metal-organic compounds

catena-Poly[[diaquanickel(II)]-*μ*-7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylato]

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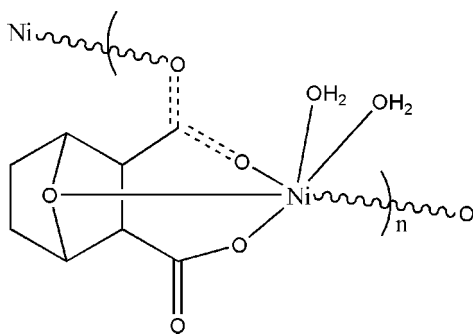Correspondence e-mail: sky51@zjnu.cn

In the crystal structure of the title compound, $[Ni(C_8H_8O_5)\text{-}(H_2O)_2]_n$, the Ni^{II} cation is in a Jahn–Teller-distorted octahedral coordination environment binding to two O atoms from water molecules, the bridging O atom of the bicyclo-heptane unit, two carboxylate O atoms from different carboxylate groups and one carboxylate O atom from a symmetry-related bridging ligand. The crystal structure is made up from layers propagating parallel to the *bc* plane.

Related literature

For the structure of the Cu(II) analogue, see: Wang *et al.* (2009).

Experimental

Crystal data

$[Ni(C_8H_8O_5)(H_2O)_2]$	$V = 975.99\ (3)$ Å^3
$M_r = 278.89$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.9145\ (2)$ Å	$\mu = 2.01$ mm^{-1}
$b = 8.6281\ (2)$ Å	$T = 296$ K
$c = 10.8581\ (2)$ Å	$0.27 \times 0.20 \times 0.10$ mm
$\beta = 107.351\ (1)°$	

Data collection

Bruker APEXII area-detector diffractometer	7972 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2213 independent reflections
	1961 reflections with $I > 2\sigma(I)$
$T_{min} = 0.618$, $T_{max} = 0.817$	$R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.059$	
$S = 1.02$	
2213 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
157 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
6 restraints	

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1$WA\cdots$O2^i	0.841 (15)	2.062 (15)	2.9027 (19)	180 (3)
O1W—H1$WB\cdots$O3^{ii}	0.822 (15)	2.135 (18)	2.7953 (17)	137 (2)
O1W—H1$WB\cdots$O1^{iii}	0.822 (15)	2.366 (19)	3.1013 (17)	149 (2)
O2W—H2$WA\cdots$O4^i	0.824 (16)	1.888 (16)	2.6967 (19)	167 (3)
O2W—H2$WB\cdots$O2^{ii}	0.802 (16)	2.341 (16)	3.135 (2)	170 (2)

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

References

Bruker (2006). *SAINT* and *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, Y.-Y., Hu, R.-D. & Wang, Y.-J. (2009). *Acta Cryst.* E**65**, m169.

supporting information

catena-Poly[[diaquanickel(II)]-*µ*-7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylato]

## Yun-Yun Wang, Rui-Ding Hu, Wen-Zhong Zhu and Qiu-Yue Lin

S1. Comment

The title compound, (I), is isostructural with the Cu(II) analogue (Wang *et al.*, 2009). In the title compound, each Ni^{II} ion is six-coordinated by two oxygen atoms from water, one bridge oxygen, two carboxylate oxygen atoms in two different carboxylate groups and one carboxylate oxygen atom in another asymmetric unit. O1, O1W, O5 and O3 lie in the equatorial plane with the torsion angle -1.121 (47)°. O2W and carboxylate oxygen atom O4 are in the axial positions. The bond angle of O2W—Ni1—O4 is 177.144 (52)°, so it forms a distorted octahedral. Owing to the binding of the bridge oxygen atom with Ni, two six-membered rings(Ni1/O4/C8/C6/C5/O5 and Ni1/O3/C7/C1/C2/O5) are created. In addition, a seven-membered ring (Ni1/O3/C7/C1/C6/C8/O4) is formed because of the coordination of carboxylate oxygen atoms O3 and O4. What's more, intermolecular O—H···O hydrogen bonds of the complex make the crystal structure more stable (Table 1).

S2. Experimental

A mixture of 1 mmol norcantharidin, 1 mmol $NiCl_2.6H_2O$ and 15 mL distilled water was sealed in a 25 mL Teflon-lined stainless vessel and heated at 443 K for 3 d, then cooled slowly to room temperature. The solution was filtered and block green crystals were obtained.

S3. Refinement

The H atoms bonded to C atoms were positioned geometrically and refined using a riding model [C—H = 0.97-0.98 Å, $U_{iso}(H) = 1.2U_{eq}(C)$]. The H atoms bonded to O atoms were located in a difference Fourier maps and refined with O—H distance restraints of 0.85 (2) and $U_{iso}(H) = 1.5U_{eq}(O)$.
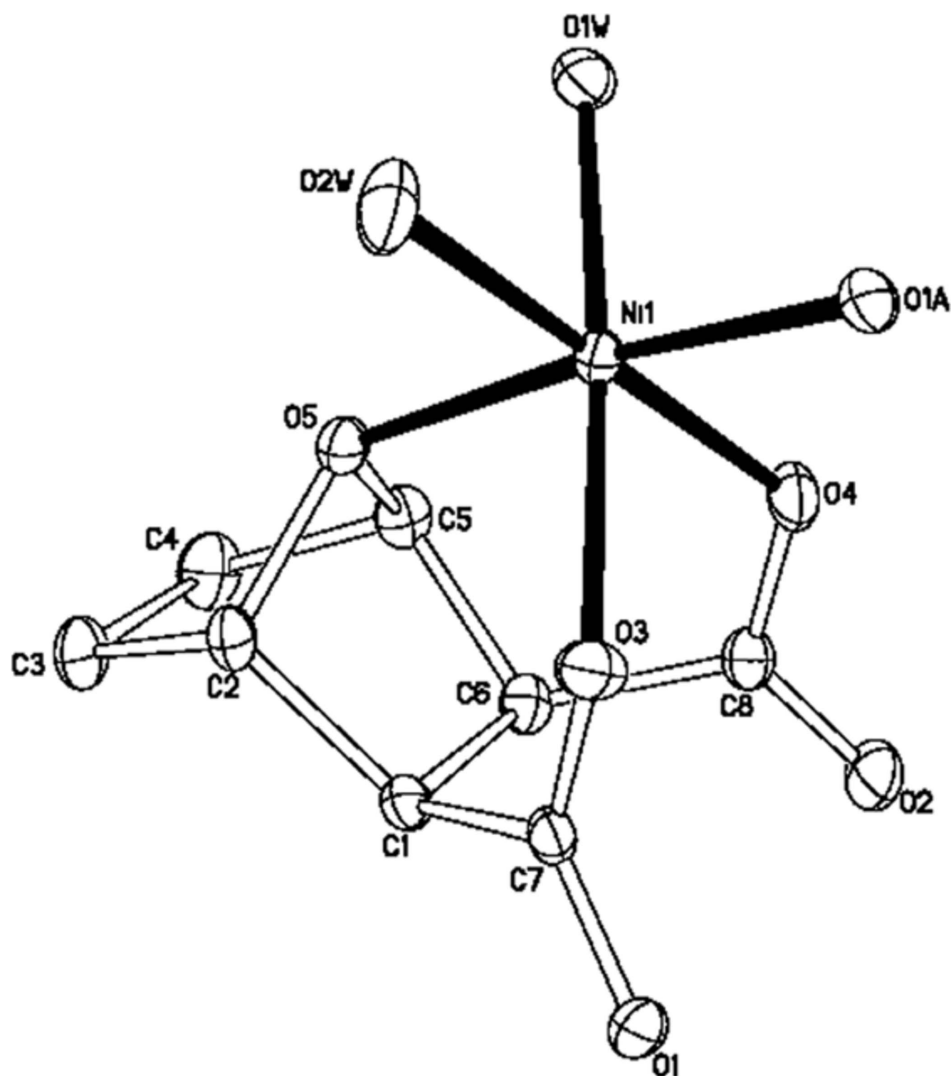
Figure 1

A view of the molecule of (I) showing the atom-labelling scheme with displacement ellipsoids drawn at the 30% probability.

***catena*-Poly[[diaquanickel(II)]-*μ*-7-oxabicyclo[2.2.1]heptane-2,3- dicarboxylato]**

Crystal data

[Ni(C$_8$H$_8$O$_5$)(H$_2$O)$_2$]	$F(000) = 576$
$M_r = 278.89$	$D_x = 1.898$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4375 reflections
$a = 10.9145$ (2) Å	$\theta = 2.0–27.5°$
$b = 8.6281$ (2) Å	$\mu = 2.01$ mm^{-1}
$c = 10.8581$ (2) Å	$T = 296$ K
$\beta = 107.351$ (1)°	Block, green
$V = 975.99$ (3) Å^3	$0.27 \times 0.20 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker APEXII area-detector diffractometer	7972 measured reflections
Radiation source: fine-focus sealed tube	2213 independent reflections
Graphite monochromator	1961 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.019$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$T_{min} = 0.618$, $T_{max} = 0.817$	$h = -13 \rightarrow 14$
	$k = -11 \rightarrow 6$
	$l = -12 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.022$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.059$	
$S = 1.02$	H atoms treated by a mixture of independent and constrained refinement
2213 reflections	
157 parameters	$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.4246P]$ where $P = (F_o^2 + 2F_c^2)/3$
6 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.30$ e Å^{-3}
	$\Delta\rho_{min} = -0.30$ e Å^{-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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.73787 (15)	−0.1019 (2)	0.71021 (15)	0.0214 (3)
H1A	0.7710	−0.2053	0.7005	0.026*
C2	0.75289 (16)	0.0067 (2)	0.60404 (16)	0.0234 (3)
H2A	0.6816	0.0008	0.5240	0.028*
C3	0.88388 (17)	−0.0131 (2)	0.58426 (19)	0.0329 (4)
H3A	0.8888	0.0402	0.5072	0.039*
H3B	0.9048	−0.1216	0.5789	0.039*
C4	0.97214 (18)	0.0632 (3)	0.7078 (2)	0.0375 (5)
H4A	1.0324	−0.0110	0.7599	0.045*
H4B	1.0192	0.1498	0.6872	0.045*
C5	0.87589 (15)	0.1175 (2)	0.77610 (17)	0.0265 (4)
H5A	0.9073	0.2034	0.8363	0.032*
C6	0.82547 (16)	−0.0207 (2)	0.83620 (16)	0.0234 (3)
H6A	0.8966	−0.0891	0.8805	0.028*
C7	0.59962 (14)	−0.1119 (2)	0.71114 (15)	0.0202 (3)

C8	0.75095 (16)	0.0292 (2)	0.92900 (15)	0.0242 (4)
O1W	0.69190 (12)	0.44064 (15)	0.74994 (13)	0.0289 (3)
H1WA	0.711 (2)	0.473 (3)	0.6846 (18)	0.043*
H1WB	0.643 (2)	0.505 (2)	0.765 (2)	0.043*
O1	0.56501 (12)	−0.23749 (14)	0.74852 (12)	0.0244 (3)
O2W	0.51314 (14)	0.29376 (19)	0.53124 (13)	0.0383 (3)
H2WA	0.557 (2)	0.322 (3)	0.486 (2)	0.057*
H2WB	0.4472 (17)	0.342 (3)	0.512 (2)	0.057*
O2	0.75780 (15)	−0.05131 (17)	1.02529 (12)	0.0389 (3)
O3	0.52811 (11)	0.00446 (14)	0.67765 (13)	0.0272 (3)
O4	0.68270 (12)	0.15134 (16)	0.89954 (11)	0.0302 (3)
O5	0.76514 (11)	0.15762 (14)	0.66735 (11)	0.0236 (3)
Ni1	0.601796 (19)	0.22532 (2)	0.714683 (19)	0.01937 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0213 (8)	0.0212 (8)	0.0245 (8)	0.0010 (6)	0.0109 (6)	−0.0017 (6)
C2	0.0216 (8)	0.0296 (9)	0.0221 (8)	−0.0007 (7)	0.0111 (6)	−0.0032 (7)
C3	0.0287 (9)	0.0413 (11)	0.0364 (10)	0.0019 (8)	0.0217 (8)	−0.0008 (8)
C4	0.0218 (9)	0.0503 (13)	0.0452 (11)	−0.0038 (8)	0.0172 (8)	0.0003 (10)
C5	0.0196 (8)	0.0318 (10)	0.0285 (9)	−0.0051 (7)	0.0080 (7)	−0.0037 (7)
C6	0.0194 (7)	0.0285 (9)	0.0225 (8)	0.0031 (7)	0.0066 (6)	0.0014 (7)
C7	0.0218 (8)	0.0217 (8)	0.0194 (8)	−0.0035 (6)	0.0098 (6)	−0.0058 (6)
C8	0.0240 (8)	0.0299 (9)	0.0181 (8)	−0.0004 (7)	0.0053 (6)	−0.0013 (7)
O1W	0.0290 (7)	0.0249 (7)	0.0357 (7)	0.0008 (5)	0.0145 (6)	−0.0014 (5)
O1	0.0236 (6)	0.0228 (6)	0.0306 (6)	−0.0026 (5)	0.0138 (5)	0.0003 (5)
O2W	0.0377 (8)	0.0566 (10)	0.0238 (7)	0.0112 (7)	0.0144 (6)	0.0103 (6)
O2	0.0555 (9)	0.0399 (8)	0.0258 (7)	0.0123 (7)	0.0187 (6)	0.0097 (6)
O3	0.0220 (6)	0.0209 (6)	0.0421 (7)	−0.0007 (5)	0.0145 (5)	−0.0011 (5)
O4	0.0364 (7)	0.0357 (8)	0.0218 (6)	0.0119 (6)	0.0137 (5)	0.0046 (5)
O5	0.0234 (6)	0.0249 (6)	0.0254 (6)	0.0001 (5)	0.0118 (5)	0.0019 (5)
Ni1	0.02056 (12)	0.02009 (13)	0.01992 (12)	0.00083 (8)	0.00979 (9)	0.00170 (8)

Geometric parameters (Å, º)

C1—C7	1.514 (2)	C6—H6A	0.9800
C1—C2	1.532 (2)	C7—O1	1.254 (2)
C1—C6	1.580 (2)	C7—O3	1.257 (2)
C1—H1A	0.9800	C8—O2	1.239 (2)
C2—O5	1.460 (2)	C8—O4	1.275 (2)
C2—C3	1.517 (2)	O1W—Ni1	2.0834 (13)
C2—H2A	0.9800	O1W—H1WA	0.841 (15)
C3—C4	1.546 (3)	O1W—H1WB	0.822 (15)
C3—H3A	0.9700	O1—Ni1[i]	2.0027 (12)
C3—H3B	0.9700	O2W—Ni1	2.0255 (13)
C4—C5	1.529 (2)	O2W—H2WA	0.824 (16)
C4—H4A	0.9700	O2W—H2WB	0.802 (16)

C4—H4B	0.9700	O3—Ni1	2.0608 (12)
C5—O5	1.457 (2)	O4—Ni1	2.0393 (12)
C5—C6	1.538 (2)	O5—Ni1	2.0809 (11)
C5—H5A	0.9800	Ni1—O1ii	2.0027 (12)
C6—C8	1.533 (2)		
C7—C1—C2	111.72 (13)	C1—C6—H6A	110.1
C7—C1—C6	111.48 (13)	O1—C7—O3	124.24 (14)
C2—C1—C6	101.96 (13)	O1—C7—C1	116.61 (15)
C7—C1—H1A	110.5	O3—C7—C1	119.13 (15)
C2—C1—H1A	110.5	O2—C8—O4	123.98 (16)
C6—C1—H1A	110.5	O2—C8—C6	119.16 (16)
O5—C2—C3	102.07 (14)	O4—C8—C6	116.85 (14)
O5—C2—C1	101.93 (12)	Ni1—O1W—H1WA	110.9 (17)
C3—C2—C1	110.77 (14)	Ni1—O1W—H1WB	109.8 (17)
O5—C2—H2A	113.6	H1WA—O1W—H1WB	106.5 (19)
C3—C2—H2A	113.6	C7—O1—Ni1i	125.62 (11)
C1—C2—H2A	113.6	Ni1—O2W—H2WA	119.0 (17)
C2—C3—C4	101.47 (14)	Ni1—O2W—H2WB	122.3 (17)
C2—C3—H3A	111.5	H2WA—O2W—H2WB	109 (2)
C4—C3—H3A	111.5	C7—O3—Ni1	120.63 (11)
C2—C3—H3B	111.5	C8—O4—Ni1	123.69 (11)
C4—C3—H3B	111.5	C5—O5—C2	96.15 (13)
H3A—C3—H3B	109.3	C5—O5—Ni1	115.54 (9)
C5—C4—C3	102.17 (14)	C2—O5—Ni1	113.69 (9)
C5—C4—H4A	111.3	O1ii—Ni1—O2W	87.33 (6)
C3—C4—H4A	111.3	O1ii—Ni1—O4	90.36 (5)
C5—C4—H4B	111.3	O2W—Ni1—O4	177.14 (6)
C3—C4—H4B	111.3	O1ii—Ni1—O3	82.07 (5)
H4A—C4—H4B	109.2	O2W—Ni1—O3	91.92 (6)
O5—C5—C4	101.75 (14)	O4—Ni1—O3	86.09 (5)
O5—C5—C6	102.27 (13)	O1ii—Ni1—O5	172.30 (5)
C4—C5—C6	110.67 (16)	O2W—Ni1—O5	91.84 (5)
O5—C5—H5A	113.7	O4—Ni1—O5	90.22 (5)
C4—C5—H5A	113.7	O3—Ni1—O5	90.31 (5)
C6—C5—H5A	113.7	O1ii—Ni1—O1W	103.17 (5)
C8—C6—C5	112.87 (15)	O2W—Ni1—O1W	88.95 (6)
C8—C6—C1	113.00 (13)	O4—Ni1—O1W	93.22 (5)
C5—C6—C1	100.21 (13)	O3—Ni1—O1W	174.73 (5)
C8—C6—H6A	110.1	O5—Ni1—O1W	84.46 (5)
C5—C6—H6A	110.1		
C7—C1—C2—O5	85.11 (15)	C6—C8—O4—Ni1	28.8 (2)
C6—C1—C2—O5	−34.06 (14)	C4—C5—O5—C2	56.04 (15)
C7—C1—C2—C3	−166.92 (14)	C6—C5—O5—C2	−58.43 (14)
C6—C1—C2—C3	73.92 (16)	C4—C5—O5—Ni1	176.00 (11)
O5—C2—C3—C4	35.96 (17)	C6—C5—O5—Ni1	61.53 (14)
C1—C2—C3—C4	−71.93 (18)	C3—C2—O5—C5	−57.44 (14)

C2—C3—C4—C5	−1.3 (2)	C1—C2—O5—C5	57.12 (13)
C3—C4—C5—O5	−33.65 (19)	C3—C2—O5—Ni1	−178.84 (10)
C3—C4—C5—C6	74.44 (18)	C1—C2—O5—Ni1	−64.27 (13)
O5—C5—C6—C8	−84.19 (16)	C8—O4—Ni1—O1ii	133.87 (14)
C4—C5—C6—C8	168.06 (15)	C8—O4—Ni1—O2W	97.8 (11)
O5—C5—C6—C1	36.27 (15)	C8—O4—Ni1—O3	51.85 (14)
C4—C5—C6—C1	−71.48 (16)	C8—O4—Ni1—O5	−38.45 (14)
C7—C1—C6—C8	−0.2 (2)	C8—O4—Ni1—O1W	−122.91 (14)
C2—C1—C6—C8	119.17 (15)	C7—O3—Ni1—O1ii	−141.13 (13)
C7—C1—C6—C5	−120.54 (14)	C7—O3—Ni1—O2W	131.83 (12)
C2—C1—C6—C5	−1.20 (15)	C7—O3—Ni1—O4	−50.22 (12)
C2—C1—C7—O1	149.60 (15)	C7—O3—Ni1—O5	39.98 (12)
C6—C1—C7—O1	−97.04 (17)	C7—O3—Ni1—O1W	32.4 (6)
C2—C1—C7—O3	−31.8 (2)	C5—O5—Ni1—O1ii	−105.7 (4)
C6—C1—C7—O3	81.59 (18)	C2—O5—Ni1—O1ii	4.1 (4)
C5—C6—C8—O2	−145.66 (17)	C5—O5—Ni1—O2W	170.59 (12)
C1—C6—C8—O2	101.50 (19)	C2—O5—Ni1—O2W	−79.56 (11)
C5—C6—C8—O4	35.8 (2)	C5—O5—Ni1—O4	−11.38 (12)
C1—C6—C8—O4	−77.0 (2)	C2—O5—Ni1—O4	98.46 (11)
O3—C7—O1—Ni1i	−8.3 (2)	C5—O5—Ni1—O3	−97.47 (11)
C1—C7—O1—Ni1i	170.29 (10)	C2—O5—Ni1—O3	12.37 (10)
O1—C7—O3—Ni1	145.58 (13)	C5—O5—Ni1—O1W	81.83 (12)
C1—C7—O3—Ni1	−32.95 (18)	C2—O5—Ni1—O1W	−168.33 (11)
O2—C8—O4—Ni1	−149.69 (15)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O2iii	0.84 (2)	2.06 (2)	2.9027 (19)	180 (3)
O1*W*—H1*WB*···O3ii	0.82 (2)	2.14 (2)	2.7953 (17)	137 (2)
O1*W*—H1*WB*···O1iv	0.82 (2)	2.37 (2)	3.1013 (17)	149 (2)
O2*W*—H2*WA*···O4iii	0.82 (2)	1.89 (2)	2.6967 (19)	167 (3)
O2*W*—H2*WB*···O2ii	0.80 (2)	2.34 (2)	3.135 (2)	170 (2)

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