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Two nickel(II) bis[(pyridin-2-yl)methyl]-amine complexes with homophthalic and benzene-1,2,4,5-tetracarboxylic acids

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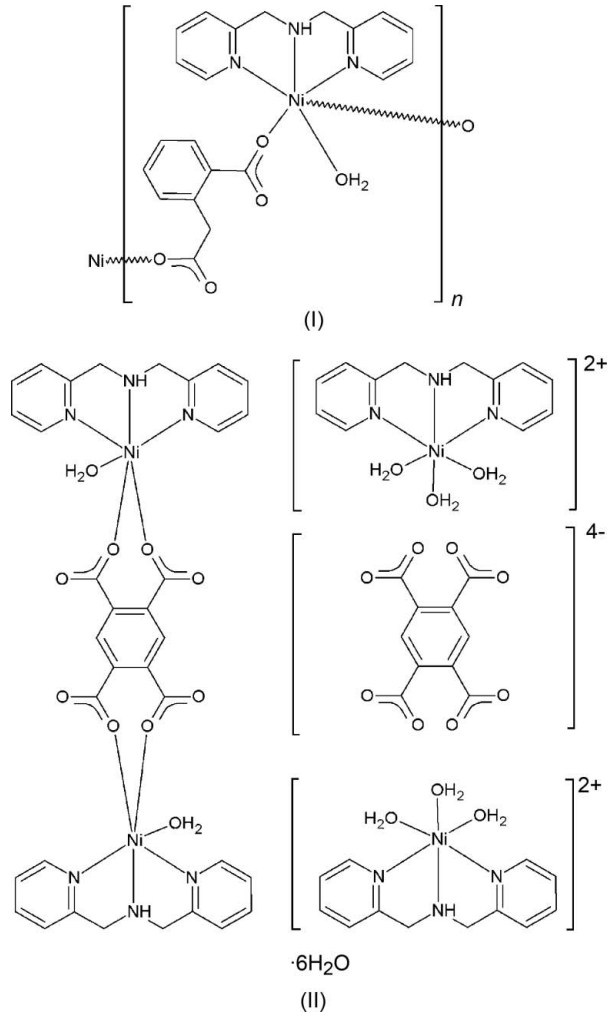
Two new Ni^{II} complexes involving the ancillary ligand bis[(pyridin-2-yl)methyl]amine (bpma) and two different carboxylate ligands, *i.e.* homophthalate [hph; systematic name: 2-(2-carboxylatophenyl)acetate] and benzene-1,2,4,5-tetracarboxylate (btc), namely *catena*-poly[[aqua{bis[(pyridin-2-yl)-methyl]amine-κ^3N,N',N''}nickel(II)]-μ-2-(2-carboxylatophenyl)aceteto-$\kappa^2O:O'$], $[Ni(C_9H_6O_4)(C_{12}H_{13}N_3)(H_2O)]_n$, and ($\mu$-benzene-1,2,4,5-tetracarboxylato-$\kappa^4O^1,O^2:O^4,O^5$)bis(aqua{bis-[(pyridin-2-yl)methyl]amine-κ^3N,N',N''}nickel(II)) bis(tri-aqua{bis[(pyridin-2-yl)methyl]amine-κ^3N,N',N''}nickel(II)) benzene-1,2,4,5-tetracarboxylate hexahydrate, $[Ni_2(C_{10}H_2O_8)-(C_{12}H_{13}N_3)_2(H_2O)_2]\cdot[Ni(C_{12}H_{13}N_3)(H_2O)_3]_2(C_{10}H_2O_8)\cdot 6H_2O$, (II), are presented. Compound (I) is a one-dimensional polymer with hph acting as a bridging ligand and with the chains linked by weak C—H$\cdots$O interactions. The structure of compound (II) is much more complex, with two independent Ni^{II} centres having different environments, one of them as part of centrosymmetric $[Ni(bpma)(H_2O)]_2(btc)$ dinuclear complexes and the other in mononuclear $[Ni(bpma)(H_2O)_3]^{2+}$ cations which (in a 2:1 ratio) provide charge balance for btc^{4-} anions. A profuse hydrogen-bonding scheme, where both coordinated and crystal water molecules play a crucial role, provides the supramolecular linkage of the different groups.

Keywords: crystal structure; hydrogen bonding; one-dimensional polymeric Ni^{II} homophthalate; dinuclear Ni^{II} benzene-1,2,4,5-tetracarboxylate.

1. Introduction

Projects on transition metal (TM) complexes incorporating organic carboxylates (OC) (and eventually also including some ancillary ligand) have proven over the years an almost inexhaustible source of crystal structures of varied complexity (zero- to three-dimensional), and many of them, initally generated out of purely academic interest, ended up being materials of practical interest (Zheng *et al.*, 2014, and references therein). Regarding magnetism (our main line of interest), some regularities linking structure and magnetic behaviour have been relatively well established over the years. For instance, *syn–syn* OC coordination modes frequently appear to lead to strong antiferromagnetism, while *syn–anti* and *anti–anti* modes tend to favour ferro- or antiferromagnetic coupling. In pursuit of our interest in TM–OC systems with potentially interesting magnetic properties, we present herein two new Ni^{II} complexes involving the ancillary ligand bis-[(pyridin-2-yl)methyl]amine (bpma) and two different OC ligands, *i.e.* homophthalate (hph) and benzene-1,2,4,5-tetra-carboxylate (btc); the complexes are $[Ni(hph)(bpma)(H_2O)]_n$, (I), and $[Ni_2(btc)(bpma)_2(H_2O)_2]\cdot[Ni(bpma)(H_2O)_3]_2(btc)\cdot\cdot 6H_2O$, (II).



(I)

(II)

2. Experimental

2.1. Synthesis and crystallization

Complexes (I) and (II) were synthesized by similar methods. The corresponding carboxylic acid [homophthalic acid for (I) and benzene-1,2,4,5-tetracarboxylic acid for (II)]

Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$[Ni(C_9H_6O_4)(C_{12}H_{13}N_3)(H_2O)]$	$[Ni_2(C_{10}H_2O_8)(C_{12}H_{13}N_3)_2(H_2O)_2]\cdot$-$[Ni(C_{12}H_{13}N_3)(H_2O)_3]_2(C_{10}H_2O_8)\cdot 6H_2O$
M_r	454.12	1784.32
Crystal system, space group	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$
Temperature (K)	150	298
a, b, c (Å)	18.233 (3), 10.4511 (14), 20.898 (3)	9.8485 (6), 10.7208 (6), 19.4924 (11)
α, β, γ (°)	90, 93.081 (2), 90	85.008 (1), 82.823 (1), 65.2930 (8)
V (Å^3)	3976.3 (9)	1853.65 (19)
Z	8	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	1.02	1.10
Crystal size (mm)	0.39 × 0.13 × 0.12	0.41 × 0.37 × 0.07
Data collection		
Diffractometer	Bruker SMART CCD area-detector diffractometer	Bruker SMART CCD area-detector diffractometer
Absorption correction	Multi-scan (*SADABS* in *SAINT-NT*; Bruker, 2002)	Multi-scan (*SADABS* in *SAINT-NT*; Bruker, 2002)
T_{min}, T_{max}	0.86, 0.88	0.65, 0.93
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	15811, 4399, 3562	15507, 7916, 6969
R_{int}	0.028	0.015
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.656	0.659
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.125, 1.43	0.032, 0.087, 1.09
No. of reflections	4399	7916
No. of parameters	280	562
No. of restraints	4	24
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.72, −0.37	0.60, −0.31

Computer programs: *SMART* (Bruker, 2001), *SAINT-NT* (Bruker, 2002), *SHELXS97* (Sheldrick, 2008), *SHELXL97* Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(1 mmol) was added slowly to an aqueous solution (20 ml) of NaOH (4 mmol or 2 mmol, respectively). Nickel acetate tetrahydrate (2 mmol) was dissolved in water (100 ml) and added to the above solution. The resulting mixture was stirred for 15 min, followed by the addition of a methanolic solution (20 ml) of bis[(pyridin-2-yl)methyl]amine (2 mmol) and the resulting solution was maintained under reflux for 4 h. Good-quality single crystals suitable for X-ray diffraction could be picked from the precipitates obtained after slow evaporation at room temperature.

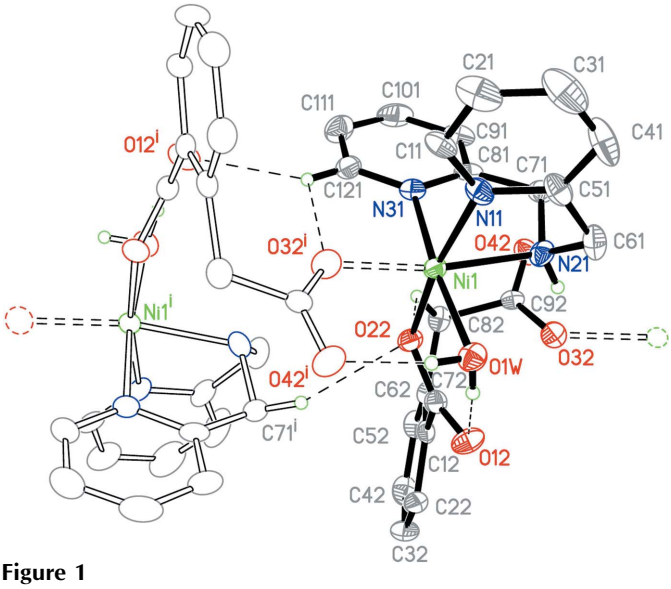


Figure 1
Two asymmetric units of (I), suggesting the way in which [010] chains are formed. Broken lines represent hydrogen bonds. [Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.]

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were originally found in a difference Fourier map but were treated differently in the refinement. H atoms attached to C atoms were repositioned in their expected positions according to the corresponding data-collection temperatures [150 (2) K for (I) and 298 (2) K for (II)] and thereafter allowed to ride, with $U_{iso}(H) = 1.2U_{eq}$(host); C—H = 0.93/0.95 Å for aromatic H atoms and C—H = 0.97/0.99 Å for methylene H atoms. H atoms attached to N and O atoms were refined with restrained X—H distances of 0.85 (1) Å and $U_{iso}(H) = 1.2U_{eq}$(N,O). In addition, H···H distances in water molecules and ligands were restrained to 1.35 (1) Å. In structure (II), an anti-bumpping restraint was imposed between atoms H3*WB* and H7*WA*(x, $y-1$, z) in order to preclude an unrealistic closeness.

3. Results and discussion

The structure of (I), $[Ni(hph)(bpma)(H_2O)]_n$, presents its Ni^{II} cations octahedrally coordinated by one aqua ligand, an N,N',N''-tridentate bpma ligand bent in its usual 'butterfly'

fashion and a μ_2-*O*:*O*′ hph ligand which bridges neighbouring metal centres into one-dimensional polymeric structures parallel to the unique *b* axis (Fig. 1). The N/O—Ni coordination distances (Table 2) are similar, spanning the rather tight range 2.062 (2)–2.106 (2) Å. Departures from regularity are more noticeable in the coordination angles [*cis* angles = 79.50 (9)–94.85 (7)° and *trans* angles = 170.50 (8)–172.97 (8)°]. The symmetry operation leading to the chain construction is the 2_1 screw along [010], through an Ni—O32^{i} (see Table 2 for symmetry code) and the repeat unit in the chain is thus the length of the short *b* axis, *i.e.* 10.4511 (14) Å. Even if not novel, the 'modest' μ_2,κ^2-bridging mode displayed by hph in (I) is rather uncommon. A search in the Cambridge Structural Database (CSD, Version 5.34 and updates; Allen, 2002) showed that the ligand usually prefers more significant involvement in cation coordination. In this context, μ_2 (κ^3 and κ^4), μ_3 (κ^2 to κ^5) and μ_4 (κ^4) modes are more usual.

There are eight relevant hydrogen-bonding interactions in the structure of (I) (Table 3), six of which are internal to the chains; among these, entries 1–3 in Table 3 are internal to a single Ni coordination polyhedron, while entries 4–6 provide for the linkage of neighbouring polyhedra within chains (Fig. 1). The compact one-dimensional arrays thus formed are disposed in a parallel fashion (Fig. 2) and interact with each other *via* two centrosymmetric hydrogen-bonding loops involving the last two hydrogen bonds in Table 3, that involving atom H22 (loop '**A**' in Fig. 2) linking chains along the [100] direction and that involving atom H52 (loop '**B**' in Fig. 2) linking chains along [001].

In contrast to the formally simple structure of (I), the structure of (II) appears more complex. An inspection of Scheme 1 discloses the most outstanding feature of the structure, *viz.* the neutral coordination complex {[Ni^{II}(bpma)(H$_2$O)]$_2$(btc)} (hereinafter denoted *C*, leftmost part of Scheme 1), co-existing with a salt formulated as [Ni^{II}(bpma)(H$_2$O)$_3$]$_2$(btc) (hereinafter denoted *S*, rightmost part of Scheme 1), the whole group being stabilized by six solvent water molecules. Substructures *C* and *S* are both centrosymmetric, with their central btc^{4-} anion lying on a centre of inversion. Fig. 3(*a*) shows a view of substructure *C*, expanded through application of the $\overline{1}$ operation, while Fig. 3(*b*) shows an equivalent view for substructure *S*.

In the case of substructure *C*, the Ni^{II} cation is octahedrally coordinated by one aqua ligand, three N atoms from a bpma ligand and two O atoms from different carboxylate groups of a btc^{4-} ligand, which serves as a bridge in the binuclear complex molecule. The case of substructure *S* is similar, the difference being that the btc^{4-} bonds to Ni^{II} are absent, being replaced by two aqua ligands, thus generating a three-membered [Ni^{II}(bpma)(H$_2$O)$_3$]$_2$(btc) group of ions.

Similar to the hph ligand in (I), the bridging mode displayed by the btc^{4-} ligand in substructure *C* of (II) is rare; a search of the CSD revealed that out of *ca* 500 complexes with coordinated btc^{4-} ligands, only three report the anion in a μ_2,κ^4-mode (all O atoms from different carboxylate groups), two with Cu [CSD refcodes GECNIL (Chaudhuri *et al.*, 1988) and

Table 2
Selected bond lengths (Å) for (I).

Ni1—N31	2.062 (2)	Ni1—N11	2.088 (2)
Ni1—O22	2.0674 (18)	Ni1—O1*W*	2.098 (2)
Ni1—O32^{i}	2.068 (2)	Ni1—N21	2.106 (2)

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

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O12	0.84 (2)	1.79 (2)	2.603 (3)	162 (2)
C82—H82*A*···O22	0.99	2.24	2.916 (3)	124
C121—H121···O32^{i}	0.95	2.55	3.110 (4)	118
O1*W*—H1*WA*···O42^{i}	0.84 (2)	1.81 (2)	2.632 (3)	163 (3)
C71—H71*B*···O22^{ii}	0.99	2.54	3.205 (4)	125
C121—H121···O12^{i}	0.95	2.33	3.114 (4)	139
C22—H22···O12^{iii}	0.95	2.49	3.309 (3)	144
C52—H52···O42^{iv}	0.95	2.48	3.191 (3)	131

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

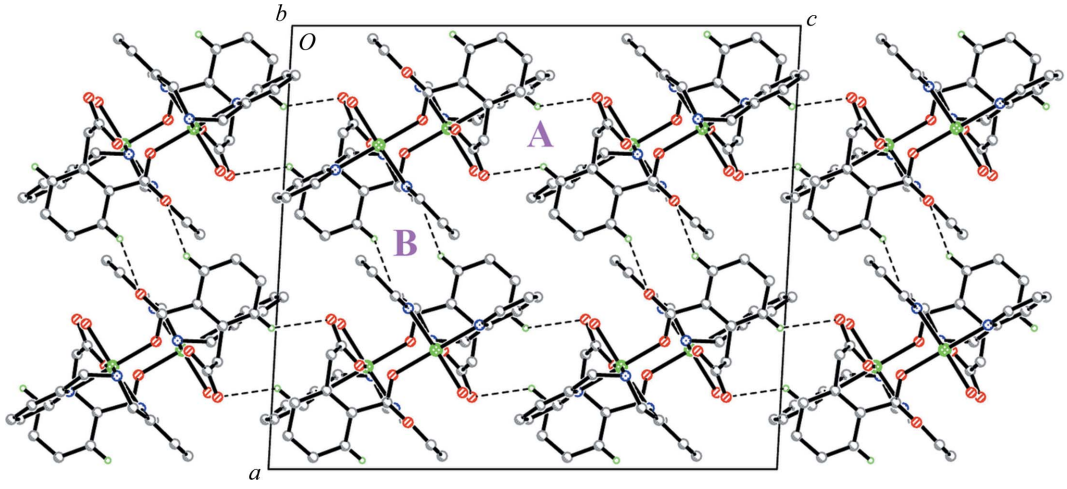


Figure 2
A packing view of (I), showing in projection the interaction between columns. Broken lines represent hydrogen bonds.

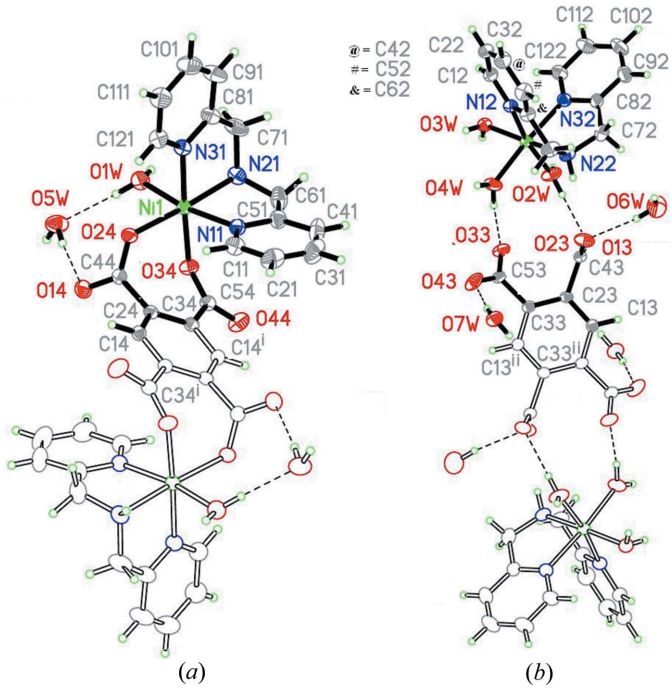


Figure 3
Molecular views of (II), showing (*a*) substructure *C* and (*b*) substructure *S* (both views expanded after application of the $\bar{1}$ operation through the inversion centre at the btc^{4-} mid-point). Displacement ellipsoids are drawn at the 40% probability level and the symmetry-independent part of both substructures is shown with full bonds. [Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+2, -z$.]

SALTIJ (Shi *et al.*, 2004)] and one with Co (GEFGAA; Xia *et al.*, 2006).

From a metrical point of view, there are no substantial differences between the Ni1 (in substructure *C*) and Ni2 (in substructure *S*) coordination polyhedra (Table 4), or that in (I), with ranges of coordination lengths and angles (listed for substructures *C* and *S*, respectively) as follows: N/O—Ni = 2.0437 (12)–2.1241 (13) and 2.0448 (13)–2.0995 (15) Å; *cis* angles = 80.95 (6)–95.65 (6) and 80.70 (6)–96.25 (6)°; *trans* angles = 168.58 (6)–177.01 (5) and 170.22 (6)–176.31 (6)°. The fundamental difference between (I) and (II) concerns the packing scheme, due to the large number of hydrogen-bonding donors/acceptors. All O—H and N—H groups are involved in hydrogen bonding, except for atom H21N in (I), which is not involved in any conventional hydrogen bond; however, it makes two rather short N21—H21N···O intermolecular contacts to O32 [H···O = 2.60 (3) Å and 145 (2)°] and O22($-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$) [H···O = 2.75 (3) Å and 118 (2)°], which could be considered as frustrated hydrogen bonds, inhibited by geometrical hindrance. Even if somewhat arbitrarily, we shall describe the interactions in a stepwise fashion, looking firstly to those interactions internal to the *C* substructures, secondly to those internal to *S* substructures, and finally those hydrogen bonds linking the substructures

Table 4
Selected bond lengths (Å) for (II).

Ni1—O24	2.0437 (12)	Ni2—O4*W*	2.0448 (13)
Ni1—N31	2.0512 (15)	Ni2—N32	2.0528 (16)
Ni1—N21	2.0764 (16)	Ni2—O2*W*	2.0668 (13)
Ni1—N11	2.0852 (15)	Ni2—O3*W*	2.0695 (13)
Ni1—O34	2.0950 (13)	Ni2—N22	2.0778 (16)
Ni1—O1*W*	2.1241 (13)	Ni2—N12	2.0995 (15)

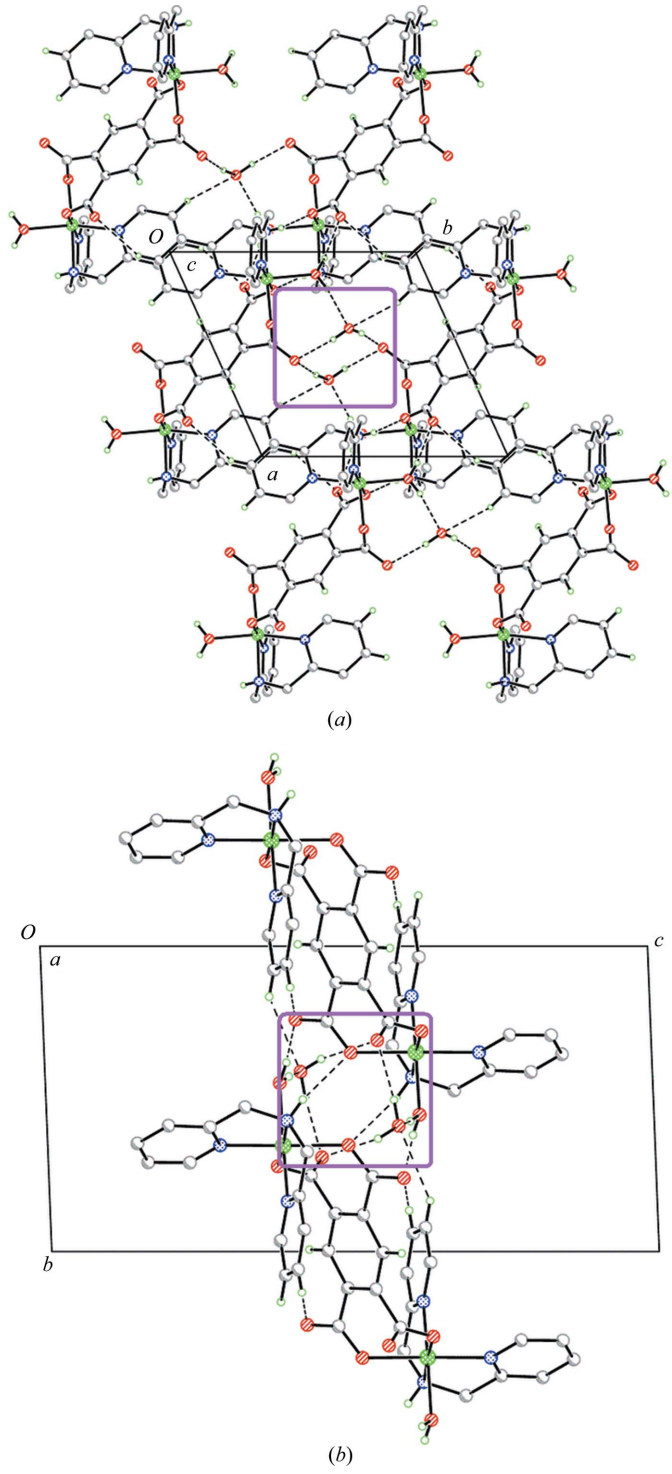


Figure 4
Partial packing views of (II), showing details of the *C* two-dimensional substructure, (*a*) projected down *c* and showing the internal hydrogen-bonding network, and (*b*) in a rotated view of the same atomic disposition now projected down *a*. Broken lines represent hydrogen bonds.

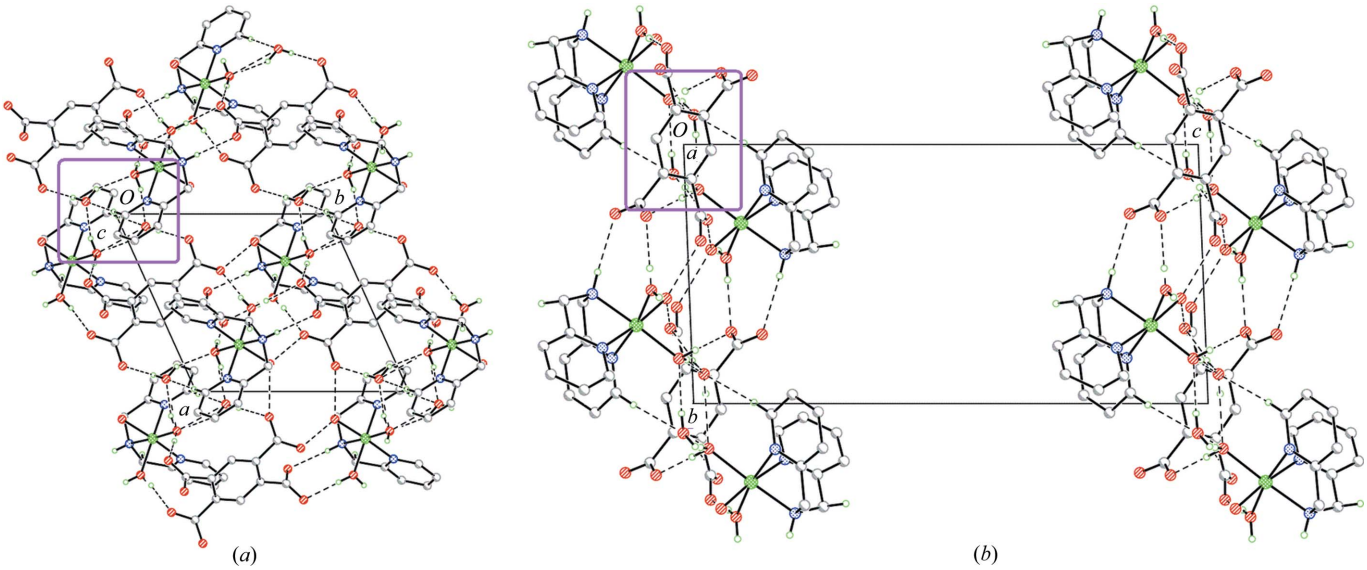


Figure 5
Partial packing views of (II), showing details of the *S* two-dimensional substructure, (*a*) projected down *c* and showing the internal hydrogen-bonding network, and (*b*) in a rotated view of the same atomic disposition now projected down *a*. Broken lines represent hydrogen bonds. The environment of water atom O7*W* is highlighted.

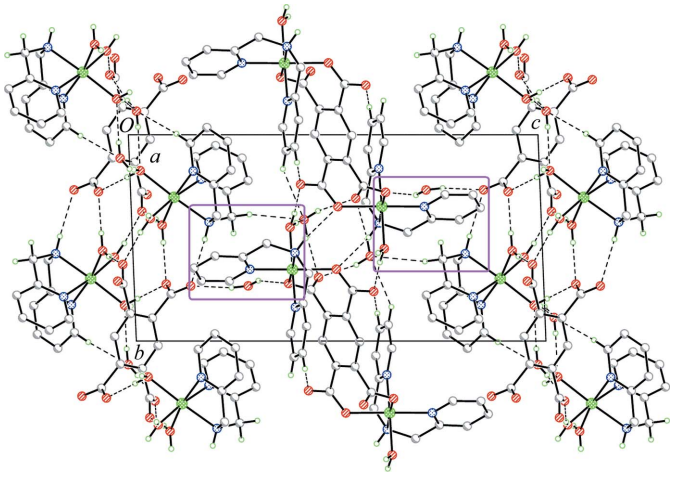


Figure 6
A full packing view of (II) projected down *a* and showing the interaction between the *C* and *S* substructures. The role of atom O5*W* is highlighted. Broken lines represent hydrogen bonds. The bridging ligands (see text for details) are highlighted.

Table 5
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21N···O34^{i}	0.85 (2)	2.15 (2)	2.978 (2)	166 (2)
O1*W*—H1*WA*···O44^{i}	0.85 (2)	1.99 (2)	2.827 (2)	170 (2)
O1*W*—H1*WB*···O5*W*	0.85 (2)	1.96 (2)	2.793 (3)	167 (2)
O5*W*—H5*WA*···O14	0.85 (1)	1.98 (1)	2.821 (2)	172 (2)
O5*W*—H5*WB*···O14^{ii}	0.84 (2)	2.02 (2)	2.852 (2)	167 (2)
C21—H21···O5*W*^{iii}	0.93	2.59	3.375 (3)	142
C31—H31···O44^{iv}	0.93	2.59	3.435 (4)	151
N22—H22N···O23	0.84 (2)	2.30 (1)	3.139 (2)	177 (3)
O2*W*—H2*WA*···O33^{v}	0.84 (2)	1.91 (2)	2.742 (2)	173 (2)
O2*W*—H2*WB*···O13	0.83 (1)	1.97 (1)	2.797 (2)	179 (3)
O3*W*—H3*WA*···O13^{v}	0.84 (2)	1.79 (2)	2.627 (2)	177 (2)
O3*W*—H3*WB*···O7*W*^{vi}	0.84 (1)	1.92 (1)	2.742 (2)	167 (2)
O4*W*—H4*WA*···O33	0.83 (2)	1.81 (2)	2.642 (2)	173 (2)
O4*W*—H4*WB*···O43^{vi}	0.83 (2)	1.89 (2)	2.677 (2)	158 (2)
O6*W*—H6*WA*···O23	0.85 (2)	2.05 (2)	2.896 (2)	171 (3)
O7*W*—H7*WA*···O3*W*^{iii}	0.82 (1)	2.07 (1)	2.816 (2)	151 (2)
O7*W*—H7*WB*···O43	0.84 (2)	1.87 (2)	2.705 (2)	174 (2)
C12—H12···O7*W*^{vii}	0.93	2.51	3.435 (3)	176
O6*W*—H6*WB*···O24	0.85 (2)	2.21 (2)	3.025 (2)	160 (3)
C72—H72*B*···O5*W*	0.97	2.60	3.490 (3)	153

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

into a three-dimensional supramolecular structure. At this stage, it is pertinent to state that each of the substructures has one hydration water molecule (O5*W* for *C* and O7*W* for *S*) fulfilling a thoroughly 'internal' role, in that they make and receive all their interactions to O atoms (and from H atoms) in the corresponding substructure, with no direct interactions with the other substructure. As we shall see, only O6*W* acts as a bridge between both substructures.

According to this, substructure *C* can be considered as formed by Ni1, bpma(1), btc(4), O1*W* and O5*W*, while substructure *S* would be composed of Ni2, bpma(2), btc(3), O2*W*, O3*W*, O4*W* and O7*W*, where numbers in parenthesis refer to the trailing numbers characterizing atoms labels in each ligand.

Table 5 presents the hydrogen bonds in (II), sorted into three blocks. The upper block (entries 1–7) contains only interactions internal to substructure *C*, which give rise to two-dimensional structures (Fig. 4*a*) parallel to (001) at $z \sim 0.50$ (Fig. 4*b*). The outstanding role of atom O5*W* (highlighted with a box) is apparent. The second block (entries 8–18) contains in turn the interactions internal to substructure *S*, also generating two-dimensional structures (Fig. 5*a*) parallel to (001), this time

at $z \sim 0, 1$ (Fig. 5*b*). Here also, hydration water molecule O7*W* (circled) is relevant for the stability of the group. Finally, the third block (entries 19–20), plus entry 15, presents the only two significant bridging hydrogen-bond interactions, the double one involving atom O6*W* and the weak nonconventional C72—H72*B*$\cdots$O5*W* hydrogen bond. Fig. 6 presents a complete [100] view of the structure, with the bridging interactions highlighted for clear identification.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: UK3094).

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supplementary materials

Acta Cryst. (2014). C**70**, 541-546 [doi:10.1107/S2053229614008134]

Two nickel(II) bis[(pyridin-2-yl)methyl]amine complexes with homophthalic and benzene-1,2,4,5-tetracarboxylic acids

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Computing details

For both compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 2002); data reduction: *SAINT-NT* (Bruker, 2002); 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: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(I) *catena*-Poly[[aqua{bis[(pyridin-2-yl)methyl]amine-κ^3N,N',N''}nickel(II)]-μ-2-(2-carboxylatophenyl)aceteto-$\kappa^2O:O'$]

Crystal data

$[Ni(C_9H_6O_4)(C_{12}H_{13}N_3)(H_2O)]$
M_r = 454.12
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 18.233 (3) Å
b = 10.4511 (14) Å
c = 20.898 (3) Å
β = 93.081 (2)°
V = 3976.3 (9) Å^3
Z = 8
$F(000)$ = 1888
D_x = 1.517 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2835 reflections
θ = 2.2–26.8°
μ = 1.02 mm^{-1}
T = 150 K
Block, blue
0.39 × 0.13 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS* in *SAINT-NT*; Bruker, 2002)
T_{min} = 0.86, T_{max} = 0.88
15811 measured reflections
4399 independent reflections
3562 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.8°, θ_{min} = 2.0°
h = −23→22
k = −13→13
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.125
S = 1.43
4399 reflections
280 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 2.6787P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.268905 (17)	0.54699 (3)	0.182666 (15)	0.01720 (13)
N11	0.32496 (12)	0.5816 (2)	0.09987 (10)	0.0220 (5)
N21	0.28959 (13)	0.7444 (2)	0.19247 (10)	0.0222 (5)
H21N	0.2611 (14)	0.785 (3)	0.2153 (12)	0.027*
N31	0.36272 (12)	0.5344 (2)	0.24201 (10)	0.0205 (5)
C11	0.35626 (14)	0.4973 (3)	0.06188 (13)	0.0245 (6)
H1A	0.3570	0.4098	0.0742	0.029*
C21	0.38752 (19)	0.5306 (3)	0.00586 (15)	0.0391 (8)
H21	0.4081	0.4675	−0.0205	0.047*
C31	0.3881 (2)	0.6591 (4)	−0.01100 (16)	0.0495 (10)
H31	0.4104	0.6858	−0.0488	0.059*
C41	0.3559 (2)	0.7475 (3)	0.02769 (14)	0.0430 (9)
H41	0.3549	0.8356	0.0164	0.052*
C51	0.32516 (16)	0.7061 (3)	0.08312 (13)	0.0260 (6)
C61	0.28692 (17)	0.7966 (3)	0.12672 (13)	0.0294 (6)
H61A	0.3113	0.8813	0.1267	0.035*
H61B	0.2352	0.8081	0.1109	0.035*
C71	0.36221 (15)	0.7644 (3)	0.22740 (13)	0.0238 (6)
H71A	0.3986	0.7900	0.1963	0.029*
H71B	0.3579	0.8355	0.2583	0.029*
C81	0.38960 (14)	0.6485 (3)	0.26261 (12)	0.0210 (5)
C91	0.44319 (15)	0.6562 (3)	0.31322 (13)	0.0284 (6)
H91	0.4603	0.7371	0.3285	0.034*
C101	0.47052 (16)	0.5456 (3)	0.34028 (14)	0.0336 (7)
H101	0.5066	0.5490	0.3748	0.040*
C111	0.44528 (17)	0.4286 (3)	0.31707 (14)	0.0323 (7)
H111	0.4652	0.3508	0.3340	0.039*
C121	0.39053 (15)	0.4277 (3)	0.26882 (13)	0.0248 (6)
H121	0.3718	0.3476	0.2540	0.030*
O12	0.10542 (10)	0.6298 (2)	0.23059 (9)	0.0275 (4)
O22	0.21205 (10)	0.54161 (17)	0.26559 (9)	0.0215 (4)
O32	0.23373 (10)	0.85118 (19)	0.33165 (9)	0.0254 (4)
O42	0.33763 (11)	0.83428 (19)	0.39241 (9)	0.0287 (4)
C12	0.12750 (14)	0.6080 (2)	0.34231 (12)	0.0187 (5)
C22	0.05291 (15)	0.5936 (3)	0.35230 (14)	0.0247 (6)
H22	0.0194	0.5791	0.3166	0.030*
C32	0.02663 (16)	0.5999 (3)	0.41350 (14)	0.0310 (7)

H32	−0.0242	0.5889	0.4197	0.037*
C42	0.07535 (17)	0.6224 (3)	0.46512 (14)	0.0308 (7)
H42	0.0583	0.6260	0.5072	0.037*
C52	0.14927 (16)	0.6398 (3)	0.45535 (13)	0.0271 (6)
H52	0.1820	0.6574	0.4912	0.033*
C62	0.17708 (14)	0.6323 (2)	0.39446 (12)	0.0188 (5)
C72	0.15014 (14)	0.5928 (2)	0.27372 (12)	0.0202 (5)
C82	0.25815 (15)	0.6559 (2)	0.38849 (12)	0.0215 (5)
H82A	0.2762	0.5963	0.3561	0.026*
H82B	0.2842	0.6354	0.4300	0.026*
C92	0.27836 (14)	0.7920 (2)	0.36970 (11)	0.0176 (5)
O1W	0.17249 (11)	0.58304 (19)	0.12623 (9)	0.0250 (4)
H1WA	0.1641 (16)	0.5069 (14)	0.1137 (14)	0.030*
H1WB	0.1416 (13)	0.598 (3)	0.1542 (11)	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01567 (19)	0.01819 (19)	0.01774 (19)	0.00132 (12)	0.00081 (12)	0.00055 (12)
N11	0.0223 (12)	0.0215 (11)	0.0223 (11)	−0.0072 (9)	0.0019 (9)	−0.0051 (9)
N21	0.0249 (12)	0.0198 (11)	0.0221 (11)	0.0041 (9)	0.0041 (9)	−0.0016 (9)
N31	0.0170 (11)	0.0245 (11)	0.0203 (11)	0.0000 (9)	0.0030 (9)	−0.0024 (9)
C11	0.0183 (13)	0.0299 (14)	0.0254 (14)	−0.0108 (12)	0.0005 (11)	−0.0017 (11)
C21	0.0360 (18)	0.052 (2)	0.0292 (16)	−0.0164 (15)	0.0055 (13)	−0.0166 (14)
C31	0.059 (2)	0.060 (2)	0.0305 (17)	−0.0253 (19)	0.0113 (16)	−0.0021 (16)
C41	0.071 (3)	0.0316 (17)	0.0268 (16)	−0.0200 (17)	0.0048 (15)	0.0074 (13)
C51	0.0303 (16)	0.0269 (14)	0.0204 (13)	−0.0110 (12)	−0.0034 (11)	0.0006 (11)
C61	0.0386 (18)	0.0196 (13)	0.0294 (15)	−0.0030 (12)	−0.0043 (12)	0.0046 (11)
C71	0.0174 (13)	0.0233 (13)	0.0308 (14)	−0.0020 (11)	0.0023 (11)	−0.0056 (11)
C81	0.0129 (12)	0.0328 (15)	0.0176 (12)	0.0005 (11)	0.0035 (9)	0.0000 (10)
C91	0.0183 (14)	0.0399 (17)	0.0271 (14)	−0.0057 (12)	0.0038 (11)	−0.0029 (12)
C101	0.0200 (14)	0.059 (2)	0.0217 (14)	0.0035 (14)	−0.0024 (11)	0.0050 (13)
C111	0.0303 (16)	0.0373 (17)	0.0292 (15)	0.0102 (13)	0.0008 (12)	0.0097 (13)
C121	0.0212 (14)	0.0262 (14)	0.0275 (14)	0.0044 (11)	0.0047 (11)	0.0049 (11)
O12	0.0228 (10)	0.0375 (11)	0.0218 (9)	0.0083 (9)	−0.0041 (8)	−0.0021 (8)
O22	0.0184 (9)	0.0241 (10)	0.0222 (9)	0.0039 (7)	0.0037 (7)	0.0034 (7)
O32	0.0262 (10)	0.0287 (10)	0.0206 (9)	−0.0021 (8)	−0.0042 (8)	0.0018 (8)
O42	0.0264 (11)	0.0305 (11)	0.0285 (10)	−0.0070 (8)	−0.0040 (8)	0.0061 (8)
C12	0.0225 (14)	0.0119 (11)	0.0221 (12)	−0.0005 (10)	0.0053 (10)	0.0012 (9)
C22	0.0181 (14)	0.0212 (13)	0.0349 (15)	0.0022 (11)	0.0030 (11)	−0.0015 (11)
C32	0.0196 (14)	0.0358 (16)	0.0387 (16)	0.0018 (12)	0.0111 (12)	0.0023 (13)
C42	0.0415 (18)	0.0273 (15)	0.0248 (14)	0.0027 (13)	0.0125 (13)	0.0034 (11)
C52	0.0355 (16)	0.0239 (14)	0.0217 (13)	0.0000 (12)	0.0001 (12)	0.0000 (11)
C62	0.0218 (13)	0.0124 (11)	0.0222 (12)	0.0032 (10)	0.0011 (10)	0.0001 (9)
C72	0.0206 (14)	0.0171 (12)	0.0228 (13)	−0.0013 (10)	0.0013 (10)	−0.0031 (10)
C82	0.0282 (15)	0.0204 (13)	0.0160 (12)	0.0015 (11)	0.0005 (10)	0.0008 (10)
C92	0.0155 (13)	0.0241 (13)	0.0134 (11)	0.0012 (10)	0.0013 (9)	−0.0029 (9)
O1W	0.0272 (11)	0.0256 (10)	0.0224 (10)	0.0036 (9)	0.0040 (8)	−0.0014 (8)

Geometric parameters (Å, º)

Ni1—N31	2.062 (2)	C91—C101	1.369 (4)
Ni1—O22	2.0674 (18)	C91—H91	0.9500
Ni1—O32^{i}	2.068 (2)	C101—C111	1.385 (5)
Ni1—N11	2.088 (2)	C101—H101	0.9500
Ni1—O1W	2.098 (2)	C111—C121	1.380 (4)
Ni1—N21	2.106 (2)	C111—H111	0.9500
N11—C11	1.334 (4)	C121—H121	0.9500
N11—C51	1.347 (4)	O12—C72	1.244 (3)
N21—C61	1.477 (3)	O22—C72	1.269 (3)
N21—C71	1.492 (4)	O32—C92	1.268 (3)
N21—H21N	0.841 (10)	O42—C92	1.238 (3)
N31—C121	1.335 (3)	C12—C22	1.395 (4)
N31—C81	1.351 (3)	C12—C62	1.401 (4)
C11—C21	1.374 (4)	C12—C72	1.521 (3)
C11—H1A	0.9500	C22—C32	1.391 (4)
C21—C31	1.388 (5)	C22—H22	0.9500
C21—H21	0.9500	C32—C42	1.380 (4)
C31—C41	1.379 (5)	C32—H32	0.9500
C31—H31	0.9500	C42—C52	1.386 (4)
C41—C51	1.383 (4)	C42—H42	0.9500
C41—H41	0.9500	C52—C62	1.397 (4)
C51—C61	1.510 (4)	C52—H52	0.9500
C61—H61A	0.9900	C62—C82	1.510 (4)
C61—H61B	0.9900	C82—C92	1.525 (4)
C71—C81	1.490 (4)	C82—H82A	0.9900
C71—H71A	0.9900	C82—H82B	0.9900
C71—H71B	0.9900	O1W—H1WA	0.849 (10)
C81—C91	1.403 (4)	O1W—H1WB	0.848 (10)
N31—Ni1—O22	86.06 (8)	N21—C71—H71B	109.0
N31—Ni1—O32^{i}	92.06 (8)	H71A—C71—H71B	107.8
O22—Ni1—O32^{i}	94.91 (7)	N31—C81—C91	121.1 (3)
N31—Ni1—N11	94.72 (9)	N31—C81—C71	117.0 (2)
O22—Ni1—N11	171.57 (8)	C91—C81—C71	121.8 (3)
O32^{i}—Ni1—N11	93.45 (8)	C101—C91—C81	119.1 (3)
N31—Ni1—O1W	172.97 (8)	C101—C91—H91	120.5
O22—Ni1—O1W	91.91 (7)	C81—C91—H91	120.5
O32^{i}—Ni1—O1W	94.83 (7)	C91—C101—C111	119.6 (3)
N11—Ni1—O1W	86.32 (8)	C91—C101—H101	120.2
N31—Ni1—N21	82.28 (9)	C111—C101—H101	120.2
O22—Ni1—N21	92.31 (8)	C121—C111—C101	118.4 (3)
O32^{i}—Ni1—N21	170.50 (8)	C121—C111—H111	120.8
N11—Ni1—N21	79.50 (9)	C101—C111—H111	120.8
O1W—Ni1—N21	91.09 (9)	N31—C121—C111	123.0 (3)
C11—N11—C51	118.4 (2)	N31—C121—H121	118.5
C11—N11—Ni1	128.51 (19)	C111—C121—H121	118.5
C51—N11—Ni1	113.01 (18)	C72—O22—Ni1	126.19 (17)
C61—N21—C71	112.8 (2)	C92—O32—Ni1^{ii}	125.65 (17)

C61—N21—Ni1	105.95 (16)	C22—C12—C62	119.8 (2)
C71—N21—Ni1	109.55 (16)	C22—C12—C72	116.5 (2)
C61—N21—H21N	110 (2)	C62—C12—C72	123.7 (2)
C71—N21—H21N	102 (2)	C32—C22—C12	121.3 (3)
Ni1—N21—H21N	116 (2)	C32—C22—H22	119.4
C121—N31—C81	118.7 (2)	C12—C22—H22	119.4
C121—N31—Ni1	126.13 (19)	C42—C32—C22	119.2 (3)
C81—N31—Ni1	114.23 (17)	C42—C32—H32	120.4
N11—C11—C21	123.4 (3)	C22—C32—H32	120.4
N11—C11—H1A	118.3	C32—C42—C52	119.8 (3)
C21—C11—H1A	118.3	C32—C42—H42	120.1
C11—C21—C31	118.0 (3)	C52—C42—H42	120.1
C11—C21—H21	121.0	C42—C52—C62	122.0 (3)
C31—C21—H21	121.0	C42—C52—H52	119.0
C41—C31—C21	119.3 (3)	C62—C52—H52	119.0
C41—C31—H31	120.3	C52—C62—C12	117.9 (2)
C21—C31—H31	120.3	C52—C62—C82	118.1 (2)
C31—C41—C51	119.1 (3)	C12—C62—C82	124.0 (2)
C31—C41—H41	120.5	O12—C72—O22	126.0 (2)
C51—C41—H41	120.5	O12—C72—C12	116.7 (2)
N11—C51—C41	121.8 (3)	O22—C72—C12	117.3 (2)
N11—C51—C61	116.1 (2)	C62—C82—C92	115.1 (2)
C41—C51—C61	122.0 (3)	C62—C82—H82A	108.5
N21—C61—C51	109.7 (2)	C92—C82—H82A	108.5
N21—C61—H61A	109.7	C62—C82—H82B	108.5
C51—C61—H61A	109.7	C92—C82—H82B	108.5
N21—C61—H61B	109.7	H82A—C82—H82B	107.5
C51—C61—H61B	109.7	O42—C92—O32	125.7 (2)
H61A—C61—H61B	108.2	O42—C92—C82	116.9 (2)
C81—C71—N21	113.1 (2)	O32—C92—C82	117.4 (2)
C81—C71—H71A	109.0	Ni1—O1W—H1WA	98 (2)
N21—C71—H71A	109.0	Ni1—O1W—H1WB	102 (2)
C81—C71—H71B	109.0	H1WA—O1W—H1WB	105.7 (15)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O12	0.84 (2)	1.79 (2)	2.603 (3)	162 (2)
C82—H82*A*···O22	0.99	2.24	2.916 (3)	124
C121—H121···O32^{i}	0.95	2.55	3.110 (4)	118
O1*W*—H1*WA*···O42^{i}	0.84 (2)	1.81 (2)	2.632 (3)	163 (3)
C71—H71*B*···O22^{ii}	0.99	2.54	3.205 (4)	125
C121—H121···O12^{i}	0.95	2.33	3.114 (4)	139
C22—H22···O12^{iii}	0.95	2.49	3.309 (3)	144
C52—H52···O42^{iv}	0.95	2.48	3.191 (3)	131

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

(II) (μ-Benzene-1,2,4,5-tetracarboxylato-κ⁴O¹,O²:O⁴,O⁵)bis(aqua{bis[(pyridin-2-yl)methyl]amine-κ³N,N′,N′′}nickel(II)) bis{triaquabis[(pyridin-2-yl)methyl]amine-κ³N,N′,N′′}nickel(II)} benzene-1,2,4,5-tetracarboxylate hexahydrate

Crystal data

$[Ni_2(C_{10}H_2O_8)(C_{12}H_{13}N_3)_2(H_2O)_2]\cdot[Ni(C_{12}H_{13}N_3)(H_2O)_3]_2(C_{10}H_2O_8)\cdot 6H_2O$
$M_r = 1784.32$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.8485$ (6) Å
$b = 10.7208$ (6) Å
$c = 19.4924$ (11) Å
$\alpha = 85.008$ (1)°
$\beta = 82.823$ (1)°
$\gamma = 65.2930$ (8)°
$V = 1853.65$ (19) Å^3
$Z = 1$
$F(000) = 928$
$D_x = 1.598$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6234 reflections
$\theta = 2.3$–$26.1°$
$\mu = 1.10$ mm^{-1}
$T = 298$ K
Plate, blue
0.41 × 0.37 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS* in *SAINT-NT*; Bruker, 2002)
$T_{min} = 0.65$, $T_{max} = 0.93$
15507 measured reflections
7916 independent reflections
6969 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.1°$
$h = -12\rightarrow12$
$k = -13\rightarrow13$
$l = -25\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.087$
$S = 1.09$
7916 reflections
562 parameters
24 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.4178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.86938 (2)	0.65317 (2)	0.387961 (11)	0.02431 (7)
Ni2	0.73509 (2)	0.30305 (2)	0.105617 (11)	0.02524 (7)
N11	0.89448 (17)	0.83613 (15)	0.39166 (8)	0.0292 (3)
N21	1.09898 (17)	0.57239 (17)	0.39571 (8)	0.0327 (3)

H21N	1.117 (2)	0.5022 (16)	0.4229 (10)	0.039*
N31	0.93585 (17)	0.65210 (15)	0.28392 (8)	0.0297 (3)
C11	0.7954 (2)	0.9644 (2)	0.37731 (11)	0.0382 (4)
H11	0.7041	0.9769	0.3626	0.046*
C21	0.8235 (3)	1.0778 (2)	0.38356 (12)	0.0518 (6)
H21	0.7519	1.1656	0.3741	0.062*
C31	0.9595 (4)	1.0592 (3)	0.40408 (14)	0.0619 (7)
H31	0.9812	1.1342	0.4091	0.074*
C41	1.0623 (3)	0.9284 (3)	0.41701 (13)	0.0551 (6)
H41	1.1555	0.9137	0.4303	0.066*
C51	1.0275 (2)	0.8184 (2)	0.41032 (10)	0.0365 (4)
C61	1.1339 (2)	0.6735 (2)	0.42659 (13)	0.0473 (5)
H61A	1.2354	0.6614	0.4097	0.057*
H61B	1.1301	0.6573	0.4764	0.057*
C71	1.1790 (2)	0.5274 (3)	0.32723 (12)	0.0512 (6)
H71A	1.2140	0.4284	0.3260	0.061*
H71B	1.2666	0.5483	0.3209	0.061*
C81	1.0852 (2)	0.5931 (2)	0.26809 (11)	0.0376 (4)
C91	1.1482 (3)	0.5879 (3)	0.20003 (12)	0.0556 (6)
H91	1.2518	0.5455	0.1897	0.067*
C101	1.0558 (3)	0.6462 (3)	0.14788 (12)	0.0571 (6)
H101	1.0963	0.6438	0.1020	0.068*
C111	0.9027 (3)	0.7082 (2)	0.16443 (11)	0.0483 (5)
H111	0.8381	0.7485	0.1301	0.058*
C121	0.8475 (2)	0.7092 (2)	0.23289 (10)	0.0368 (4)
H121	0.7442	0.7515	0.2442	0.044*
N12	0.92631 (17)	0.17616 (15)	0.15501 (8)	0.0292 (3)
N22	0.70576 (19)	0.42348 (16)	0.18884 (9)	0.0336 (4)
H22N	0.666 (2)	0.5074 (11)	0.1768 (11)	0.040*
N32	0.62005 (17)	0.21498 (16)	0.17317 (8)	0.0311 (3)
C12	1.0065 (2)	0.0405 (2)	0.15041 (10)	0.0349 (4)
H12	0.9860	−0.0049	0.1173	0.042*
C22	1.1178 (2)	−0.0348 (2)	0.19231 (11)	0.0407 (5)
H22	1.1706	−0.1291	0.1878	0.049*
C32	1.1496 (2)	0.0323 (2)	0.24112 (11)	0.0453 (5)
H32	1.2257	−0.0159	0.2696	0.054*
C42	1.0669 (2)	0.1719 (2)	0.24712 (12)	0.0451 (5)
H42	1.0861	0.2190	0.2798	0.054*
C52	0.9548 (2)	0.2409 (2)	0.20384 (10)	0.0344 (4)
C62	0.8572 (2)	0.3926 (2)	0.20759 (12)	0.0426 (5)
H62A	0.8501	0.4217	0.2541	0.051*
H62B	0.9024	0.4432	0.1763	0.051*
C72	0.6044 (3)	0.3974 (2)	0.24439 (12)	0.0466 (5)
H72A	0.5092	0.4775	0.2474	0.056*
H72B	0.6470	0.3859	0.2880	0.056*
C82	0.5750 (2)	0.2728 (2)	0.23503 (10)	0.0342 (4)
C92	0.5004 (2)	0.2221 (2)	0.28741 (11)	0.0448 (5)
H92	0.4718	0.2618	0.3304	0.054*
C102	0.4694 (3)	0.1133 (3)	0.27522 (13)	0.0523 (6)

H102	0.4171	0.0801	0.3095	0.063*
C112	0.5162 (3)	0.0529 (3)	0.21160 (13)	0.0490 (6)
H112	0.4975	−0.0218	0.2024	0.059*
C122	0.5915 (2)	0.1072 (2)	0.16249 (11)	0.0390 (5)
H122	0.6241	0.0669	0.1197	0.047*
O13	0.38940 (14)	0.72407 (13)	0.07835 (7)	0.0322 (3)
O23	0.56320 (17)	0.73292 (15)	0.13791 (7)	0.0423 (3)
O33	0.68113 (15)	0.62855 (13)	−0.02583 (8)	0.0366 (3)
O43	0.85608 (15)	0.70986 (15)	−0.03987 (10)	0.0522 (4)
C13	0.38596 (19)	1.02223 (17)	0.05253 (9)	0.0266 (4)
H13	0.3084	1.0380	0.0878	0.032*
C23	0.49120 (18)	0.88841 (17)	0.04133 (9)	0.0237 (3)
C33	0.60597 (18)	0.86647 (17)	−0.01208 (9)	0.0245 (3)
C43	0.48171 (19)	0.77153 (17)	0.08953 (9)	0.0265 (4)
C53	0.72385 (19)	0.72304 (17)	−0.02725 (10)	0.0280 (4)
O14	0.46858 (16)	0.69176 (14)	0.45087 (8)	0.0393 (3)
O24	0.64556 (13)	0.72099 (13)	0.37746 (6)	0.0294 (3)
O34	0.81310 (14)	0.65154 (12)	0.49520 (6)	0.0298 (3)
O44	0.82825 (16)	0.75958 (14)	0.58413 (7)	0.0406 (3)
C14	0.40566 (19)	0.99632 (18)	0.45498 (9)	0.0246 (3)
H14	0.3419	0.9937	0.4243	0.030*
C24	0.53018 (18)	0.87609 (17)	0.46901 (9)	0.0231 (3)
C34	0.62721 (18)	0.87996 (17)	0.51503 (9)	0.0229 (3)
C44	0.55201 (18)	0.75083 (17)	0.43124 (9)	0.0250 (3)
C54	0.76667 (19)	0.75516 (17)	0.53266 (9)	0.0255 (3)
O1W	0.89054 (16)	0.44803 (13)	0.38595 (7)	0.0355 (3)
H1WA	0.9691 (13)	0.3815 (17)	0.3980 (11)	0.043*
H1WB	0.8181 (14)	0.427 (2)	0.4012 (11)	0.043*
O2W	0.52974 (16)	0.43747 (14)	0.07384 (9)	0.0416 (4)
H2WA	0.471 (2)	0.410 (2)	0.0592 (12)	0.050*
H2WB	0.489 (2)	0.5225 (10)	0.0753 (13)	0.050*
O3W	0.77663 (15)	0.17377 (13)	0.02531 (7)	0.0309 (3)
H3WA	0.7214 (17)	0.206 (2)	−0.0069 (8)	0.037*
H3WB	0.8619 (11)	0.1694 (16)	0.0105 (10)	0.037*
O4W	0.84376 (16)	0.40591 (15)	0.04555 (9)	0.0445 (4)
H4WA	0.798 (2)	0.4736 (18)	0.0202 (11)	0.053*
H4WB	0.9340 (12)	0.392 (2)	0.0418 (12)	0.053*
O5W	0.62544 (19)	0.41145 (16)	0.42080 (8)	0.0485 (4)
H5WA	0.572 (2)	0.4963 (10)	0.4270 (12)	0.058*
H5WB	0.602 (3)	0.3695 (19)	0.4556 (9)	0.058*
O6W	0.4270 (2)	0.7324 (2)	0.27846 (10)	0.0684 (5)
H6WA	0.464 (3)	0.742 (3)	0.2376 (6)	0.082*
H6WB	0.499 (2)	0.708 (3)	0.3036 (11)	0.082*
O7W	0.92981 (16)	0.88579 (15)	0.02230 (8)	0.0403 (3)
H7WA	0.8597 (17)	0.9619 (10)	0.0243 (12)	0.048*
H7WB	0.907 (2)	0.8344 (17)	0.0004 (11)	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02213 (12)	0.02136 (12)	0.02693 (13)	−0.00661 (9)	−0.00231 (9)	−0.00046 (9)
Ni2	0.02518 (12)	0.02285 (12)	0.02769 (13)	−0.00958 (9)	−0.00516 (9)	0.00117 (9)
N11	0.0310 (8)	0.0272 (8)	0.0304 (8)	−0.0134 (7)	−0.0018 (6)	0.0002 (6)
N21	0.0271 (8)	0.0314 (9)	0.0338 (9)	−0.0072 (7)	−0.0058 (7)	0.0062 (7)
N31	0.0297 (8)	0.0268 (8)	0.0300 (8)	−0.0098 (6)	−0.0010 (6)	0.0003 (6)
C11	0.0406 (11)	0.0301 (10)	0.0405 (11)	−0.0124 (9)	−0.0022 (9)	0.0026 (8)
C21	0.0727 (17)	0.0290 (11)	0.0500 (13)	−0.0191 (11)	−0.0010 (12)	0.0005 (9)
C31	0.097 (2)	0.0496 (15)	0.0613 (16)	−0.0513 (16)	−0.0092 (15)	−0.0011 (12)
C41	0.0644 (16)	0.0653 (17)	0.0583 (15)	−0.0474 (14)	−0.0157 (12)	0.0029 (12)
C51	0.0399 (11)	0.0415 (11)	0.0344 (10)	−0.0231 (9)	−0.0053 (8)	0.0021 (8)
C61	0.0366 (11)	0.0508 (13)	0.0588 (14)	−0.0194 (10)	−0.0206 (10)	0.0066 (11)
C71	0.0279 (10)	0.0614 (15)	0.0439 (12)	0.0003 (10)	0.0001 (9)	0.0002 (11)
C81	0.0327 (10)	0.0352 (11)	0.0376 (11)	−0.0088 (9)	0.0024 (8)	−0.0005 (8)
C91	0.0420 (13)	0.0657 (16)	0.0452 (13)	−0.0129 (12)	0.0128 (10)	−0.0034 (12)
C101	0.0656 (17)	0.0691 (17)	0.0309 (12)	−0.0259 (14)	0.0069 (11)	0.0001 (11)
C111	0.0587 (14)	0.0528 (14)	0.0345 (11)	−0.0250 (12)	−0.0087 (10)	0.0093 (10)
C121	0.0385 (11)	0.0343 (10)	0.0359 (10)	−0.0136 (9)	−0.0071 (8)	0.0045 (8)
N12	0.0299 (8)	0.0273 (8)	0.0310 (8)	−0.0120 (6)	−0.0063 (6)	0.0016 (6)
N22	0.0387 (9)	0.0221 (8)	0.0371 (9)	−0.0083 (7)	−0.0084 (7)	−0.0021 (7)
N32	0.0295 (8)	0.0302 (8)	0.0315 (8)	−0.0106 (7)	−0.0045 (6)	0.0026 (6)
C12	0.0358 (10)	0.0310 (10)	0.0347 (10)	−0.0106 (8)	−0.0036 (8)	−0.0008 (8)
C22	0.0338 (10)	0.0341 (11)	0.0442 (12)	−0.0057 (9)	−0.0028 (9)	0.0047 (9)
C32	0.0334 (10)	0.0536 (14)	0.0415 (12)	−0.0093 (10)	−0.0143 (9)	0.0074 (10)
C42	0.0417 (12)	0.0544 (14)	0.0423 (12)	−0.0192 (11)	−0.0163 (10)	−0.0026 (10)
C52	0.0339 (10)	0.0344 (10)	0.0378 (10)	−0.0157 (8)	−0.0078 (8)	−0.0012 (8)
C62	0.0483 (12)	0.0344 (11)	0.0503 (13)	−0.0177 (10)	−0.0179 (10)	−0.0065 (9)
C72	0.0544 (14)	0.0396 (12)	0.0405 (12)	−0.0148 (10)	0.0049 (10)	−0.0124 (9)
C82	0.0288 (9)	0.0320 (10)	0.0328 (10)	−0.0046 (8)	−0.0017 (8)	0.0019 (8)
C92	0.0370 (11)	0.0519 (13)	0.0341 (11)	−0.0096 (10)	0.0012 (9)	0.0046 (9)
C102	0.0456 (13)	0.0656 (16)	0.0481 (14)	−0.0295 (12)	−0.0046 (10)	0.0206 (12)
C112	0.0519 (13)	0.0531 (14)	0.0531 (14)	−0.0337 (12)	−0.0106 (11)	0.0129 (11)
C122	0.0431 (11)	0.0409 (11)	0.0385 (11)	−0.0226 (10)	−0.0069 (9)	0.0031 (9)
O13	0.0347 (7)	0.0292 (7)	0.0381 (7)	−0.0186 (6)	−0.0081 (6)	0.0060 (5)
O23	0.0504 (9)	0.0413 (8)	0.0423 (8)	−0.0238 (7)	−0.0234 (7)	0.0157 (6)
O33	0.0299 (7)	0.0215 (6)	0.0593 (9)	−0.0103 (5)	−0.0099 (6)	0.0004 (6)
O43	0.0217 (7)	0.0294 (8)	0.1024 (14)	−0.0077 (6)	0.0034 (8)	−0.0149 (8)
C13	0.0226 (8)	0.0248 (9)	0.0318 (9)	−0.0097 (7)	−0.0009 (7)	−0.0011 (7)
C23	0.0226 (8)	0.0213 (8)	0.0288 (9)	−0.0104 (7)	−0.0058 (7)	0.0025 (7)
C33	0.0202 (8)	0.0203 (8)	0.0329 (9)	−0.0076 (7)	−0.0058 (7)	0.0003 (7)
C43	0.0274 (9)	0.0213 (8)	0.0283 (9)	−0.0081 (7)	−0.0018 (7)	0.0006 (7)
C53	0.0247 (9)	0.0216 (8)	0.0358 (10)	−0.0074 (7)	−0.0050 (7)	0.0002 (7)
O14	0.0398 (8)	0.0343 (7)	0.0493 (8)	−0.0221 (6)	0.0054 (6)	−0.0079 (6)
O24	0.0229 (6)	0.0324 (7)	0.0296 (7)	−0.0075 (5)	−0.0013 (5)	−0.0059 (5)
O34	0.0325 (7)	0.0212 (6)	0.0277 (6)	−0.0028 (5)	−0.0039 (5)	−0.0008 (5)
O44	0.0393 (8)	0.0311 (7)	0.0415 (8)	0.0002 (6)	−0.0194 (6)	−0.0053 (6)
C14	0.0232 (8)	0.0262 (9)	0.0246 (8)	−0.0101 (7)	−0.0048 (6)	0.0004 (7)
C24	0.0204 (8)	0.0206 (8)	0.0252 (8)	−0.0064 (6)	0.0010 (6)	−0.0009 (6)

C34	0.0201 (8)	0.0207 (8)	0.0247 (8)	−0.0056 (6)	−0.0018 (6)	0.0000 (6)
C44	0.0209 (8)	0.0212 (8)	0.0296 (9)	−0.0045 (7)	−0.0064 (7)	−0.0001 (7)
C54	0.0237 (8)	0.0223 (8)	0.0268 (9)	−0.0063 (7)	−0.0028 (7)	0.0017 (7)
O1W	0.0334 (7)	0.0256 (7)	0.0435 (8)	−0.0085 (6)	−0.0046 (6)	0.0010 (6)
O2W	0.0345 (8)	0.0258 (7)	0.0665 (10)	−0.0102 (6)	−0.0232 (7)	0.0028 (7)
O3W	0.0305 (7)	0.0307 (7)	0.0309 (7)	−0.0112 (6)	−0.0068 (5)	0.0000 (5)
O4W	0.0260 (7)	0.0390 (8)	0.0642 (10)	−0.0131 (6)	−0.0082 (7)	0.0239 (7)
O5W	0.0579 (10)	0.0347 (8)	0.0489 (9)	−0.0187 (8)	0.0117 (8)	−0.0067 (7)
O6W	0.0612 (12)	0.0782 (14)	0.0539 (11)	−0.0149 (11)	−0.0140 (9)	−0.0025 (10)
O7W	0.0400 (8)	0.0335 (8)	0.0486 (9)	−0.0154 (6)	−0.0061 (7)	−0.0041 (7)

Geometric parameters (Å, º)

Ni1—O24	2.0437 (12)	C32—H32	0.9300
Ni1—N31	2.0512 (15)	C42—C52	1.385 (3)
Ni1—N21	2.0764 (16)	C42—H42	0.9300
Ni1—N11	2.0852 (15)	C52—C62	1.507 (3)
Ni1—O34	2.0950 (13)	C62—H62A	0.9700
Ni1—O1W	2.1241 (13)	C62—H62B	0.9700
Ni2—O4W	2.0448 (13)	C72—C82	1.510 (3)
Ni2—N32	2.0528 (16)	C72—H72A	0.9700
Ni2—O2W	2.0668 (13)	C72—H72B	0.9700
Ni2—O3W	2.0695 (13)	C82—C92	1.388 (3)
Ni2—N22	2.0778 (16)	C92—C102	1.370 (3)
Ni2—N12	2.0995 (15)	C92—H92	0.9300
N11—C51	1.335 (2)	C102—C112	1.385 (4)
N11—C11	1.342 (2)	C102—H102	0.9300
N21—C61	1.460 (3)	C112—C122	1.375 (3)
N21—C71	1.472 (3)	C112—H112	0.9300
N21—H21N	0.850 (9)	C122—H122	0.9300
N31—C121	1.336 (2)	O13—C43	1.260 (2)
N31—C81	1.342 (2)	O23—C43	1.244 (2)
C11—C21	1.373 (3)	O33—C53	1.246 (2)
C11—H11	0.9300	O43—C53	1.244 (2)
C21—C31	1.375 (4)	C13—C23	1.391 (2)
C21—H21	0.9300	C13—C33^{i}	1.397 (2)
C31—C41	1.367 (4)	C13—H13	0.9300
C31—H31	0.9300	C23—C33	1.394 (2)
C41—C51	1.379 (3)	C23—C43	1.527 (2)
C41—H41	0.9300	C33—C13^{i}	1.397 (2)
C51—C61	1.500 (3)	C33—C53	1.517 (2)
C61—H61A	0.9700	O14—C44	1.240 (2)
C61—H61B	0.9700	O24—C44	1.273 (2)
C71—C81	1.503 (3)	O34—C54	1.269 (2)
C71—H71A	0.9700	O44—C54	1.249 (2)
C71—H71B	0.9700	C14—C34^{ii}	1.391 (2)
C81—C91	1.388 (3)	C14—C24	1.393 (2)
C91—C101	1.378 (4)	C14—H14	0.9300
C91—H91	0.9300	C24—C34	1.405 (2)
C101—C111	1.378 (3)	C24—C44	1.511 (2)

C101—H101	0.9300	C34—C14^{ii}	1.391 (2)
C111—C121	1.376 (3)	C34—C54	1.515 (2)
C111—H111	0.9300	O1W—H1WA	0.847 (9)
C121—H121	0.9300	O1W—H1WB	0.845 (9)
N12—C12	1.338 (2)	O2W—H2WA	0.835 (9)
N12—C52	1.346 (2)	O2W—H2WB	0.830 (9)
N22—C72	1.469 (3)	O3W—H3WA	0.836 (9)
N22—C62	1.473 (3)	O3W—H3WB	0.837 (9)
N22—H22N	0.842 (9)	O4W—H4WA	0.833 (9)
N32—C122	1.337 (3)	O4W—H4WB	0.833 (9)
N32—C82	1.342 (2)	O5W—H5WA	0.850 (9)
C12—C22	1.375 (3)	O5W—H5WB	0.843 (9)
C12—H12	0.9300	O6W—H6WA	0.849 (10)
C22—C32	1.379 (3)	O6W—H6WB	0.846 (10)
C22—H22	0.9300	O7W—H7WA	0.820 (9)
C32—C42	1.380 (3)	O7W—H7WB	0.837 (9)

O24—Ni1—N31	95.65 (6)	C122—N32—C82	118.79 (17)
O24—Ni1—N21	176.24 (6)	C122—N32—Ni2	127.29 (14)
N31—Ni1—N21	82.88 (6)	C82—N32—Ni2	113.88 (13)
O24—Ni1—N11	102.50 (6)	N12—C12—C22	123.12 (19)
N31—Ni1—N11	88.32 (6)	N12—C12—H12	118.4
N21—Ni1—N11	80.95 (6)	C22—C12—H12	118.4
O24—Ni1—O34	87.32 (5)	C12—C22—C32	118.7 (2)
N31—Ni1—O34	177.01 (5)	C12—C22—H22	120.6
N21—Ni1—O34	94.17 (6)	C32—C22—H22	120.6
N11—Ni1—O34	90.80 (5)	C22—C32—C42	118.95 (19)
O24—Ni1—O1W	88.92 (5)	C22—C32—H32	120.5
N31—Ni1—O1W	90.39 (6)	C42—C32—H32	120.5
N21—Ni1—O1W	87.62 (6)	C32—C42—C52	119.2 (2)
N11—Ni1—O1W	168.58 (6)	C32—C42—H42	120.4
O34—Ni1—O1W	89.92 (5)	C52—C42—H42	120.4
O4W—Ni2—N32	174.15 (7)	N12—C52—C42	121.82 (19)
O4W—Ni2—O2W	91.77 (6)	N12—C52—C62	115.34 (17)
N32—Ni2—O2W	87.28 (6)	C42—C52—C62	122.84 (18)
O4W—Ni2—O3W	89.55 (6)	N22—C62—C52	110.56 (15)
N32—Ni2—O3W	96.25 (6)	N22—C62—H62A	109.5
O2W—Ni2—O3W	92.61 (6)	C52—C62—H62A	109.5
O4W—Ni2—N22	90.77 (7)	N22—C62—H62B	109.5
N32—Ni2—N22	83.48 (7)	C52—C62—H62B	109.5
O2W—Ni2—N22	91.06 (6)	H62A—C62—H62B	108.1
O3W—Ni2—N22	176.31 (6)	N22—C72—C82	114.44 (16)
O4W—Ni2—N12	93.58 (6)	N22—C72—H72A	108.6
N32—Ni2—N12	86.57 (6)	C82—C72—H72A	108.6
O2W—Ni2—N12	170.22 (6)	N22—C72—H72B	108.6
O3W—Ni2—N12	95.61 (6)	C82—C72—H72B	108.6
N22—Ni2—N12	80.70 (6)	H72A—C72—H72B	107.6
C51—N11—C11	118.44 (17)	N32—C82—C92	121.1 (2)
C51—N11—Ni1	113.61 (13)	N32—C82—C72	117.32 (17)

C11—N11—Ni1	127.95 (14)	C92—C82—C72	121.54 (19)
C61—N21—C71	115.25 (19)	C102—C92—C82	119.4 (2)
C61—N21—Ni1	108.77 (12)	C102—C92—H92	120.3
C71—N21—Ni1	108.88 (12)	C82—C92—H92	120.3
C61—N21—H21N	109.8 (15)	C92—C102—C112	119.7 (2)
C71—N21—H21N	108.7 (15)	C92—C102—H102	120.1
Ni1—N21—H21N	104.9 (15)	C112—C102—H102	120.1
C121—N31—C81	118.80 (17)	C122—C112—C102	117.7 (2)
C121—N31—Ni1	126.87 (13)	C122—C112—H112	121.1
C81—N31—Ni1	114.23 (13)	C102—C112—H112	121.1
N11—C11—C21	122.5 (2)	N32—C122—C112	123.3 (2)
N11—C11—H11	118.7	N32—C122—H122	118.4
C21—C11—H11	118.7	C112—C122—H122	118.4
C11—C21—C31	118.8 (2)	C23—C13—C33^{i}	121.54 (16)
C11—C21—H21	120.6	C23—C13—H13	119.2
C31—C21—H21	120.6	C33^{i}—C13—H13	119.2
C41—C31—C21	118.9 (2)	C13—C23—C33	118.50 (15)
C41—C31—H31	120.6	C13—C23—C43	119.30 (15)
C21—C31—H31	120.6	C33—C23—C43	122.14 (15)
C31—C41—C51	119.8 (2)	C23—C33—C13^{i}	119.95 (15)
C31—C41—H41	120.1	C23—C33—C53	121.16 (15)
C51—C41—H41	120.1	C13^{i}—C33—C53	118.89 (15)
N11—C51—C41	121.5 (2)	O23—C43—O13	125.05 (17)
N11—C51—C61	116.53 (17)	O23—C43—C23	117.32 (15)
C41—C51—C61	121.9 (2)	O13—C43—C23	117.60 (15)
N21—C61—C51	112.56 (16)	O43—C53—O33	125.45 (17)
N21—C61—H61A	109.1	O43—C53—C33	116.74 (15)
C51—C61—H61A	109.1	O33—C53—C33	117.81 (15)
N21—C61—H61B	109.1	C44—O24—Ni1	119.20 (11)
C51—C61—H61B	109.1	C54—O34—Ni1	125.34 (11)
H61A—C61—H61B	107.8	C34^{ii}—C14—C24	122.74 (16)
N21—C71—C81	113.87 (16)	C34^{ii}—C14—H14	118.6
N21—C71—H71A	108.8	C24—C14—H14	118.6
C81—C71—H71A	108.8	C14—C24—C34	118.73 (15)
N21—C71—H71B	108.8	C14—C24—C44	116.35 (15)
C81—C71—H71B	108.8	C34—C24—C44	124.91 (15)
H71A—C71—H71B	107.7	C14^{ii}—C34—C24	118.54 (15)
N31—C81—C91	121.2 (2)	C14^{ii}—C34—C54	118.87 (15)
N31—C81—C71	116.60 (18)	C24—C34—C54	122.59 (15)
C91—C81—C71	122.08 (19)	O14—C44—O24	124.36 (16)
C101—C91—C81	119.3 (2)	O14—C44—C24	117.95 (15)
C101—C91—H91	120.3	O24—C44—C24	117.36 (15)
C81—C91—H91	120.3	O44—C54—O34	123.66 (16)
C91—C101—C111	119.2 (2)	O44—C54—C34	117.88 (15)
C91—C101—H101	120.4	O34—C54—C34	118.45 (15)
C111—C101—H101	120.4	Ni1—O1W—H1WA	120.3 (14)
C121—C111—C101	118.5 (2)	Ni1—O1W—H1WB	120.8 (14)
C121—C111—H111	120.8	H1WA—O1W—H1WB	106.1 (13)
C101—C111—H111	120.8	Ni2—O2W—H2WA	122.1 (14)

N31—C121—C111	122.9 (2)	Ni2—O2W—H2WB	128.8 (15)
N31—C121—H121	118.5	H2WA—O2W—H2WB	109.0 (14)
C111—C121—H121	118.5	Ni2—O3W—H3WA	116.2 (15)
C12—N12—C52	118.14 (16)	Ni2—O3W—H3WB	97.6 (13)
C12—N12—Ni2	128.48 (13)	H3WA—O3W—H3WB	107.7 (13)
C52—N12—Ni2	112.71 (12)	Ni2—O4W—H4WA	121.5 (15)
C72—N22—C62	115.19 (17)	Ni2—O4W—H4WB	129.7 (15)
C72—N22—Ni2	109.51 (12)	H4WA—O4W—H4WB	108.7 (15)
C62—N22—Ni2	106.37 (12)	H5WA—O5W—H5WB	105.7 (14)
C72—N22—H22N	107.9 (16)	H6WA—O6W—H6WB	105.7 (15)
C62—N22—H22N	107.6 (16)	H7WA—O7W—H7WB	109.3 (14)
Ni2—N22—H22N	110.3 (16)		

O24—Ni1—N11—C51	169.15 (13)	N12—Ni2—N32—C122	−94.24 (17)
N31—Ni1—N11—C51	−95.43 (13)	O2W—Ni2—N32—C82	−88.96 (13)
N21—Ni1—N11—C51	−12.38 (13)	O3W—Ni2—N32—C82	178.71 (13)
O34—Ni1—N11—C51	81.72 (13)	N22—Ni2—N32—C82	2.42 (13)
O1W—Ni1—N11—C51	−11.8 (4)	N12—Ni2—N32—C82	83.44 (13)
O24—Ni1—N11—C11	−11.38 (17)	C52—N12—C12—C22	−1.1 (3)
N31—Ni1—N11—C11	84.03 (17)	Ni2—N12—C12—C22	−170.96 (15)
N21—Ni1—N11—C11	167.09 (17)	N12—C12—C22—C32	−0.6 (3)
O34—Ni1—N11—C11	−98.82 (16)	C12—C22—C32—C42	1.3 (3)
O1W—Ni1—N11—C11	167.6 (3)	C22—C32—C42—C52	−0.3 (3)
N31—Ni1—N21—C61	112.18 (14)	C12—N12—C52—C42	2.1 (3)
N11—Ni1—N21—C61	22.76 (13)	Ni2—N12—C52—C42	173.49 (16)
O34—Ni1—N21—C61	−67.39 (14)	C12—N12—C52—C62	−178.17 (18)
O1W—Ni1—N21—C61	−157.13 (14)	Ni2—N12—C52—C62	−6.8 (2)
N31—Ni1—N21—C71	−14.15 (15)	C32—C42—C52—N12	−1.4 (3)
N11—Ni1—N21—C71	−103.57 (15)	C32—C42—C52—C62	178.9 (2)
O34—Ni1—N21—C71	166.28 (15)	C72—N22—C62—C52	80.8 (2)
O1W—Ni1—N21—C71	76.54 (15)	Ni2—N22—C62—C52	−40.7 (2)
O24—Ni1—N31—C121	12.69 (16)	N12—C52—C62—N22	32.6 (3)
N21—Ni1—N31—C121	−170.79 (17)	C42—C52—C62—N22	−147.7 (2)
N11—Ni1—N31—C121	−89.70 (16)	C62—N22—C72—C82	−107.2 (2)
O1W—Ni1—N31—C121	101.65 (16)	Ni2—N22—C72—C82	12.6 (2)
O24—Ni1—N31—C81	−171.06 (14)	C122—N32—C82—C92	0.2 (3)
N21—Ni1—N31—C81	5.45 (14)	Ni2—N32—C82—C92	−177.70 (15)
N11—Ni1—N31—C81	86.54 (14)	C122—N32—C82—C72	−177.90 (18)
O1W—Ni1—N31—C81	−82.11 (14)	Ni2—N32—C82—C72	4.2 (2)
C51—N11—C11—C21	−2.4 (3)	N22—C72—C82—N32	−11.7 (3)
Ni1—N11—C11—C21	178.11 (16)	N22—C72—C82—C92	170.26 (19)
N11—C11—C21—C31	1.2 (3)	N32—C82—C92—C102	−1.4 (3)
C11—C21—C31—C41	0.5 (4)	C72—C82—C92—C102	176.6 (2)
C21—C31—C41—C51	−0.9 (4)	C82—C92—C102—C112	1.7 (3)
C11—N11—C51—C41	2.0 (3)	C92—C102—C112—C122	−0.8 (3)
Ni1—N11—C51—C41	−178.51 (18)	C82—N32—C122—C112	0.8 (3)
C11—N11—C51—C61	179.36 (18)	Ni2—N32—C122—C112	178.38 (16)
Ni1—N11—C51—C61	−1.1 (2)	C102—C112—C122—N32	−0.5 (3)
C31—C41—C51—N11	−0.3 (4)	C33^{i}—C13—C23—C33	−0.6 (3)

C31—C41—C51—C61	−177.6 (2)	C33^{i}—C13—C23—C43	176.80 (15)
C71—N21—C61—C51	92.9 (2)	C13—C23—C33—C13^{i}	0.5 (3)
Ni1—N21—C61—C51	−29.7 (2)	C43—C23—C33—C13^{i}	−176.73 (15)
N11—C51—C61—N21	21.2 (3)	C13—C23—C33—C53	−179.70 (16)
C41—C51—C61—N21	−161.5 (2)	C43—C23—C33—C53	3.0 (2)
C61—N21—C71—C81	−102.0 (2)	C13—C23—C43—O23	−96.9 (2)
Ni1—N21—C71—C81	20.5 (2)	C33—C23—C43—O23	80.4 (2)
C121—N31—C81—C91	−1.5 (3)	C13—C23—C43—O13	81.3 (2)
Ni1—N31—C81—C91	−178.03 (18)	C33—C23—C43—O13	−101.4 (2)
C121—N31—C81—C71	−178.55 (19)	C23—C33—C53—O43	−137.43 (19)
Ni1—N31—C81—C71	4.9 (2)	C13^{i}—C33—C53—O43	42.3 (2)
N21—C71—C81—N31	−17.5 (3)	C23—C33—C53—O33	42.3 (2)
N21—C71—C81—C91	165.4 (2)	C13^{i}—C33—C53—O33	−137.96 (18)
N31—C81—C91—C101	0.9 (4)	N31—Ni1—O24—C44	−173.47 (13)
C71—C81—C91—C101	177.9 (2)	N11—Ni1—O24—C44	−83.95 (13)
C81—C91—C101—C111	−0.1 (4)	O34—Ni1—O24—C44	6.28 (13)
C91—C101—C111—C121	−0.2 (4)	O1W—Ni1—O24—C44	96.25 (13)
C81—N31—C121—C111	1.2 (3)	O24—Ni1—O34—C54	−82.65 (14)
Ni1—N31—C121—C111	177.28 (16)	N21—Ni1—O34—C54	100.82 (14)
C101—C111—C121—N31	−0.4 (3)	N11—Ni1—O34—C54	19.83 (14)
O4W—Ni2—N12—C12	−112.57 (17)	O1W—Ni1—O34—C54	−171.57 (14)
N32—Ni2—N12—C12	73.28 (16)	C34^{ii}—C14—C24—C34	−0.4 (3)
O3W—Ni2—N12—C12	−22.67 (17)	C34^{ii}—C14—C24—C44	−178.86 (16)
N22—Ni2—N12—C12	157.23 (17)	C14—C24—C34—C14^{ii}	0.4 (3)
O4W—Ni2—N12—C52	77.12 (14)	C44—C24—C34—C14^{ii}	178.70 (16)
N32—Ni2—N12—C52	−97.02 (14)	C14—C24—C34—C54	−179.04 (15)
O3W—Ni2—N12—C52	167.02 (13)	C44—C24—C34—C54	−0.7 (3)
N22—Ni2—N12—C52	−13.07 (13)	Ni1—O24—C44—O14	−118.09 (17)
O4W—Ni2—N22—C72	170.62 (14)	Ni1—O24—C44—C24	68.69 (17)
N32—Ni2—N22—C72	−8.30 (14)	C14—C24—C44—O14	−74.9 (2)
O2W—Ni2—N22—C72	78.84 (14)	C34—C24—C44—O14	106.7 (2)
N12—Ni2—N22—C72	−95.87 (14)	C14—C24—C44—O24	98.72 (18)
O4W—Ni2—N22—C62	−64.30 (13)	C34—C24—C44—O24	−79.6 (2)
N32—Ni2—N22—C62	116.78 (13)	Ni1—O34—C54—O44	−124.30 (17)
O2W—Ni2—N22—C62	−156.09 (13)	Ni1—O34—C54—C34	56.8 (2)
N12—Ni2—N22—C62	29.21 (13)	C14^{ii}—C34—C54—O44	15.3 (2)
O2W—Ni2—N32—C122	93.37 (17)	C24—C34—C54—O44	−165.23 (17)
O3W—Ni2—N32—C122	1.04 (17)	C14^{ii}—C34—C54—O34	−165.70 (16)
N22—Ni2—N32—C122	−175.25 (17)	C24—C34—C54—O34	13.7 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*N*···O34^{iii}	0.85 (2)	2.15 (2)	2.978 (2)	166 (2)
O1*W*—H1*WA*···O44^{iii}	0.85 (2)	1.99 (2)	2.827 (2)	170 (2)
O1*W*—H1*WB*···O5*W*	0.85 (2)	1.96 (2)	2.793 (3)	167 (2)
O5*W*—H5*WA*···O14	0.85 (1)	1.98 (1)	2.821 (2)	172 (2)
O5*W*—H5*WB*···O14^{iv}	0.84 (2)	2.02 (2)	2.852 (2)	167 (2)

C21—H21···O5*W*^{v}	0.93	2.59	3.375 (3)	142
C31—H31···O44^{vi}	0.93	2.59	3.435 (4)	151
N22—H22*N*···O23	0.84 (2)	2.30 (1)	3.139 (2)	177 (3)
O2*W*—H2*WA*···O33^{vii}	0.84 (2)	1.91 (2)	2.742 (2)	173 (2)
O2*W*—H2*WB*···O13	0.83 (1)	1.97 (1)	2.797 (2)	179 (3)
O3*W*—H3*WA*···O13^{vii}	0.84 (2)	1.79 (2)	2.627 (2)	177 (2)
O3*W*—H3*WB*···O7*W*^{viii}	0.84 (1)	1.92 (1)	2.742 (2)	167 (2)
O4*W*—H4*WA*···O33	0.83 (2)	1.81 (2)	2.642 (2)	173 (2)
O4*W*—H4*WB*···O43^{viii}	0.83 (2)	1.89 (2)	2.677 (2)	158 (2)
O6*W*—H6*WA*···O23	0.85 (2)	2.05 (2)	2.896 (2)	171 (3)
O7*W*—H7*WA*···O3*W*^{v}	0.82 (1)	2.07 (1)	2.816 (2)	151 (2)
O7*W*—H7*WB*···O43	0.84 (2)	1.87 (2)	2.705 (2)	174 (2)
C12—H12···O7*W*^{ix}	0.93	2.51	3.435 (3)	176
O6*W*—H6*WB*···O24	0.85 (2)	2.21 (2)	3.025 (2)	160 (3)
C72—H72*B*···O5*W*	0.97	2.60	3.490 (3)	153

Symmetry codes: (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, *y*+1, *z*; (vi) −*x*+2, −*y*+2, −*z*+1; (vii) −*x*+1, −*y*+1, −*z*; (viii) −*x*+2, −*y*+1, −*z*; (ix) *x*, *y*−1, *z*.