metal-organic compounds

catena-Poly[[triaquanickel(II)]-*μ*-5-carboxybenzene-1,3-dicarboxylato-$κ^2O^1$:O^3]

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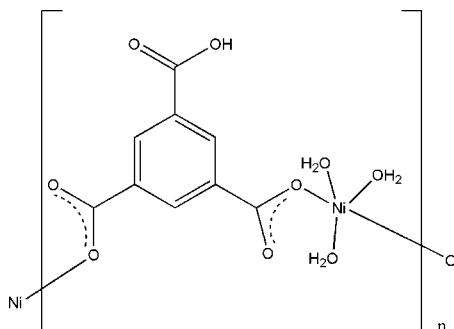Correspondence e-mail: y_xingjun01@163.com

In the title compound, [Ni(C₉H₄O₆)(H₂O)₃]$_n$, the Ni^{II} ion has a distorted NiO₅ square-pyramidal geometry, the maximum deviation from the least-squares plane formed by the basal atoms being 0.9351 (13) Å. The basal plane is formed by two O atoms from carboxylate residues of the 5-carboxybenzene-1,3-dicarboxylate ligand and by two O atoms from water molecules. The O atom of the third water molecule is axially positioned, 1.7890 (19) Å perpendicular to the basal plane. The 5-carboxybenzene-1,3-dicarboxylate ligand bridges the metal atoms, forming a polymeric chain along the *b* axis. O—H⋯O hydrogen bonds between the water molecules and carboxylate groups stabilize the crystal structure.

Related literature

For the applications and stuctures of related metal complexes of 1,3,5-benzenetricarboxylic acid, see: Xia *et al.* (2004); Modec & Brencic (2005); Wei & Han (2005); Han & Wei (2005); Wang *et al.* (2005); Che *et al.* (2008); He *et al.* (2008); Li *et al.* (2008); Gao *et al.* (2009). For bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

[Ni(C₉H₄O₆)(H₂O)₃]	V = 1101.0 (5) Å³
M_r = 320.88	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 6.838 (2) Å	μ = 1.81 mm⁻¹
b = 18.809 (5) Å	T = 296 K
c = 10.705 (3) Å	0.30 × 0.25 × 0.20 mm
β = 126.901 (14)°	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	7973 measured reflections
	1938 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	1864 reflections with $I > 2\sigma(I)$
T_{min} = 0.613, T_{max} = 0.714	R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023	173 parameters
$wR(F^2)$ = 0.102	H-atom parameters constrained
S = 1.01	$\Delta\rho_{max}$ = 0.32 e Å⁻³
1938 reflections	$\Delta\rho_{min}$ = −0.38 e Å⁻³

Table 1
Selected bond lengths (Å).

Ni1—O1	1.9292 (14)	Ni1—O3W	2.2536 (16)
Ni1—O2W	1.9781 (17)	O6—Ni1^i	1.9129 (15)
Ni1—O1W	1.9884 (18)		

Symmetry code: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3A⋯O2^{ii}	0.82	1.81	2.568 (2)	152
O1W—H1W⋯O3^{iii}	0.85	2.21	2.869 (3)	134
O1W—H2W⋯O5^{iv}	0.85	1.94	2.680 (2)	145
O2W—H4W⋯O5^v	0.85	1.89	2.715 (2)	165
O3W—H5W⋯O4^{vi}	0.85	2.03	2.778 (2)	147
O3W—H6W⋯O2^{vii}	0.85	2.49	3.068 (3)	126
O2W—H3W⋯O1^{viii}	0.85	2.34	3.123 (2)	154

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2022).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2005). *APEX2, SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Che, G. B., Liu, C. B., Liu, B., Wang, Q. W. & Xu, Z. L. (2008). *CrystEngComm*, **10**, 184–191.

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Gao, C. Y., Liu, S. X., Xie, L. H., Sun, C. Y., Cao, J. F., Ren, Y. H., Feng, D. & Su, Z. M. (2009). *CrystEngComm*, **11**, 177–182.

Han, J.-Y. & Wei, W.-Y. (2005). *Acta Cryst.* E**61**, m2242–m2243.

He, H. Y., Dai, F. N., Xie, A. P., Tong, X. & Sun, D. F. (2008). *CrystEngComm*, **10**, 1429–1435.

Li, W., Li, M. X., Yang, J. J., Shao, M. & Liu, H. J. (2008). *J. Coord. Chem.* **61**, 2715–2724.

Modec, B. & Brencic, J. V. (2005). *Eur. J. Inorg. Chem.* **21**, 4325–4334.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wang, X.-L., Liu, F.-C., Li, J.-R. & Ng, S. W. (2005). *Acta Cryst.* E**61**, m123–m125.

Wei, W.-Y. & Han, J.-Y. (2005). *Acta Cryst.* E**61**, m1792–m1793.

Xia, S. Q., Hu, S. M., Dai, J. C., Wu, X. T., Fu, Z. Y., Zhang, J. J. & Du, W. X. (2004). *Polyhedron*, **23**, 1003–1009.

supplementary materials

catena-Poly[[triaquanickel(II)]-*μ*-5-carboxybenzene-1,3-dicarboxylato-κ^2O^1:O^3]

X.-J. Yao and Q. Yuan

Comment

In recent years, the construction of metal complexes based on 1,3,5-benzenetricarboxylic acid ligand has been investigated owing to their potential applications in many fields (Xia *et al.*, 2004; Modec & Brencic, 2005; Wei & Han, 2005; Han & Wei, 2005; Wang *et al.*, 2005; Che *et al.*, 2008; He *et al.*, 2008; Li *et al.*, 2008; Gao *et al.*, 2009). In order to search for new metal complex based on 1,3,5-benzenetricarboxylic acid ligand, the title complex, (I) was synthesized and its crystal determined (Fig. 1). The bond lengths and angles are normal (Allen *et al.*, 1987). In the crystal structure, the HBTC ligands bridge the Ni atoms, forming a chain along the *b* axis (Fig. 2). O—H⋯O hydrogen bonds between the water molecules and carboxylate groups stabilize the structure.

Experimental

A mixture of $NiNO_3\cdot6H_2O$ (0.10 mmol), 1,3,5-benzenetricarboxylic acid (H3BTC, 0.10 mmol), Et_3N (0.1 ml), EtOH (2 ml) and H_2O (2 ml) was sealed in a 10 ml Tefon-lined stainless-steel reactor and then heated to 393 K for 48 h under autogenous pressure. The mixture was slowly cooled to room temperature. Green block crystals suitable for X-ray diffraction analysis were collected by filtration..

Refinement

H atoms attached to C atoms were placed in calculated positions (C—H = 0.93 Å) and refined as riding atoms and with $U_{iso}(H) = 1.2\ U_{eq}(C, N)$,respectively. The carboxy and water H atoms were located in a difference map and refined with O—H bond length from 0.82 to 0.85 Å and $U_{iso}(H) = 1.5\ U_{eq}(O)$.

Figures
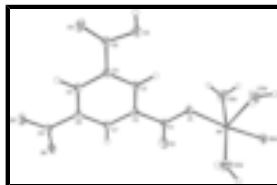

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level [symmetry codes: (A) -*x*, 1/2 + *y*, 1/2 - *z*]

Fig. 2. View of the chain structure in the title compound.

supplementary materials

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Crystal data

[Ni(C$_9$H$_4$O$_6$)(H$_2$O)$_3$]	$F(000) = 656$
$M_r = 320.88$	$D_x = 1.936$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 6807 reflections
$a = 6.838$ (2) Å	$\theta = 2.6–27.8°$
$b = 18.809$ (5) Å	$\mu = 1.81$ mm^{-1}
$c = 10.705$ (3) Å	$T = 296$ K
$\beta = 126.901$ (14)°	Block, green
$V = 1101.0$ (5) Å^3	$0.30 \times 0.25 \times 0.20$ mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1938 independent reflections
Radiation source: fine-focus sealed tube	1864 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.021$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -8 \rightarrow 8$
$T_{min} = 0.613$, $T_{max} = 0.714$	$k = -22 \rightarrow 21$
7973 measured reflections	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.023$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2 + 0.090P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.002$
1938 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
0 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.036 (3)

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	$U_{iso}*/U_{eq}$
C1	0.1199 (3)	0.63000 (10)	0.3748 (2)	0.0228 (4)
C2	0.2227 (3)	0.56454 (10)	0.4747 (2)	0.0203 (4)
C3	0.3935 (3)	0.57173 (10)	0.6364 (2)	0.0213 (4)
H3	0.4431	0.6168	0.6806	0.026*
C4	0.4892 (3)	0.51227 (11)	0.7312 (2)	0.0220 (4)
C5	0.4154 (3)	0.44487 (11)	0.6640 (2)	0.0231 (4)
H5	0.4805	0.4047	0.7273	0.028*
C6	0.2465 (3)	0.43711 (10)	0.5040 (2)	0.0196 (4)
C7	0.1480 (3)	0.49745 (10)	0.4085 (2)	0.0209 (4)
H7	0.0327	0.4925	0.3010	0.025*
C8	0.6645 (4)	0.51904 (11)	0.9031 (2)	0.0281 (5)
C9	0.1625 (3)	0.36441 (10)	0.4346 (2)	0.0214 (4)
Ni1	0.11091 (4)	0.773616 (12)	0.32089 (3)	0.01894 (19)
O1	0.2116 (3)	0.68864 (7)	0.44634 (16)	0.0274 (4)
O2	−0.0451 (3)	0.62603 (8)	0.23299 (18)	0.0408 (4)
O3	0.7481 (3)	0.58432 (8)	0.95119 (18)	0.0371 (4)
H3A	0.8391	0.5853	1.0470	0.056*
O4	0.7292 (4)	0.47053 (9)	0.99436 (19)	0.0532 (6)
O5	0.2577 (3)	0.31100 (8)	0.51934 (18)	0.0314 (4)
O6	−0.0066 (3)	0.36214 (7)	0.28913 (17)	0.0304 (4)
O1W	−0.1375 (4)	0.78863 (9)	0.3587 (2)	0.0448 (5)
H1W	−0.0823	0.8266	0.4123	0.067*
H2W	−0.1097	0.7563	0.4230	0.067*
O2W	0.3070 (3)	0.74742 (11)	0.2478 (2)	0.0430 (4)

H4W	0.4388	0.7241	0.3076	0.065*
H3W	0.3268	0.7712	0.1886	0.065*
O3W	0.4158 (3)	0.82950 (8)	0.53994 (18)	0.0394 (4)
H5W	0.4312	0.8744	0.5432	0.059*
H6W	0.5507	0.8132	0.5647	0.059*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0263 (9)	0.0193 (10)	0.0166 (9)	0.0008 (7)	0.0096 (7)	−0.0008 (8)
C2	0.0241 (9)	0.0153 (10)	0.0193 (9)	−0.0015 (7)	0.0119 (8)	0.0002 (7)
C3	0.0246 (9)	0.0140 (10)	0.0209 (9)	−0.0032 (7)	0.0113 (8)	−0.0021 (8)
C4	0.0240 (10)	0.0193 (9)	0.0181 (10)	−0.0005 (8)	0.0103 (8)	−0.0008 (8)
C5	0.0285 (9)	0.0155 (10)	0.0227 (10)	0.0014 (7)	0.0141 (8)	0.0032 (7)
C6	0.0238 (9)	0.0163 (10)	0.0192 (9)	−0.0016 (7)	0.0132 (8)	−0.0018 (7)
C7	0.0239 (9)	0.0194 (10)	0.0181 (9)	−0.0020 (8)	0.0119 (8)	−0.0025 (8)
C8	0.0316 (10)	0.0233 (11)	0.0215 (10)	−0.0010 (8)	0.0117 (9)	−0.0021 (8)
C9	0.0246 (9)	0.0170 (10)	0.0243 (10)	−0.0003 (7)	0.0157 (8)	−0.0014 (8)
Ni1	0.0249 (2)	0.0112 (3)	0.0160 (3)	0.00245 (7)	0.00978 (19)	0.00329 (7)
O1	0.0346 (8)	0.0141 (7)	0.0212 (7)	−0.0010 (5)	0.0102 (6)	0.0009 (6)
O2	0.0520 (10)	0.0239 (9)	0.0193 (8)	0.0012 (7)	0.0070 (7)	0.0010 (7)
O3	0.0508 (10)	0.0231 (8)	0.0189 (8)	−0.0080 (7)	0.0111 (7)	−0.0034 (6)
O4	0.0761 (13)	0.0245 (9)	0.0189 (8)	−0.0031 (8)	0.0072 (8)	0.0062 (7)
O5	0.0348 (8)	0.0179 (8)	0.0331 (8)	−0.0015 (6)	0.0159 (7)	0.0024 (6)
O6	0.0358 (8)	0.0196 (7)	0.0222 (7)	−0.0037 (6)	0.0102 (6)	−0.0057 (6)
O1W	0.0583 (11)	0.0283 (8)	0.0682 (13)	0.0123 (9)	0.0489 (11)	0.0156 (9)
O2W	0.0453 (10)	0.0524 (11)	0.0384 (9)	0.0201 (9)	0.0289 (9)	0.0161 (9)
O3W	0.0399 (8)	0.0279 (9)	0.0331 (8)	−0.0027 (7)	0.0126 (7)	−0.0066 (7)

Geometric parameters (Å, °)

C1—O2	1.236 (3)	C9—O5	1.244 (2)
C1—O1	1.273 (2)	C9—O6	1.266 (3)
C1—C2	1.500 (3)	Ni1—O6[i]	1.9129 (15)
C2—C7	1.386 (3)	Ni1—O1	1.9292 (14)
C2—C3	1.397 (3)	Ni1—O2W	1.9781 (17)
C3—C4	1.383 (3)	Ni1—O1W	1.9884 (18)
C3—H3	0.9300	Ni1—O3W	2.2536 (16)
C4—C5	1.394 (3)	O3—H3A	0.8200
C4—C8	1.480 (3)	O6—Ni1[ii]	1.9129 (15)
C5—C6	1.383 (3)	O1W—H1W	0.8501
C5—H5	0.9300	O1W—H2W	0.8500
C6—C7	1.400 (3)	O2W—H4W	0.8501
C6—C9	1.496 (3)	O2W—H3W	0.8500
C7—H7	0.9300	O3W—H5W	0.8501
C8—O4	1.210 (3)	O3W—H6W	0.8500
C8—O3	1.321 (3)		
O2—C1—O1	123.25 (18)	O5—C9—C6	119.96 (17)

O2—C1—C2	121.17 (17)	O6—C9—C6	115.87 (16)
O1—C1—C2	115.58 (16)	O6^{i}—Ni1—O1	174.25 (7)
C7—C2—C3	119.92 (18)	O6^{i}—Ni1—O2W	93.67 (7)
C7—C2—C1	120.79 (17)	O1—Ni1—O2W	91.48 (7)
C3—C2—C1	119.28 (17)	O6^{i}—Ni1—O1W	87.32 (7)
C4—C3—C2	120.44 (18)	O1—Ni1—O1W	88.13 (7)
C4—C3—H3	119.8	O2W—Ni1—O1W	168.69 (8)
C2—C3—H3	119.8	O6^{i}—Ni1—O3W	90.27 (6)
C3—C4—C5	119.43 (18)	O1—Ni1—O3W	86.65 (6)
C3—C4—C8	121.08 (18)	O2W—Ni1—O3W	96.01 (8)
C5—C4—C8	119.48 (17)	O1W—Ni1—O3W	95.25 (8)
C6—C5—C4	120.64 (18)	C1—O1—Ni1	117.27 (12)
C6—C5—H5	119.7	C8—O3—H3A	109.5
C4—C5—H5	119.7	C9—O6—Ni1^{ii}	120.96 (13)
C5—C6—C7	119.76 (18)	Ni1—O1W—H1W	99.8
C5—C6—C9	119.82 (18)	Ni1—O1W—H2W	105.1
C7—C6—C9	120.37 (16)	H1W—O1W—H2W	105.1
C2—C7—C6	119.80 (17)	Ni1—O2W—H4W	119.7
C2—C7—H7	120.1	Ni1—O2W—H3W	127.8
C6—C7—H7	120.1	H4W—O2W—H3W	105.1
O4—C8—O3	121.55 (19)	Ni1—O3W—H5W	121.4
O4—C8—C4	124.72 (19)	Ni1—O3W—H6W	108.0
O3—C8—C4	113.73 (18)	H5W—O3W—H6W	105.1
O5—C9—O6	124.16 (18)		
O2—C1—C2—C7	4.9 (3)	C3—C4—C8—O4	169.1 (2)
O1—C1—C2—C7	−176.04 (17)	C5—C4—C8—O4	−9.6 (3)
O2—C1—C2—C3	−174.3 (2)	C3—C4—C8—O3	−10.9 (3)
O1—C1—C2—C3	4.8 (3)	C5—C4—C8—O3	170.43 (19)
C7—C2—C3—C4	0.2 (3)	C5—C6—C9—O5	−5.2 (3)
C1—C2—C3—C4	179.35 (17)	C7—C6—C9—O5	177.22 (18)
C2—C3—C4—C5	0.6 (3)	C5—C6—C9—O6	174.46 (18)
C2—C3—C4—C8	−178.15 (18)	C7—C6—C9—O6	−3.1 (3)
C3—C4—C5—C6	−0.6 (3)	O2—C1—O1—Ni1	−7.3 (3)
C8—C4—C5—C6	178.15 (19)	C2—C1—O1—Ni1	173.69 (13)
C4—C5—C6—C7	−0.1 (3)	O2W—Ni1—O1—C1	−72.99 (16)
C4—C5—C6—C9	−177.68 (17)	O1W—Ni1—O1—C1	95.70 (16)
C3—C2—C7—C6	−0.8 (3)	O3W—Ni1—O1—C1	−168.93 (15)
C1—C2—C7—C6	179.97 (17)	O5—C9—O6—Ni1^{ii}	−6.8 (3)
C5—C6—C7—C2	0.8 (3)	C6—C9—O6—Ni1^{ii}	173.52 (12)
C9—C6—C7—C2	178.38 (17)		

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) −*x*, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O3—H3A⋯O2^{iii}	0.82	1.81	2.568 (2)	152.
O1W—H1W⋯O3^{iv}	0.85	2.21	2.869 (3)	134.

O1W—H2W···O5^{v}	0.85	1.94	2.680 (2)	145.
O2W—H4W···O5^{vi}	0.85	1.89	2.715 (2)	165.
O3W—H5W···O4^{vii}	0.85	2.03	2.778 (2)	147.
O3W—H6W···O2^{viii}	0.85	2.49	3.068 (3)	126.
O2W—H3W···O1^{ix}	0.85	2.34	3.123 (2)	154.

Symmetry codes: (iii) $x+1, y, z+1$; (iv) $x-1, -y+3/2, z-1/2$; (v) $-x, -y+1, -z+1$; (vi) $-x+1, -y+1, -z+1$; (vii) $-x+1, y+1/2, -z+3/2$; (viii) $x+1, -y+3/2, z+1/2$; (ix) $x, -y+3/2, z-1/2$.

Fig. 1
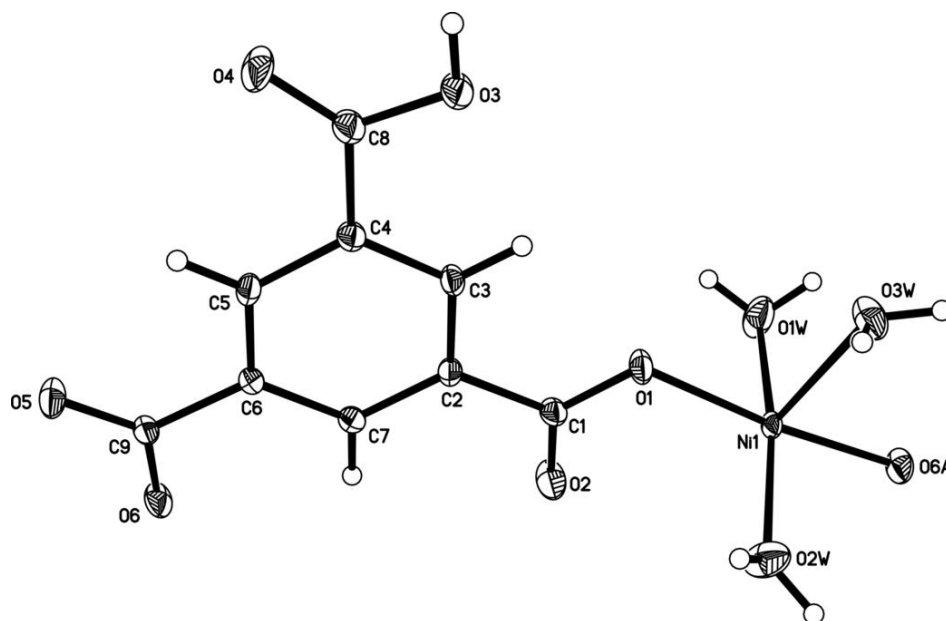
Fig. 2