints of O—H 0.85 (3) and H···H 1.39 (3) Å. The carbon-bound H atoms were treated as riding, (C—H: 0.93–0.97 Å). The displacement factors of all H atoms were tied to those of their hosts through a factor of 1.2–1.5

Figures

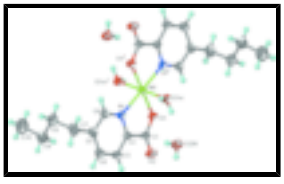

Fig. 1. Unit-cell contents of (I). Thermal ellipsoids are shown at the 50% probability level. Symmetry code (i): $1-x$, $1-y$, $1-z$. The minor disorder component of the butyl chain is not shown.

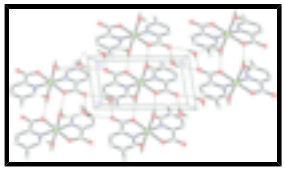

Fig. 2. Packing view of the two-dimensional structure generated by H-bonding, projected down [001].

Diaquabis(5-*n*-butylpyridine-2-carboxylato-\ κ^2*N*,*O*)nickel(II) dihydrate

Crystal data

$[Ni(C_{10}H_{13}NO_2)_2(H_2O)_2]\cdot 2H_2O$	$Z = 1$
$M_r = 487.19$	$F_{000} = 258$
Triclinic, $P\overline{1}$	$D_x = 1.414$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.2217$ (10) Å	Cell parameters from 1053 reflections
$b = 9.9614$ (17) Å	$\theta = 3.6$–24.8°
$c = 11.5070$ (19) Å	$\mu = 0.90$ mm^{-1}
$\alpha = 79.378$ (3)°	$T = 293$ (2) K
$\beta = 79.191$ (3)°	Block, green
$\gamma = 80.152$ (3)°	0.16 × 0.15 × 0.09 mm
$V = 572.05$ (17) Å^3	

Data collection

Bruker APEX area-detector diffractometer	2022 independent reflections
Radiation source: fine-focus sealed tube	1747 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 293(2)$ K	$\theta_{max} = 25.2^\circ$
φ and ω scans	$\theta_{min} = 3.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -6\rightarrow 6$
$T_{min} = 0.870$, $T_{max} = 0.924$	$k = -11\rightarrow 11$
3673 measured reflections	$l = -13\rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.0105P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2022 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}

176 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
48 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni1	0.5000	0.5000	0.5000	0.02811 (19)	
O2	0.2417 (4)	0.1359 (2)	0.5223 (2)	0.0458 (6)	
O1	0.2861 (4)	0.35853 (18)	0.47295 (17)	0.0322 (5)	
O1W	0.8216 (4)	0.41784 (19)	0.38023 (19)	0.0356 (5)	
N1	0.5972 (4)	0.3419 (2)	0.6331 (2)	0.0294 (5)	
C2	0.5090 (5)	0.2248 (3)	0.6261 (2)	0.0299 (6)	
C3	0.5740 (6)	0.1037 (3)	0.6988 (3)	0.0392 (7)	
H3	0.5154	0.0233	0.6909	0.047*	
C4	0.7286 (7)	0.1028 (3)	0.7844 (3)	0.0441 (8)	
H4	0.7744	0.0210	0.8346	0.053*	
C5	0.8152 (5)	0.2221 (3)	0.7959 (3)	0.0358 (7)	
C6	0.7457 (6)	0.3387 (3)	0.7170 (3)	0.0355 (7)	
H6	0.8052	0.4199	0.7222	0.043*	
C1	0.3318 (5)	0.2391 (3)	0.5330 (3)	0.0303 (6)	
C7	0.9731 (6)	0.2290 (4)	0.8915 (3)	0.0506 (9)	
H7A	1.1202	0.2786	0.8553	0.061*	
H7B	1.0445	0.1361	0.9237	0.061*	
O2W	0.8816 (5)	0.1367 (2)	0.3708 (2)	0.0498 (6)	
H1W1	0.785 (7)	0.345 (2)	0.363 (3)	0.075*	
H2W1	0.814 (7)	0.063 (2)	0.392 (3)	0.075*	
H1W2	0.963 (5)	0.400 (3)	0.410 (3)	0.075*	
H2W2	0.995 (6)	0.137 (3)	0.415 (3)	0.075*	
C8	0.8110 (11)	0.3002 (6)	0.9948 (4)	0.0532 (17)	0.681 (8)
H8A	0.9281	0.3142	1.0463	0.064*	0.681 (8)
H8B	0.7260	0.3900	0.9622	0.064*	0.681 (8)
C9	0.6039 (12)	0.2166 (7)	1.0683 (5)	0.0642 (19)	0.681 (8)
H9A	0.6881	0.1277	1.1032	0.077*	0.681 (8)
H9B	0.4881	0.2007	1.0170	0.077*	0.681 (8)
C10	0.442 (3)	0.2930 (18)	1.1683 (11)	0.070 (3)	0.681 (8)

H10A	0.3120	0.2386	1.2144	0.105*	0.681 (8)
H10B	0.3564	0.3802	1.1336	0.105*	0.681 (8)
H10C	0.5568	0.3079	1.2194	0.105*	0.681 (8)
C8'	0.8037 (19)	0.2008 (11)	1.0182 (8)	0.048 (3)	0.319 (8)
H8'1	0.7264	0.1173	1.0250	0.058*	0.319 (8)
H8'2	0.9166	0.1861	1.0787	0.058*	0.319 (8)
C9'	0.584 (2)	0.3203 (12)	1.0417 (10)	0.053 (4)	0.319 (8)
H9'1	0.4832	0.3413	0.9764	0.064*	0.319 (8)
H9'2	0.6628	0.4014	1.0430	0.064*	0.319 (8)
C10'	0.397 (8)	0.289 (4)	1.161 (2)	0.070 (3)	0.319 (8)
H10D	0.2659	0.3676	1.1716	0.105*	0.319 (8)
H10E	0.4956	0.2680	1.2258	0.105*	0.319 (8)
H10F	0.3125	0.2110	1.1587	0.105*	0.319 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0258 (3)	0.0260 (3)	0.0334 (3)	−0.00582 (19)	−0.0074 (2)	−0.0022 (2)
O2	0.0487 (14)	0.0328 (11)	0.0626 (15)	−0.0153 (10)	−0.0209 (12)	−0.0037 (10)
O1	0.0299 (11)	0.0282 (10)	0.0399 (12)	−0.0061 (8)	−0.0122 (9)	−0.0003 (9)
O1W	0.0306 (11)	0.0353 (11)	0.0419 (12)	−0.0047 (9)	−0.0078 (9)	−0.0070 (10)
N1	0.0265 (12)	0.0287 (12)	0.0334 (13)	−0.0058 (9)	−0.0047 (10)	−0.0048 (10)
C2	0.0242 (14)	0.0299 (15)	0.0334 (16)	−0.0036 (11)	0.0003 (12)	−0.0047 (12)
C3	0.0421 (18)	0.0279 (15)	0.0462 (19)	−0.0066 (13)	−0.0093 (15)	0.0010 (13)
C4	0.049 (2)	0.0360 (17)	0.0428 (19)	0.0013 (14)	−0.0125 (15)	0.0050 (14)
C5	0.0256 (15)	0.0441 (17)	0.0349 (16)	0.0004 (12)	−0.0045 (12)	−0.0044 (13)
C6	0.0331 (16)	0.0393 (17)	0.0353 (17)	−0.0086 (13)	−0.0044 (13)	−0.0068 (13)
C1	0.0234 (14)	0.0321 (15)	0.0366 (16)	−0.0063 (11)	−0.0027 (12)	−0.0084 (13)
C7	0.041 (2)	0.069 (2)	0.0423 (19)	−0.0061 (16)	−0.0145 (16)	−0.0036 (17)
O2W	0.0513 (15)	0.0373 (13)	0.0634 (16)	−0.0082 (10)	−0.0165 (12)	−0.0059 (11)
C8	0.060 (4)	0.062 (4)	0.044 (3)	−0.012 (3)	−0.021 (3)	−0.010 (3)
C9	0.072 (4)	0.059 (4)	0.057 (4)	−0.005 (3)	−0.005 (3)	−0.009 (3)
C10	0.074 (6)	0.080 (3)	0.056 (3)	−0.006 (3)	−0.008 (3)	−0.015 (2)
C8'	0.048 (6)	0.051 (6)	0.044 (6)	0.003 (5)	−0.016 (5)	−0.007 (5)
C9'	0.058 (7)	0.050 (7)	0.046 (6)	0.008 (5)	−0.009 (5)	−0.005 (5)
C10'	0.074 (6)	0.080 (3)	0.056 (3)	−0.006 (3)	−0.008 (3)	−0.015 (2)

Geometric parameters (Å, °)

Ni1—O1	2.0398 (18)	C7—H7A	0.9700
Ni1—O1^{i}	2.0398 (18)	C7—H7B	0.9700
Ni1—N1^{i}	2.053 (2)	O2W—H2W1	0.85 (3)
Ni1—N1	2.053 (2)	O2W—H2W2	0.85 (3)
Ni1—O1W	2.113 (2)	C8—C9	1.511 (7)
Ni1—O1W^{i}	2.113 (2)	C8—H8A	0.9700
O2—C1	1.236 (3)	C8—H8B	0.9700
O1—C1	1.269 (3)	C9—C10	1.534 (9)
O1W—H1W1	0.85 (3)	C9—H9A	0.9700

O1W—H1W2	0.85 (3)	C9—H9B	0.9700
N1—C6	1.339 (4)	C10—H10A	0.9600
N1—C2	1.347 (3)	C10—H10B	0.9600
C2—C3	1.365 (4)	C10—H10C	0.9600
C2—C1	1.515 (4)	C8'—C9'	1.528 (9)
C3—C4	1.383 (4)	C8'—H8'1	0.9700
C3—H3	0.9300	C8'—H8'2	0.9700
C4—C5	1.378 (4)	C9'—C10'	1.541 (10)
C4—H4	0.9300	C9'—H9'1	0.9700
C5—C6	1.377 (4)	C9'—H9'2	0.9700
C5—C7	1.512 (4)	C10'—H10D	0.9600
C6—H6	0.9300	C10'—H10E	0.9600
C7—C8	1.536 (5)	C10'—H10F	0.9600
O1—Ni1—O1^{i}	180.00 (10)	C8—C7—H7A	108.9
O1—Ni1—N1^{i}	99.56 (8)	C8'—C7—H7A	136.8
O1^{i}—Ni1—N1^{i}	80.44 (8)	C5—C7—H7B	108.9
O1—Ni1—N1	80.44 (8)	C8—C7—H7B	108.9
O1^{i}—Ni1—N1	99.56 (8)	H7A—C7—H7B	107.7
N1^{i}—Ni1—N1	180.000 (1)	H2W1—O2W—H2W2	109.2 (17)
O1—Ni1—O1W	89.28 (8)	C9—C8—C7	112.1 (5)
O1^{i}—Ni1—O1W	90.72 (8)	C9—C8—H8A	109.2
N1^{i}—Ni1—O1W	90.35 (8)	C7—C8—H8A	109.2
N1—Ni1—O1W	89.65 (8)	C9—C8—H8B	109.2
O1—Ni1—O1W^{i}	90.72 (8)	C7—C8—H8B	109.2
O1^{i}—Ni1—O1W^{i}	89.28 (8)	H8A—C8—H8B	107.9
N1^{i}—Ni1—O1W^{i}	89.65 (8)	C8—C9—C10	110.4 (7)
N1—Ni1—O1W^{i}	90.35 (8)	C8—C9—H9A	109.6
O1W—Ni1—O1W^{i}	180.000 (1)	C10—C9—H9A	109.6
C1—O1—Ni1	115.02 (17)	C8—C9—H9B	109.6
Ni1—O1W—H1W1	108 (3)	C10—C9—H9B	109.6
Ni1—O1W—H1W2	112 (3)	H9A—C9—H9B	108.1
H1W1—O1W—H1W2	110.0 (17)	C9—C10—H10A	109.5
C6—N1—C2	118.2 (2)	C9—C10—H10B	109.5
C6—N1—Ni1	129.16 (19)	H10A—C10—H10B	109.5
C2—N1—Ni1	112.47 (18)	C9—C10—H10C	109.5
N1—C2—C3	121.9 (3)	H10A—C10—H10C	109.5
N1—C2—C1	114.8 (2)	H10B—C10—H10C	109.5
C3—C2—C1	123.3 (3)	C9'—C8'—C7	112.1 (8)
C2—C3—C4	118.9 (3)	C9'—C8'—H8'1	109.2
C2—C3—H3	120.6	C7—C8'—H8'1	109.2
C4—C3—H3	120.6	C9'—C8'—H8'2	109.2
C5—C4—C3	120.4 (3)	C7—C8'—H8'2	109.2
C5—C4—H4	119.8	H8'1—C8'—H8'2	107.9
C3—C4—H4	119.8	C8'—C9'—C10'	113.0 (18)
C6—C5—C4	116.8 (3)	C8'—C9'—H9'1	109.0
C6—C5—C7	120.3 (3)	C10'—C9'—H9'1	109.0

C4—C5—C7	122.8 (3)	C8'—C9'—H9'2	109.0
N1—C6—C5	123.7 (3)	C10'—C9'—H9'2	109.0
N1—C6—H6	118.1	H9'1—C9'—H9'2	107.8
C5—C6—H6	118.1	C9'—C10'—H10D	109.5
O2—C1—O1	125.4 (3)	C9'—C10'—H10E	109.5
O2—C1—C2	118.3 (2)	H10D—C10'—H10E	109.5
O1—C1—C2	116.3 (2)	C9'—C10'—H10F	109.5
C5—C7—C8	113.2 (3)	H10D—C10'—H10F	109.5
C5—C7—C8'	110.1 (5)	H10E—C10'—H10F	109.5
C5—C7—H7A	108.9		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W1···O2W	0.85 (3)	2.04 (2)	2.784 (3)	147 (3)
O1W—H1W2···O1^{ii}	0.85 (3)	1.92 (2)	2.760 (3)	179 (4)
O2W—H2W2···O2^{ii}	0.85 (3)	1.94 (2)	2.792 (3)	177 (4)
O2W—H2W1···O2^{iii}	0.85 (3)	2.08 (2)	2.893 (3)	160 (3)

Symmetry codes: (ii) *x*+1, *y*, *z*; (iii) −*x*+1, −*y*, −*z*+1.

Fig. 1

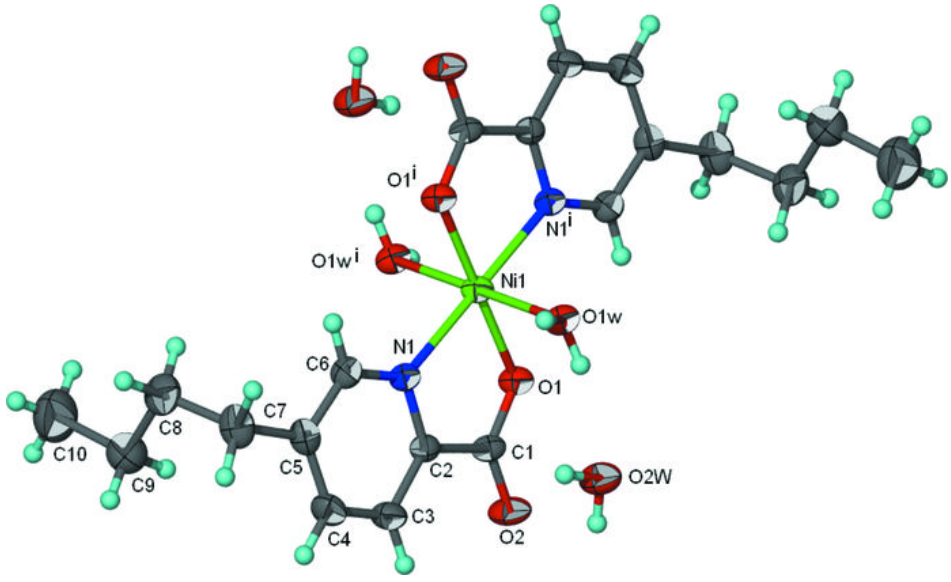

Fig. 2