

## Poly[propane-1,3-diammonium [diaqua-tetrakis( $\mu_4$ -benzene-1,2,4,5-tetracarboxylato)nickelate(II)] hemihydrate]

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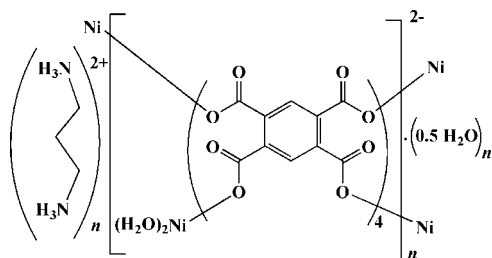
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; disorder in solvent or counterion;  $R$  factor = 0.028;  $wR$  factor = 0.073; data-to-parameter ratio = 18.4.

The reaction of nickel(II) nitrate hexahydrate with the proton-transfer compound  $(\text{pnH}_2)_2(\text{btc})\cdot 2\text{H}_2\text{O}$  (where  $\text{pn} = \text{propane-1,3-diamine}$  and  $\text{btcH}_4 = \text{benzene-1,2,4,5-tetracarboxylic acid}$ ) in aqueous solution resulted in the formation of the title compound,  $\{(\text{C}_3\text{H}_{12}\text{N}_2)[\text{Ni}(\text{C}_{10}\text{H}_2\text{O}_8)(\text{H}_2\text{O})_2]\cdot 0.5\text{H}_2\text{O}\}_n$ . Each  $\text{Ni}^{2+}$  ion is situated on a crystallographic twofold rotation axis and is coordinated in a distorted octahedral geometry by six O atoms [ $\text{Ni}-\text{O} = 2.0540(15)\text{--}2.0804(11)$  Å] from two water molecules and four  $\text{btc}^{4-}$  ligands, which also act as bridging ligands between  $\text{Ni}^{2+}$  ions. In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{O}$ ,  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds and  $\text{C}-\text{H}\cdots\pi$  interactions contribute to the formation of a three-dimensional supramolecular structure. The crystal studied was an inversion twin.

### Related literature

For details of the preparation of related proton-transfer compounds, see: Aghabozorg *et al.* (2007); Aghabozorg, Ghadermazi & Attar Gharamaleki (2006). For the crystal structures of related complexes, see: Aghabozorg, Ghasemikhah *et al.* (2006); Aghabozorg, Zabihi *et al.* (2006).



### Experimental

#### Crystal data

$(\text{C}_3\text{H}_{12}\text{N}_2)[\text{Ni}(\text{C}_{10}\text{H}_2\text{O}_8)(\text{H}_2\text{O})_2]\cdot 0.5\text{H}_2\text{O}$   
 $V = 1664.63(15)$  Å<sup>3</sup>  
 $Z = 4$   
 $M_r = 430.01$   
 Orthorhombic,  $Ima2$   
 $a = 16.3724(6)$  Å  
 $b = 7.1673(4)$  Å  
 $c = 14.1857(8)$  Å  
 $\text{Mo } K\alpha$  radiation  
 $\mu = 1.23$  mm<sup>-1</sup>  
 $T = 100.0(2)$  K  
 $0.30 \times 0.30 \times 0.20$  mm

#### Data collection

Bruker SMART APEXII CCD  
 area-detector diffractometer  
 Absorption correction: multi-scan  
 (*SADABS*; Sheldrick, 2003)  
 $T_{\text{min}} = 0.710$ ,  $T_{\text{max}} = 0.792$   
 10153 measured reflections  
 2424 independent reflections  
 2353 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.073$   
 $S = 1.00$   
 2424 reflections  
 132 parameters  
 1 restraint  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.44$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.24$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 with 1113 Friedel pairs  
 Flack parameter: 0.412 (17)

**Table 1**

Hydrogen-bond geometry and  $\pi$  interactions (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{i}}$	0.91	1.91	2.819 (2)	174
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{ii}}$	0.90	2.03	2.838 (2)	149
$\text{N1}-\text{H1C}\cdots\text{O4}^{\text{iii}}$	0.92	2.09	2.985 (2)	163
$\text{O5}-\text{H5A}\cdots\text{O4}^{\text{i}}$	0.90	1.82	2.668 (2)	156
$\text{O5}-\text{H5B}\cdots\text{O2}$	0.90	1.85	2.731 (2)	164
$\text{O6}-\text{H6B}\cdots\text{O4}^{\text{iii}}$	0.96	2.39	3.321 (3)	164
$\text{C7}-\text{H7A}\cdots\text{Cg}^{\text{iv}}$	0.99	2.97	3.757 (2)	137
$\text{C8}-\text{H8A}\cdots\text{Cg}^{\text{iv}}$	0.99	2.60	3.433 (2)	142

Symmetry codes: (i)  $x, -y + \frac{1}{2}, z - \frac{1}{2}$ ; (ii)  $-x + 1, y + \frac{1}{2}, z - \frac{1}{2}$ ; (iii)  $-x + 1, y - \frac{1}{2}, z - \frac{1}{2}$ ; (iv)  $x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$ . Note:  $\text{Cg}$  is the centroid of ring C1-C4/C2'/C3'.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2264).

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**supplementary materials**

*Acta Cryst.* (2007). E63, m2022-m2023 [ doi:10.1107/S1600536807030978 ]

## Poly[propane-1,3-diammonium [diaquatetrakis(<sup>4</sup>-benzene-1,2,4,5-tetracarboxylato)nickelate(II)] hemihydrate]

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### Comment

Just as there is a field of *molecular chemistry* based on the covalent bond, there is a field of *supramolecular chemistry*, the chemistry of molecular assemblies and intermolecular interactions. The importance of weak hydrogen bonds in the context of crystal engineering, molecular recognition and supramolecular chemistry have been well recognized in recent years. In this regard, we have reported cases in which proton transfer from pyridine-2,6-dicarboxylic acid (pydcH<sub>2</sub>) and benzene-1,2,4,5-tetracarboxylic acid (btcH<sub>4</sub>) to piperazine (pipz), propane-1,3-diamine and 1,10-phenanthroline (phen) resulted in the formation of novel self-assembled (pipzH<sub>2</sub>)(pydc), (pnH<sub>2</sub>)<sub>2</sub>(btc)·2H<sub>2</sub>O (Aghabozorg, *et al.*, 2007) and (phenH)<sub>4</sub>(btcH<sub>3</sub>)<sub>2</sub>(btcH<sub>2</sub>) (Aghabozorg, Ghadermazi & Gharamaleki, 2006) systems, respectively. The resulting compounds with some remaining sites as electron donors can coordinate to metallic ions (Aghabozorg, Ghasemikhah *et al.*, 2006; Aghabozorg, Zabihiet *al.*, 2006).

Here we report a new polymeric compound obtained from reaction of (pnH<sub>2</sub>)<sub>2</sub>(btc)·2H<sub>2</sub>O with nickel(II) nitrate. The crystal structure of the title polymeric compound is shown in Fig. 1. The intermolecular hydrogen bond distances are listed in the Table. The negative charge of the anionic complex is neutralized by dicationic propane-1,3-diaminium species. According to the crystal structure of (I), the coordination around Ni<sup>II</sup> is distorted octahedral. A considerable feature of the compound (I) is the presence of C—H···π stacking interactions between C—H groups of (pnH<sub>2</sub>)<sup>2+</sup> cations and aromatic rings of (btc)<sup>4-</sup> fragments. The C—H···π distances (measured to the centre of phenyl ring) are 2.60 Å for C8—H8A···Cg1 and 2.97 Å for C7—H7A···Cg1 with the angles of 142° and 137°, respectively [Cg1 is a centroid of C1—C4/C2<sup>i</sup>/C3<sup>i</sup>; symmetry code: (i) 1/2 + x, -1/2 + y, -1/2 + z]. The most important features of the crystal structure of (I) is a number of O—H···O, N—H···O and C—H···O hydrogen bonds between (pnH<sub>2</sub>)<sup>2+</sup> and [Ni(H<sub>2</sub>O)<sub>2</sub>(btc)]<sup>2-</sup> fragments and uncoordinated water molecules with D···A distances ranging from 2.668 (2) Å to 3.242 (2) Å (Table). Ion pairing, C—H···π stacking and van der Waals forces are also effective in the stabilization of the crystal structure, resulting in the formation of an interesting supramolecular structure (Fig. 2).

### Experimental

The proton-transfer compound was prepared by a reaction between propane-1,3-diamine and benzene-1,2,4,5-tetracarboxylic acid (Aghabozorg *et al.*, 2007). Starting with a 1:1 molar ratio of the reactants in THF, a puffy white precipitate was obtained. By recrystallization in an aqueous solution, pale-yellow crystals were obtained. A solution of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (143 mg, 0.5 mmol) in water (15 ml) was added to an aqueous solution of (pnH<sub>2</sub>)<sub>2</sub>(btc)·2H<sub>2</sub>O (253 mg, 1.0 mmol) in water (15 ml) in a 1:2 molar ratio. Crystals of (I) suitable for X-ray characterization were obtained after a few days at room temperature.

## Refinement

The value of the Flack parameter of 0.412 (17) indicates that the absolute structure in this case cannot be determined unambiguously due to the specific centrosymmetric arrangement of the anionic pattern. The refinement of the structure in the centrosymmetric space group *Imma* gives rise to disordering of the dication.

The hydrogen atoms of coordinate water molecules and  $-\text{NH}_3$  groups were localized in difference Fourier synthesis, but placed in idealized positions (O—H 0.90 Å, N—H 0.90–0.92 Å). C-bound H atoms were placed in calculated positions (C—H 0.95–0.99 Å). The crystalline water molecule was treated as disordered between two positions related by a mirror plane with the occupancies of 0.25 (for O6). The attached H atoms were positioned geometrically (O—H 0.96, 0.97 Å). All H atoms were refined in a riding model with  $U_{\text{iso}}(\text{H})=1.2\text{--}1.3U_{\text{eq}}$  of the parent atom.

## Figures

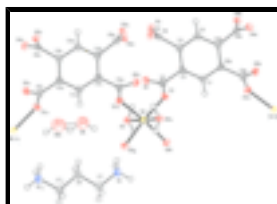


Fig. 1. The structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

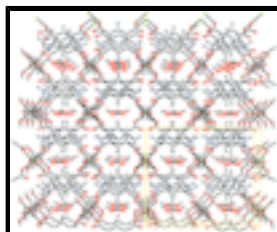


Fig. 2. The crystal packing of (I). Hydrogen bonds are shown as dashed lines.

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### Crystal data

$(\text{C}_3\text{H}_{12}\text{N}_2)[\text{Ni}(\text{C}_{10}\text{H}_2\text{O}_8)(\text{H}_2\text{O})_2] \cdot 0.5\text{H}_2\text{O}$

$M_r = 430.01$

Orthorhombic, *Ima2*

Hall symbol: I 2 -2a

$a = 16.3724$  (6) Å

$b = 7.1673$  (4) Å

$c = 14.1857$  (8) Å

$V = 1664.63$  (15) Å<sup>3</sup>

$Z = 4$

$F_{000} = 892$

$D_x = 1.716$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 6046 reflections

$\theta = 2.5\text{--}30.0^\circ$

$\mu = 1.23$  mm<sup>-1</sup>

$T = 100.0$  (2) K

Prism, colourless

$0.30 \times 0.30 \times 0.20$  mm

### Data collection

Bruker SMART APEXII CCD area-detector

2424 independent reflections

diffractometer	
Radiation source: fine-focus sealed tube	2353 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.022$
$T = 100.0(2)$ K	$\theta_{\text{max}} = 30.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -23 \rightarrow 22$
$T_{\text{min}} = 0.710$ , $T_{\text{max}} = 0.792$	$k = -10 \rightarrow 10$
10153 measured reflections	$l = -19 \rightarrow 19$

### Refinement

Refinement on $F^2$	Hydrogen site location: mixed
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.028$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.1P]$
$wR(F^2) = 0.073$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2424 reflections	$\Delta\rho_{\text{max}} = 0.44 \text{ e } \text{\AA}^{-3}$
132 parameters	$\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1113 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.412 (17)

### 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.59831 (9)	0.2336 (2)	0.13471 (12)	0.0224 (3)	
H1A	0.5517	0.2517	0.1007	0.027*	
H1B	0.6023	0.3068	0.1862	0.027*	
H1C	0.5982	0.1168	0.1618	0.027*	
C7	0.67406 (11)	0.2598 (3)	0.07757 (14)	0.0223 (4)	
H7A	0.6754	0.3883	0.0521	0.027*	
H7B	0.6737	0.1719	0.0237	0.027*	
C8	0.7500	0.2259 (4)	0.1374 (2)	0.0227 (4)	

## supplementary materials

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H8A	0.7500	0.0960	0.1611	0.027*	
H8B	0.7500	0.3113	0.1922	0.027*	
Ni1	0.5000	0.5000	0.35324 (4)	0.01460 (8)	
O1	0.40867 (9)	0.4393 (2)	0.44765 (10)	0.0196 (3)	
O2	0.46016 (8)	0.19013 (19)	0.52250 (10)	0.0223 (3)	
O3	0.41573 (8)	0.0581 (2)	0.74825 (10)	0.0186 (3)	
O4	0.43957 (8)	0.36127 (18)	0.71826 (10)	0.0217 (2)	
O5	0.54310 (7)	0.22702 (15)	0.35648 (11)	0.0198 (2)	
H5A	0.5196	0.1739	0.3058	0.024*	
H5B	0.5204	0.1936	0.4118	0.024*	
C1	0.2500	0.3097 (4)	0.50968 (18)	0.0180 (5)	
H1	0.2500	0.3499	0.4459	0.022*	
C2	0.32398 (11)	0.2805 (3)	0.55610 (13)	0.0166 (3)	
C3	0.32397 (11)	0.2229 (2)	0.65041 (13)	0.0164 (3)	
C4	0.2500	0.1941 (4)	0.69672 (17)	0.0161 (4)	
H4	0.2500	0.1544	0.7606	0.019*	
C5	0.40460 (11)	0.3053 (3)	0.50457 (12)	0.0179 (3)	
C6	0.40005 (11)	0.2128 (2)	0.70882 (13)	0.0162 (3)	
O6	0.7040 (7)	0.0338 (19)	0.3588 (14)	0.088 (4)	0.25
H6A	0.7500	-0.0326	0.3312	0.105*	0.50
H6B	0.6575	-0.0252	0.3298	0.105*	0.25

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0173 (6)	0.0238 (7)	0.0261 (7)	-0.0001 (5)	0.0008 (5)	-0.0031 (6)
C7	0.0207 (7)	0.0201 (8)	0.0261 (9)	-0.0002 (6)	0.0001 (6)	-0.0003 (7)
C8	0.0197 (10)	0.0217 (11)	0.0266 (11)	0.000	0.000	-0.0006 (9)
Ni1	0.01386 (12)	0.01624 (12)	0.01370 (12)	-0.00143 (8)	0.000	0.000
O1	0.0189 (6)	0.0204 (6)	0.0194 (6)	0.0014 (5)	0.0036 (5)	0.0037 (5)
O2	0.0193 (6)	0.0244 (6)	0.0233 (6)	0.0033 (5)	0.0031 (5)	0.0027 (5)
O3	0.0186 (6)	0.0191 (6)	0.0181 (6)	0.0010 (5)	-0.0030 (5)	0.0009 (5)
O4	0.0202 (5)	0.0209 (6)	0.0240 (6)	-0.0022 (5)	-0.0049 (5)	0.0026 (5)
O5	0.0211 (5)	0.0205 (5)	0.0177 (5)	-0.0003 (4)	-0.0020 (6)	-0.0010 (5)
C1	0.0179 (11)	0.0218 (11)	0.0145 (11)	0.000	0.000	0.0021 (9)
C2	0.0160 (7)	0.0181 (8)	0.0157 (8)	-0.0012 (6)	0.0027 (7)	0.0021 (6)
C3	0.0146 (7)	0.0164 (7)	0.0181 (8)	-0.0006 (6)	0.0001 (6)	-0.0002 (6)
C4	0.0156 (10)	0.0151 (10)	0.0175 (12)	0.000	0.000	0.0009 (8)
C5	0.0154 (7)	0.0210 (8)	0.0173 (8)	-0.0020 (6)	0.0014 (6)	-0.0016 (6)
C6	0.0144 (7)	0.0199 (8)	0.0144 (7)	0.0010 (6)	0.0013 (6)	0.0005 (6)
O6	0.071 (6)	0.137 (10)	0.055 (6)	0.048 (6)	-0.004 (8)	-0.008 (8)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C7	1.493 (2)	O2—C5	1.254 (2)
N1—H1A	0.9121	O3—C6	1.268 (2)
N1—H1B	0.9011	O4—C6	1.253 (2)
N1—H1C	0.9210	O5—H5A	0.9000
C7—C8	1.525 (3)	O5—H5B	0.9011

C7—H7A	0.9900	C1—C2	1.394 (2)
C7—H7B	0.9900	C1—H1	0.9500
C8—C7 <sup>i</sup>	1.525 (3)	C2—C3	1.400 (2)
C8—H8A	0.9900	C2—C5	1.519 (3)
C8—H8B	0.9900	C3—C4	1.393 (2)
Ni1—O1	2.0540 (15)	C3—C6	1.498 (3)
Ni1—O3 <sup>ii</sup>	2.0724 (14)	C4—H4	0.9500
Ni1—O5	2.0804 (11)	O6—H6A	0.9729
O1—C5	1.257 (2)	O6—H6B	0.9631
C7—N1—H1A	112.9	O5 <sup>iii</sup> —Ni1—O5	177.46 (10)
C7—N1—H1B	107.8	C5—O1—Ni1	128.29 (13)
H1A—N1—H1B	114.0	C6—O3—Ni1 <sup>v</sup>	128.88 (12)
C7—N1—H1C	110.0	Ni1—O5—H5A	103.6
H1A—N1—H1C	110.4	Ni1—O5—H5B	97.4
H1B—N1—H1C	101.0	H5A—O5—H5B	114.0
N1—C7—C8	110.80 (16)	C2 <sup>vi</sup> —C1—C2	120.6 (2)
N1—C7—H7A	109.5	C2—C1—H1	119.7
C8—C7—H7A	109.5	C1—C2—C3	119.7 (2)
N1—C7—H7B	109.5	C1—C2—C5	120.65 (17)
C8—C7—H7B	109.5	C3—C2—C5	119.63 (19)
H7A—C7—H7B	108.1	C4—C3—C2	119.6 (2)
C7—C8—C7 <sup>i</sup>	109.3 (2)	C4—C3—C6	117.06 (17)
C7—C8—H8A	109.8	C2—C3—C6	122.88 (19)
C7—C8—H8B	109.8	C3 <sup>vi</sup> —C4—C3	120.8 (2)
H8A—C8—H8B	108.3	C3—C4—H4	119.6
O1 <sup>iii</sup> —Ni1—O1	98.61 (9)	O2—C5—O1	126.49 (17)
O1—Ni1—O3 <sup>ii</sup>	86.64 (5)	O2—C5—C2	117.11 (16)
O1—Ni1—O3 <sup>iv</sup>	174.74 (7)	O1—C5—C2	116.40 (17)
O3 <sup>ii</sup> —Ni1—O3 <sup>iv</sup>	88.12 (8)	O4—C6—O3	126.22 (17)
O1—Ni1—O5 <sup>iii</sup>	86.44 (6)	O4—C6—C3	116.58 (16)
O1—Ni1—O5	91.91 (6)	O3—C6—C3	117.07 (16)
O3 <sup>ii</sup> —Ni1—O5	93.02 (6)	H6A—O6—H6B	103.0
O3 <sup>iv</sup> —Ni1—O5	88.81 (6)		
N1—C7—C8—C7 <sup>i</sup>	-178.27 (13)	Ni1—O1—C5—O2	8.0 (3)
O1 <sup>iii</sup> —Ni1—O1—C5	-66.36 (16)	Ni1—O1—C5—C2	-171.57 (12)
O3 <sup>ii</sup> —Ni1—O1—C5	113.24 (18)	C1—C2—C5—O2	-140.7 (2)
O5 <sup>iii</sup> —Ni1—O1—C5	-157.75 (17)	C3—C2—C5—O2	37.6 (2)
O5—Ni1—O1—C5	20.32 (17)	C1—C2—C5—O1	39.0 (3)
C2 <sup>vi</sup> —C1—C2—C3	-0.6 (4)	C3—C2—C5—O1	-142.72 (16)
C2 <sup>vi</sup> —C1—C2—C5	177.70 (17)	Ni1 <sup>v</sup> —O3—C6—O4	8.7 (3)
C1—C2—C3—C4	0.5 (2)	Ni1 <sup>v</sup> —O3—C6—C3	-166.96 (12)
C5—C2—C3—C4	-177.8 (2)	C4—C3—C6—O4	-113.8 (2)
C1—C2—C3—C6	-171.7 (2)	C2—C3—C6—O4	58.6 (2)
C5—C2—C3—C6	10.0 (2)	C4—C3—C6—O3	62.3 (2)



## supplementary materials

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C2—C3—C4—C3 <sup>vi</sup>	-0.3 (3)	C2—C3—C6—O3	-125.31 (17)
C6—C3—C4—C3 <sup>vi</sup>	172.29 (15)		

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

### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

<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>
N1—H1A $\cdots$ O2 <sup>ii</sup>	0.91	1.91	2.819 (2)	174
N1—H1B $\cdots$ O3 <sup>iv</sup>	0.90	2.03	2.838 (2)	149
N1—H1C $\cdots$ O4 <sup>vii</sup>	0.92	2.09	2.985 (2)	163
O5—H5A $\cdots$ O4 <sup>ii</sup>	0.90	1.82	2.668 (2)	156
O5—H5B $\cdots$ O2	0.90	1.85	2.731 (2)	164
O6—H6B $\cdots$ O4 <sup>vii</sup>	0.96	2.39	3.321 (3)	164
C7—H7A $\cdots$ Cg(C1-C4/C2 <sup>i</sup> /C3 <sup>i</sup> ) <sup>viii</sup>	0.99	2.97	3.757 (2)	137
C8—H8A $\cdots$ Cg(C1-C4/C2 <sup>i</sup> /C3 <sup>i</sup> ) <sup>ix</sup>	0.99	2.60	3.433 (2)	142

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

Fig. 1

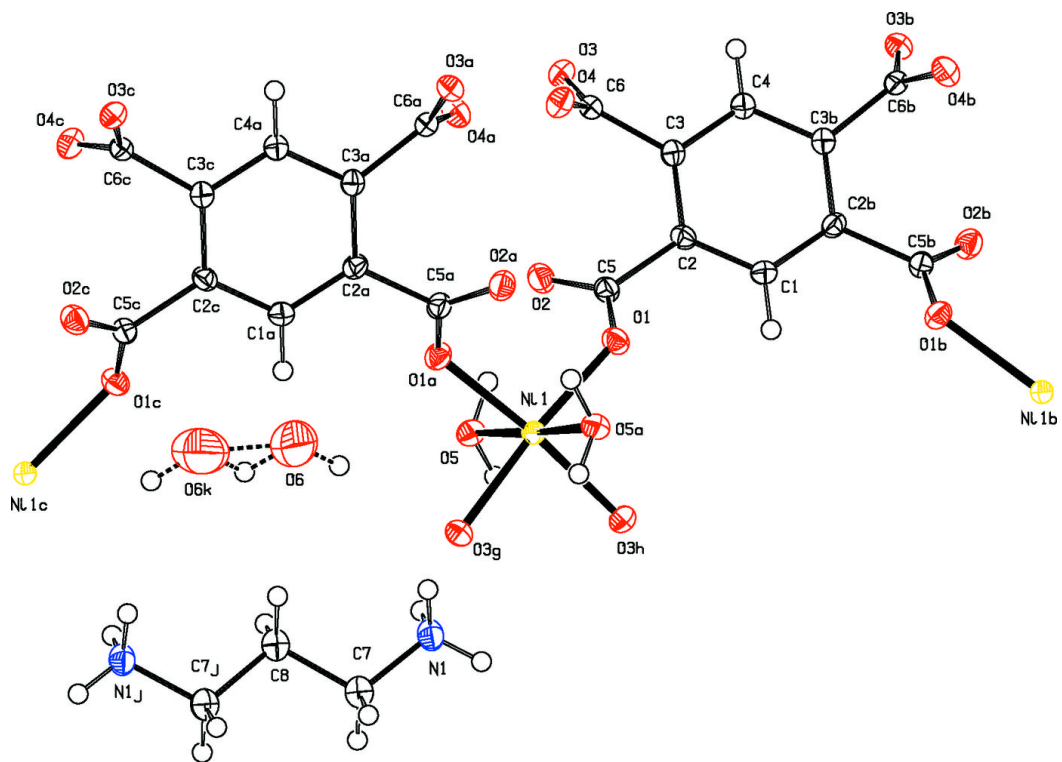


Fig. 2

