Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Xing-Jun Yao[a]* and Qian Yuan[b]

[a]College of Chemistry and Chemical Engineering, Liaocheng University, 252059 Liaocheng, Shandong, People's Republic of China, and [b]Guodian Liaocheng Power Co. Ltd, 252033 Liaocheng, Shandong, People's Republic of China
Correspondence e-mail: y_xingjun01@163.com

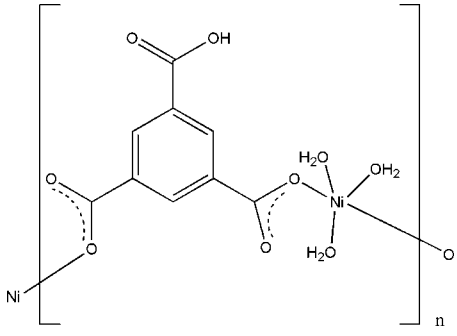
Received 15 August 2011; accepted 29 August 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.102; data-to-parameter ratio = 11.2.

In the title compound, $[Ni(C_9H_4O_6)(H_2O)_3]_n$, the Ni^{II} ion has a distorted NiO_5 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

$[Ni(C_9H_4O_6)(H_2O)_3]$
M_r = 320.88
Monoclinic, $P2_1/c$
a = 6.838 (2) Å
b = 18.809 (5) Å
c = 10.705 (3) Å
β = 126.901 (14)°
V = 1101.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.81 mm^{-1}
T = 296 K
0.30 × 0.25 × 0.20 mm

Data collection

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

Refinement

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

Table 1
Selected bond lengths (Å).

Ni1—O1	1.9292 (14)	Ni1—O3*W*	2.2536 (16)
Ni1—O2*W*	1.9781 (17)	O6—Ni1^{i}	1.9129 (15)
Ni1—O1*W*	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—H3*A*···O2^{ii}	0.82	1.81	2.568 (2)	152
O1*W*—H1*W*···O3^{iii}	0.85	2.21	2.869 (3)	134
O1*W*—H2*W*···O5^{iv}	0.85	1.94	2.680 (2)	145
O2*W*—H4*W*···O5^{v}	0.85	1.89	2.715 (2)	165
O3*W*—H5*W*···O4^{vi}	0.85	2.03	2.778 (2)	147
O3*W*—H6*W*···O2^{vii}	0.85	2.49	3.068 (3)	126
O2*W*—H3*W*···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.

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.

supporting information

Acta Cryst. (2011). E**67**, m1331–m1332 [https://doi.org/10.1107/S1600536811035227]

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

Xing-Jun Yao and Qian Yuan

S1. 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.

S2. Experimental

A mixture of $NiNO_3{\cdot}6H_2O$ (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..

S3. 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).

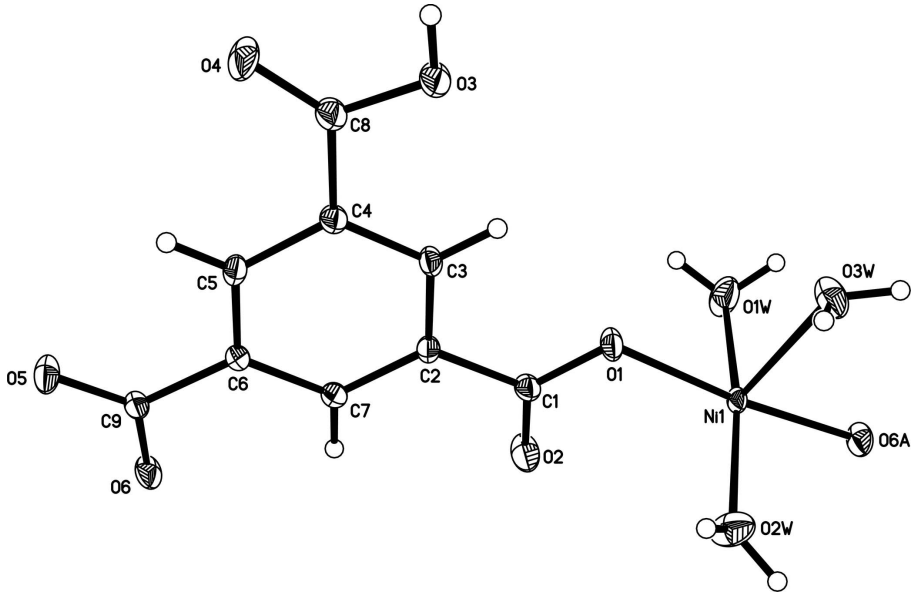


Figure 1

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

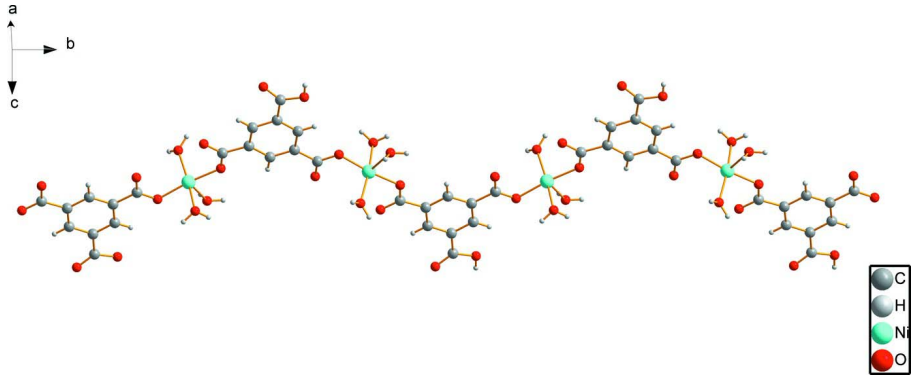


Figure 2

View of the chain structure in the title compound.

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

Crystal data

$[Ni(C_9H_4O_6)(H_2O)_3]$
M_r = 320.88
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.838 (2) Å
b = 18.809 (5) Å
c = 10.705 (3) Å
β = 126.901 (14)°
V = 1101.0 (5) Å^3
Z = 4
$F(000)$ = 656
D_x = 1.936 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6807 reflections
θ = 2.6–27.8°
μ = 1.81 mm^{-1}
T = 296 K
Block, green
0.30 × 0.25 × 0.20 mm

Data collection

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

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.102
S = 1.01
1938 reflections
173 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2 + 0.090P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 (Å²)

	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, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2^{iii}	0.82	1.81	2.568 (2)	152
O1*W*—H1*W*···O3^{iv}	0.85	2.21	2.869 (3)	134
O1*W*—H2*W*···O5^{v}	0.85	1.94	2.680 (2)	145
O2*W*—H4*W*···O5^{vi}	0.85	1.89	2.715 (2)	165
O3*W*—H5*W*···O4^{vii}	0.85	2.03	2.778 (2)	147
O3*W*—H6*W*···O2^{viii}	0.85	2.49	3.068 (3)	126
O2*W*—H3*W*···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.