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catena-Poly[[aqua(iminodiacetato-κ^3O,N,O')-nickel(II)]-μ-4,4′-dipyridylamine-$\kappa^2N:N'$]

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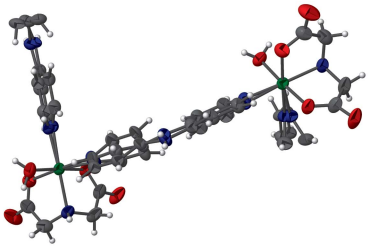
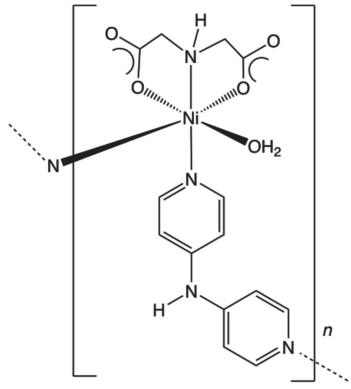
In the title compound, $[Ni(C_4H_5NO_4)(C_{10}H_9N_3)(H_2O)]_n$, the Ni^{II} cations are octahedrally coordinated by an N atom donor and two O donor atoms belonging to a tridentate iminodiacetate (ida) ligand in a *fac* fashion, an O atom donor from an aqua ligand, and *cis*-disposed pyridyl N donor atoms from two 4,4′-dipyridylamine (dpa) ligands. The dpa ligands are disordered over two sets of sites in a 0.594 (7):0.406 (7) ratio. Through the bridging dpa ligands, $[Ni(ida)(dpa)(H_2O)]_n$ zigzag coordination polymer chains are formed that are oriented along the *b*-axis direction. These chain motifs are anchored into the three-dimensional supramolecular crystal structure of the title compound by means of O—H···O and N—H···O hydrogen-bonding pathways.

3D view



Chemical scheme



Structure description

The dipodal tethering ligand 4,4′-dipyridylamine (dpa) has proven exceptionally useful in preparing coordination polymers with intriguing and diverse topologies (LaDuca, 2009). The title compound was prepared during synthetic attempts to prepare divalent metal coordination polymers containing both iminodiacetate (ida) and dpa ligands.

The asymmetric unit of the title compound contains an Ni^{II} atom, an aqua ligand, a doubly deprotonated ida ligand and a dpa ligand. The atoms in the dpa ligand show positional disorder and were modeled successfully using refined partial occupancies [0.594 (7):0.406 (7) ratio] and two parts. The Ni^{II} atom is octahedrally coordinated (Fig. 1) with the ida ligand binding in a tridentate manner, in which O atoms of the carboxylate termini and the central imine N atom occupy three coordination sites in a *fac* arrangement. The bound water molecule occupies a fourth site. The remaining *cis* coordination sites are taken up by pyridyl N atom donors from two dpa ligands. One carboxylate O atom at each ida carboxylate terminus remains unligated.

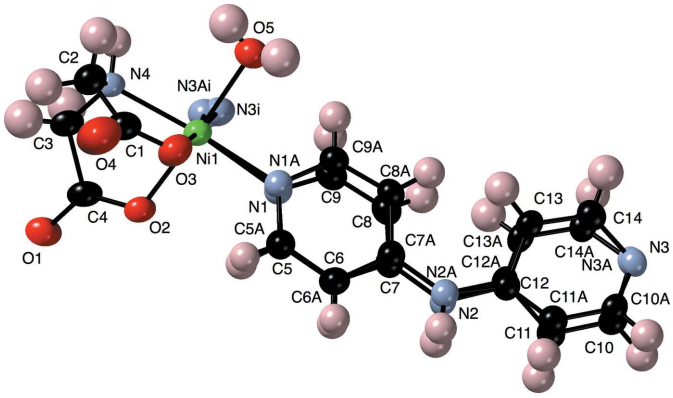


Figure 1
The coordination environment within the title compound, showing octahedral coordination at the Ni^{II} cation. Displacement ellipsoids are drawn at the 50% probability level. All non-H atoms are labeled. Both disorder components of the dpa ligands are shown. Color code: Ni, green; N, blue; O, red; C, black; H, pink. Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

The dpa ligands act as exobidentate tethering ligands, conjoining neighboring $[Ni(ida)(H_2O)]$ coordination fragments into a one-dimensional $[Ni(ida)(dpa)(H_2O)]_n$ coordination polymer ribbon (Fig. 2). These have a zigzag topology and are arranged parallel to [010], with an Ni···Ni through-ligand distance of 11.450 (4) Å.

The central amine moieties of the dpa ligands in one $[Ni(ida)(dpa)(H_2O)]_n$ coordination polymer ribbon provide N—H···O hydrogen-bonding points of contact (Table 1) to unligated ida carboxylate groups in adjacent ribbon motifs. Additionally, the aqua ligands provide inter-ribbon connections (Table 1) *via* O—H···O hydrogen bonding to other unligated ida carboxylate groups. By these two distinct hydrogen-bonding pathways, the individual $[Ni(ida)(dpa)(H_2O)]_n$ coordination polymer ribbons aggregate into the supramolecular three-dimensional crystal of the title compound (Fig. 3)

Synthesis and crystallization

$Ni(NO_3)_2{\cdot}6H_2O$ (108 mg, 0.37 mmol), iminodiacetic acid (49 mg, 0.37 mol), 4,4-dipyridylamine (73 mg, 0.37 mol) and 0.75 ml of a 1.0 *M* NaOH solution were placed into 10 ml distilled H_2O in a Teflon-lined acid digestion bomb. The bomb

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O1^{ii}	0.81 (2)	1.99 (2)	2.774 (4)	161 (5)
O5—H5*B*···O1^{iii}	0.86 (2)	1.86 (2)	2.706 (5)	172 (5)
N4—H4···O2^{iii}	1.00	2.28	3.233 (4)	158
C2—H2*A*···O1^{iii}	0.99	2.58	3.374 (6)	137
C6—H6···O3^{iv}	0.95	2.32	3.259 (14)	170
C9—H9···N3^{i}	0.95	2.56	3.037 (16)	112
N2—H2···O4^{iv}	0.88	1.86	2.718 (16)	166
C6*A*—H6*A*···O3^{iv}	0.95	2.50	3.37 (2)	152
C9*A*—H9*A*···O5	0.95	2.47	2.952 (13)	111
N2*A*—H2*AA*···O4^{iv}	0.88	1.99	2.82 (2)	156
C10*A*—H10*A*···O5^{v}	0.95	2.52	3.131 (15)	123
C14*A*—H14*A*···O2^{v}	0.95	2.28	2.904 (9)	122

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_4H_5NO_4)(C_{10}H_9N_3)(H_2O)]$
M_r	379.02
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, *b*, *c* (Å)	10.186 (2), 14.541 (3), 11.489 (2)
β (°)	100.531 (2)
V (Å^3)	1673.0 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.19
Crystal size (mm)	0.31 × 0.14 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.633, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15567, 3823, 2480
R_{int}	0.073
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.054, 0.158, 0.99
No. of reflections	3823
No. of parameters	341
No. of restraints	526
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.98, −0.34

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *OLEX2* (Dolomanov *et al.*, 2009), *SHELXL2018* (Sheldrick, 2015) and *CrystalMaker* (Palmer, 2018).

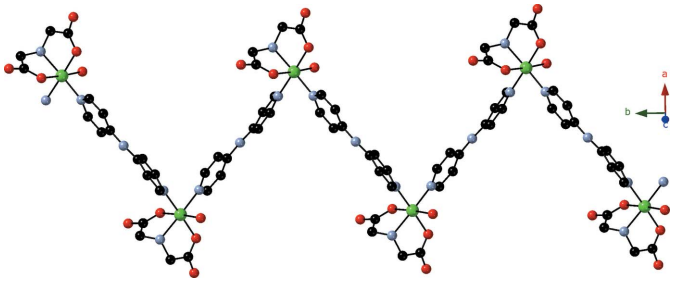


Figure 2
$[Ni(ima)(dpa)(H_2O)]_n$ coordination polymer zigzag chains parallel to [010] in the title compound. Only one dpa disorder component is shown.

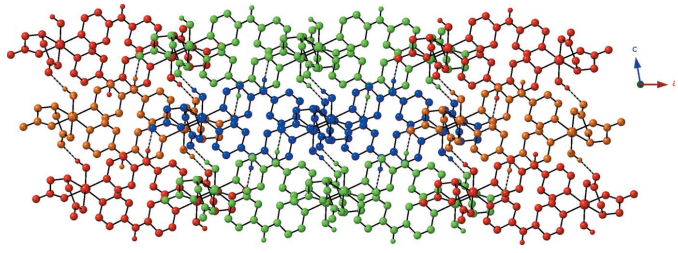


Figure 3
Interaction of the $[Ni(ima)(dpa)(H_2O)]_n$ coordination polymer chain motifs by O—H···O and N—H···O hydrogen bonds, which are shown as dashed lines. Individual ribbon motifs are colored for clarity.

was sealed and heated in an oven at 393 K for 48 h, and then cooled slowly to 278 K. Blue block-shaped crystals of the title compound (57 mg, 41% yield based on nickel) were isolated after washing with distilled water and acetone, and drying in air.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The atoms of the dpa ligand are disordered in place over two sets of positions with refined occupancies in a 0.594 (7):0.406 (7) ratio. Chemically equivalent bond distances and angles were restrained to be similar using SADI and SAME commands, with an e.s.d. of 0.02 Å. SIMU commands were used to treat the thermal ellipsoids within the dpa disorder components within 2 Å with 1σ of 0.01 and σ for terminal atoms of 0.02 Å^2.

Acknowledgements

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Funding information

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full crystallographic data

IUCrData (2018). **3**, x180865 [https://doi.org/10.1107/S2414314618008659]

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Crystal data

$[Ni(C_4H_5NO_4)(C_{10}H_9N_3)(H_2O)]$
M_r = 379.02
Monoclinic, $P2_1/c$
a = 10.186 (2) Å
b = 14.541 (3) Å
c = 11.489 (2) Å
β = 100.531 (2)°
V = 1673.0 (6) Å^3
Z = 4
$F(000)$ = 784
D_x = 1.505 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3842 reflections
θ = 2.3–25.3°
μ = 1.19 mm^{-1}
T = 173 K
Block, blue
0.31 × 0.14 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.633, T_{max} = 0.745
15567 measured reflections
3823 independent reflections
2480 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −13→13
k = −18→18
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.158
S = 0.99
3823 reflections
341 parameters
526 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.98 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Hydrogen atoms bound to C and N were placed in calculated positions with a riding model with U_{iso} = 1.2U_{eq}. Hydrogen atoms bound to O were found by Fourier difference map and refined with with U_{iso} = 1.5U_{eq}. The O–H bonds were restrained to be 0.84 (2) Å using *DFIX* commands.
1. Fixed Uiso At 1.2 times of: All C(H) groups, All C(H,H) groups, All N(H) groups At 1.5 times of: All O(H,H) groups
2. Restrained distances O5-H5A = O5-H5B 0.84 with sigma of 0.02 N2-C12 ~ N2A-C12A ~ N2-C7 ~ N2A-C7A with sigma of 0.02 N3-C10 ~ N3A-C10A ~ N3-C14 ~ N3A-C14A ~ N1-C5 ~ N1-C9 ~ N1A-C5A ~ N1A-C9A with sigma of 0.02 C12-C11 ~ C12-C13 ~ C12A-C11A ~ C12A-C13A ~ C7-C6 ~ C7-C8 ~ C7A-C6A ~ C7A-C8A with sigma of 0.02
3. Uiso/Uaniso restraints and constraints N1 ~ C5 ~ C6 ~ C7 ~ C8 ~ C9 ~ N2 ~ N1A ~ C5A ~ C6A ~ C7A ~ C8A ~ C9A ~ N2A ~ N3 ~ C10 ~ C11 ~ C12 ~ C13 ~ C14 ~ N3A ~ C10A ~ C11A ~ C12A ~ C13A ~ C14A: within 2A with sigma of 0.01 and sigma for terminal atoms of 0.02 4. Same fragment restrains {N1, C5, C6, C7, C8, C9} sigma for 1-2: 0.02, 1-3: 0.04 as {N1A, C5A, C6A, C7A, C8A, C9A} {N1, C5, C6, C7, C8, C9} sigma for 1-2: 0.02, 1-3: 0.04 as {N3, C10, C11, C12, C13, C14} {N1, C5, C6, C7, C8, C9} sigma for 1-2: 0.02, 1-3: 0.04 as {N3A, C10A, C11A, C12A, C13A, C14A}
5. Others Sof(N1A)=Sof(C5A)=Sof(H5AA)=Sof(C6A)=Sof(H6A)=Sof(C7A)=Sof(C8A)=Sof(H8A)=Sof(C9A)=Sof(H9A)=Sof(N2A)=Sof(H2AA)=Sof(N3A)=Sof(C10A)=Sof(H10A)=Sof(C11A)=Sof(H11A)=Sof(C12A)=Sof(C13A)=Sof(H13A)=Sof(C14A)=Sof(H14A)=1-FVAR(1) Sof(N1)=Sof(C5)=Sof(H5)=Sof(C6)=Sof(H6)=Sof(C7)=Sof(C8)=Sof(H8)=Sof(C9)=Sof(H9)=Sof(N2)=Sof(H2)=Sof(N3)=Sof(C10)=Sof(H10)=Sof(C11)=Sof(H11)=Sof(C12)=Sof(C13)=Sof(H13)=Sof(C14)=Sof(H14)=FVAR(1) 6.a Ternary CH refined with riding coordinates: N4(H4) 6.b Secondary CH2 refined with riding coordinates: C2(H2A,H2B), C3(H3A,H3B) 6.c Aromatic/amide H refined with riding coordinates: C5(H5), C6(H6), C8(H8), C9(H9), N2(H2), C5A(H5AA), C6A(H6A), C8A(H8A), C9A(H9A), N2A(H2AA), C10(H10), C11(H11), C13(H13), C14(H14), C10A(H10A), C11A(H11A), C13A(H13A), C14A(H14A)

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni1	0.42446 (5)	0.33031 (3)	0.21251 (4)	0.03457 (19)	
O1	0.5512 (4)	0.10358 (18)	0.4044 (3)	0.0701 (10)	
O2	0.4700 (3)	0.24245 (17)	0.3568 (2)	0.0461 (7)	
O3	0.6004 (3)	0.39538 (19)	0.2771 (2)	0.0483 (7)	
O4	0.8202 (3)	0.3844 (3)	0.2862 (3)	0.0738 (10)	
O5	0.3899 (3)	0.41790 (19)	0.0653 (3)	0.0480 (7)	
H5A	0.389 (5)	0.4731 (14)	0.076 (4)	0.072*	
H5B	0.447 (4)	0.410 (3)	0.020 (4)	0.072*	
N4	0.5502 (3)	0.24046 (19)	0.1415 (3)	0.0403 (8)	
H4	0.514058	0.229381	0.055764	0.048*	
C1	0.7034 (5)	0.3602 (3)	0.2478 (4)	0.0497 (10)	
C2	0.6830 (4)	0.2840 (3)	0.1556 (4)	0.0540 (11)	
H2A	0.694940	0.309763	0.078467	0.065*	
H2B	0.752364	0.236389	0.178576	0.065*	
C3	0.5507 (6)	0.1544 (3)	0.2082 (4)	0.0589 (13)	
H3A	0.638836	0.124394	0.213439	0.071*	
H3B	0.482450	0.112505	0.164325	0.071*	
C4	0.5225 (5)	0.1680 (3)	0.3335 (4)	0.0508 (11)	
N1	0.3086 (14)	0.4127 (12)	0.3028 (13)	0.036 (2)	0.594 (7)
C5	0.3571 (17)	0.4465 (14)	0.4106 (15)	0.035 (2)	0.594 (7)
H5	0.444357	0.428586	0.448093	0.042*	0.594 (7)
C6	0.2872 (16)	0.5058 (13)	0.4698 (14)	0.037 (2)	0.594 (7)
H6	0.325579	0.527181	0.546567	0.044*	0.594 (7)
C7	0.1607 (18)	0.5340 (14)	0.4171 (10)	0.0380 (19)	0.594 (7)
C8	0.1061 (9)	0.4969 (6)	0.3077 (8)	0.0427 (18)	0.594 (7)
H8	0.017262	0.511208	0.270651	0.051*	0.594 (7)

C9	0.1836 (9)	0.4390 (6)	0.2544 (8)	0.0437 (19)	0.594 (7)
H9	0.146575	0.415819	0.178127	0.052*	0.594 (7)
N2	0.0880 (13)	0.5915 (10)	0.4784 (14)	0.040 (2)	0.594 (7)
H2	0.111539	0.589972	0.556019	0.048*	0.594 (7)
N1A	0.321 (2)	0.4209 (19)	0.294 (2)	0.037 (3)	0.406 (7)
C5A	0.348 (3)	0.438 (2)	0.410 (2)	0.038 (3)	0.406 (7)
H5AA	0.423078	0.408253	0.456121	0.046*	0.406 (7)
C6A	0.275 (2)	0.496 (2)	0.468 (2)	0.037 (3)	0.406 (7)
H6A	0.300689	0.506816	0.550128	0.044*	0.406 (7)
C7A	0.162 (3)	0.539 (2)	0.4037 (15)	0.040 (2)	0.406 (7)
C8A	0.1327 (13)	0.5229 (8)	0.2833 (11)	0.039 (2)	0.406 (7)
H8A	0.058397	0.552148	0.235498	0.047*	0.406 (7)
C9A	0.2128 (13)	0.4638 (8)	0.2336 (12)	0.040 (2)	0.406 (7)
H9A	0.189875	0.452713	0.150900	0.048*	0.406 (7)
N2A	0.0984 (19)	0.6021 (15)	0.466 (2)	0.040 (3)	0.406 (7)
H2AA	0.139846	0.618221	0.537260	0.048*	0.406 (7)
N3	−0.2448 (11)	0.7591 (8)	0.3590 (10)	0.0416 (19)	0.594 (7)
C10	−0.1977 (10)	0.7441 (7)	0.4736 (8)	0.041 (2)	0.594 (7)
H10	−0.243491	0.770769	0.530282	0.049*	0.594 (7)
C11	−0.0861 (9)	0.6921 (6)	0.5135 (8)	0.0417 (19)	0.594 (7)
H11	−0.056494	0.684199	0.596263	0.050*	0.594 (7)
C12	−0.0165 (12)	0.6511 (10)	0.4354 (10)	0.0403 (16)	0.594 (7)
C13	−0.0612 (7)	0.6711 (5)	0.3157 (6)	0.0472 (16)	0.594 (7)
H13	−0.014160	0.648297	0.257621	0.057*	0.594 (7)
C14	−0.1723 (7)	0.7233 (5)	0.2831 (6)	0.0500 (16)	0.594 (7)
H14	−0.200814	0.735312	0.201164	0.060*	0.594 (7)
N3A	−0.2610 (16)	0.7430 (11)	0.3535 (15)	0.043 (2)	0.406 (7)
C10A	−0.1774 (15)	0.7604 (11)	0.4548 (13)	0.043 (3)	0.406 (7)
H10A	−0.199719	0.808832	0.503081	0.051*	0.406 (7)
C11A	−0.0608 (14)	0.7126 (9)	0.4939 (12)	0.044 (2)	0.406 (7)
H11A	−0.005251	0.727873	0.567026	0.053*	0.406 (7)
C12A	−0.0261 (18)	0.6417 (14)	0.4245 (16)	0.042 (2)	0.406 (7)
C13A	−0.1218 (10)	0.6158 (7)	0.3269 (9)	0.048 (2)	0.406 (7)
H13A	−0.109876	0.561189	0.284776	0.057*	0.406 (7)
C14A	−0.2321 (10)	0.6688 (7)	0.2921 (9)	0.046 (2)	0.406 (7)
H14A	−0.291507	0.652863	0.221304	0.055*	0.406 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0394 (3)	0.0317 (3)	0.0326 (3)	−0.00311 (19)	0.0064 (2)	−0.00321 (18)
O1	0.134 (3)	0.0361 (15)	0.0441 (17)	0.0179 (17)	0.0256 (18)	0.0099 (13)
O2	0.072 (2)	0.0348 (14)	0.0343 (14)	0.0032 (13)	0.0165 (13)	−0.0015 (11)
O3	0.0441 (17)	0.0523 (17)	0.0486 (17)	−0.0074 (13)	0.0091 (13)	−0.0146 (13)
O4	0.0414 (19)	0.112 (3)	0.068 (2)	−0.0160 (19)	0.0092 (16)	−0.019 (2)
O5	0.064 (2)	0.0358 (14)	0.0430 (16)	−0.0068 (14)	0.0070 (13)	0.0013 (13)
N4	0.058 (2)	0.0352 (16)	0.0286 (15)	0.0038 (15)	0.0091 (14)	−0.0023 (12)
C1	0.046 (3)	0.064 (3)	0.039 (2)	−0.003 (2)	0.0086 (19)	−0.0007 (19)

C2	0.049 (3)	0.067 (3)	0.047 (2)	0.007 (2)	0.011 (2)	−0.003 (2)
C3	0.106 (4)	0.038 (2)	0.038 (2)	0.014 (2)	0.027 (2)	0.0018 (17)
C4	0.084 (3)	0.037 (2)	0.034 (2)	−0.002 (2)	0.018 (2)	−0.0020 (17)
N1	0.035 (3)	0.037 (4)	0.037 (3)	−0.002 (3)	0.006 (3)	−0.002 (3)
C5	0.031 (3)	0.035 (4)	0.038 (3)	−0.003 (3)	0.005 (3)	−0.001 (3)
C6	0.034 (4)	0.041 (4)	0.036 (3)	−0.005 (3)	0.007 (3)	−0.001 (3)
C7	0.033 (3)	0.040 (3)	0.042 (3)	0.000 (3)	0.007 (3)	0.002 (3)
C8	0.034 (3)	0.045 (4)	0.046 (4)	−0.002 (3)	0.001 (3)	0.002 (3)
C9	0.040 (4)	0.044 (4)	0.043 (4)	−0.005 (3)	−0.002 (3)	−0.001 (3)
N2	0.033 (3)	0.048 (4)	0.041 (3)	0.008 (3)	0.013 (3)	0.004 (3)
N1A	0.036 (4)	0.037 (4)	0.037 (4)	−0.001 (4)	0.003 (4)	−0.005 (4)
C5A	0.036 (4)	0.037 (5)	0.041 (4)	0.000 (4)	0.005 (4)	0.000 (4)
C6A	0.034 (4)	0.041 (5)	0.036 (4)	−0.004 (4)	0.007 (4)	0.001 (4)
C7A	0.035 (3)	0.040 (4)	0.044 (4)	0.002 (3)	0.008 (3)	0.002 (4)
C8A	0.035 (4)	0.043 (4)	0.037 (4)	0.004 (4)	0.003 (3)	0.006 (4)
C9A	0.040 (4)	0.043 (5)	0.035 (4)	0.001 (4)	0.005 (4)	0.002 (4)
N2A	0.037 (4)	0.043 (4)	0.041 (4)	0.004 (4)	0.011 (4)	0.004 (4)
N3	0.042 (3)	0.043 (4)	0.040 (3)	0.012 (3)	0.008 (3)	0.007 (3)
C10	0.043 (4)	0.043 (4)	0.039 (3)	0.002 (3)	0.013 (3)	0.000 (3)
C11	0.041 (4)	0.045 (4)	0.041 (3)	0.005 (3)	0.011 (3)	0.007 (3)
C12	0.033 (3)	0.047 (3)	0.041 (3)	0.005 (3)	0.009 (3)	0.003 (3)
C13	0.044 (3)	0.059 (3)	0.042 (3)	0.016 (3)	0.019 (3)	0.005 (3)
C14	0.048 (3)	0.057 (4)	0.046 (3)	0.013 (3)	0.011 (3)	0.011 (3)
N3A	0.042 (4)	0.045 (5)	0.040 (4)	0.010 (4)	0.005 (3)	0.003 (3)
C10A	0.046 (4)	0.041 (4)	0.042 (4)	0.009 (4)	0.011 (4)	−0.004 (4)
C11A	0.040 (4)	0.050 (5)	0.043 (4)	0.005 (4)	0.008 (4)	0.002 (4)
C12A	0.037 (4)	0.047 (4)	0.045 (4)	0.010 (3)	0.014 (3)	0.003 (3)
C13A	0.044 (4)	0.052 (4)	0.048 (4)	0.013 (3)	0.012 (3)	−0.007 (3)
C14A	0.043 (4)	0.052 (4)	0.043 (4)	0.013 (4)	0.006 (3)	−0.003 (3)

Geometric parameters (Å, º)

Ni1—O2	2.078 (3)	N2—C12	1.391 (10)
Ni1—O3	2.043 (3)	N1A—C5A	1.339 (13)
Ni1—O5	2.095 (3)	N1A—C9A	1.343 (12)
Ni1—N4	2.096 (3)	C5A—H5AA	0.9500
Ni1—N1	2.086 (14)	C5A—C6A	1.371 (12)
Ni1—N1A	2.02 (2)	C6A—H6A	0.9500
Ni1—N3^{i}	2.132 (10)	C6A—C7A	1.390 (12)
Ni1—N3A^{i}	2.121 (14)	C7A—C8A	1.381 (12)
O1—C4	1.241 (5)	C7A—N2A	1.400 (13)
O2—C4	1.257 (5)	C8A—H8A	0.9500
O3—C1	1.267 (5)	C8A—C9A	1.378 (11)
O4—C1	1.242 (5)	C9A—H9A	0.9500
O5—H5A	0.812 (19)	N2A—H2AA	0.8800
O5—H5B	0.856 (19)	N2A—C12A	1.395 (13)
N4—H4	1.0000	N3—C10	1.334 (10)
N4—C2	1.475 (5)	N3—C14	1.347 (10)

N4—C3	1.467 (5)	C10—H10	0.9500
C1—C2	1.520 (6)	C10—C11	1.372 (9)
C2—H2A	0.9900	C11—H11	0.9500
C2—H2B	0.9900	C11—C12	1.378 (10)
C3—H3A	0.9900	C12—C13	1.397 (11)
C3—H3B	0.9900	C13—H13	0.9500
C3—C4	1.531 (6)	C13—C14	1.357 (8)
N1—C5	1.341 (10)	C14—H14	0.9500
N1—C9	1.348 (9)	N3A—C10A	1.334 (12)
C5—H5	0.9500	N3A—C14A	1.351 (12)
C5—C6	1.374 (9)	C10A—H10A	0.9500
C6—H6	0.9500	C10A—C11A	1.378 (12)
C6—C7	1.382 (10)	C11A—H11A	0.9500
C7—C8	1.388 (10)	C11A—C12A	1.388 (12)
C7—N2	1.391 (9)	C12A—C13A	1.395 (12)
C8—H8	0.9500	C13A—H13A	0.9500
C8—C9	1.373 (9)	C13A—C14A	1.360 (10)
C9—H9	0.9500	C14A—H14A	0.9500
N2—H2	0.8800		

O2—Ni1—O5	176.65 (12)	C8—C7—N2	122.0 (13)
O2—Ni1—N4	82.18 (11)	C7—C8—H8	120.8
O2—Ni1—N1	91.2 (4)	C9—C8—C7	118.4 (8)
O2—Ni1—N3^{i}	93.4 (3)	C9—C8—H8	120.8
O2—Ni1—N3A^{i}	87.9 (4)	N1—C9—C8	124.5 (8)
O3—Ni1—O2	86.66 (11)	N1—C9—H9	117.7
O3—Ni1—O5	91.05 (11)	C8—C9—H9	117.7
O3—Ni1—N4	82.64 (12)	C7—N2—H2	115.2
O3—Ni1—N1	95.3 (5)	C7—N2—C12	129.6 (13)
O3—Ni1—N3^{i}	177.8 (4)	C12—N2—H2	115.2
O3—Ni1—N3A^{i}	170.6 (5)	C5A—N1A—Ni1	123.6 (14)
O5—Ni1—N4	95.11 (12)	C5A—N1A—C9A	115.3 (14)
O5—Ni1—N3^{i}	88.8 (3)	C9A—N1A—Ni1	120.9 (13)
O5—Ni1—N3A^{i}	94.0 (4)	N1A—C5A—H5AA	117.8
N4—Ni1—N3^{i}	95.2 (4)	N1A—C5A—C6A	124.3 (16)
N4—Ni1—N3A^{i}	89.0 (6)	C6A—C5A—H5AA	117.8
N1—Ni1—O5	91.5 (4)	C5A—C6A—H6A	120.3
N1—Ni1—N4	173.1 (4)	C5A—C6A—C7A	119.3 (15)
N1—Ni1—N3^{i}	87.0 (6)	C7A—C6A—H6A	120.3
N1—Ni1—N3A^{i}	92.5 (8)	C6A—C7A—N2A	116.1 (16)
N1A—Ni1—O2	95.2 (7)	C8A—C7A—C6A	117.6 (13)
N1A—Ni1—O3	91.6 (8)	C8A—C7A—N2A	125.9 (18)
N1A—Ni1—O5	87.2 (7)	C7A—C8A—H8A	120.6
N1A—Ni1—N4	173.8 (7)	C9A—C8A—C7A	118.8 (11)
N1A—Ni1—N3A^{i}	96.5 (10)	C9A—C8A—H8A	120.6
C4—O2—Ni1	113.6 (2)	N1A—C9A—C8A	124.7 (12)
C1—O3—Ni1	115.5 (3)	N1A—C9A—H9A	117.7
Ni1—O5—H5A	119 (3)	C8A—C9A—H9A	117.7

Ni1—O5—H5B	112 (3)	C7A—N2A—H2AA	117.5
H5A—O5—H5B	105 (5)	C12A—N2A—C7A	125 (2)
Ni1—N4—H4	110.0	C12A—N2A—H2AA	117.5
C2—N4—Ni1	107.6 (2)	C10—N3—Ni1^{ii}	126.0 (8)
C2—N4—H4	110.0	C10—N3—C14	115.8 (9)
C3—N4—Ni1	105.8 (2)	C14—N3—Ni1^{ii}	118.2 (7)
C3—N4—H4	110.0	N3—C10—H10	118.4
C3—N4—C2	113.2 (4)	N3—C10—C11	123.1 (9)
O3—C1—C2	117.7 (4)	C11—C10—H10	118.4
O4—C1—O3	125.3 (4)	C10—C11—H11	119.5
O4—C1—C2	117.0 (4)	C10—C11—C12	120.9 (8)
N4—C2—C1	113.3 (3)	C12—C11—H11	119.5
N4—C2—H2A	108.9	N2—C12—C13	124.8 (11)
N4—C2—H2B	108.9	C11—C12—N2	119.3 (11)
C1—C2—H2A	108.9	C11—C12—C13	115.9 (8)
C1—C2—H2B	108.9	C12—C13—H13	120.2
H2A—C2—H2B	107.7	C14—C13—C12	119.6 (7)
N4—C3—H3A	108.9	C14—C13—H13	120.2
N4—C3—H3B	108.9	N3—C14—C13	124.5 (7)
N4—C3—C4	113.4 (3)	N3—C14—H14	117.8
H3A—C3—H3B	107.7	C13—C14—H14	117.8
C4—C3—H3A	108.9	C10A—N3A—C14A	116.2 (13)
C4—C3—H3B	108.9	N3A—C10A—H10A	117.9
O1—C4—O2	124.6 (4)	N3A—C10A—C11A	124.3 (13)
O1—C4—C3	117.2 (4)	C11A—C10A—H10A	117.9
O2—C4—C3	118.2 (4)	C10A—C11A—H11A	120.6
C5—N1—Ni1	121.6 (10)	C10A—C11A—C12A	118.8 (11)
C5—N1—C9	116.0 (10)	C12A—C11A—H11A	120.6
C9—N1—Ni1	122.4 (9)	N2A—C12A—C13A	128.5 (15)
N1—C5—H5	118.3	C11A—C12A—N2A	114.8 (14)
N1—C5—C6	123.4 (11)	C11A—C12A—C13A	116.6 (11)
C6—C5—H5	118.3	C12A—C13A—H13A	120.0
C5—C6—H6	120.1	C14A—C13A—C12A	120.1 (10)
C5—C6—C7	119.8 (10)	C14A—C13A—H13A	120.0
C7—C6—H6	120.1	N3A—C14A—C13A	123.1 (10)
C6—C7—C8	117.9 (9)	N3A—C14A—H14A	118.5
C6—C7—N2	119.8 (12)	C13A—C14A—H14A	118.5

Ni1—O2—C4—O1	−175.9 (4)	C9—N1—C5—C6	−1 (3)
Ni1—O2—C4—C3	2.7 (5)	N2—C7—C8—C9	−178.4 (14)
Ni1—O3—C1—O4	−173.2 (4)	N2—C12—C13—C14	173.6 (14)
Ni1—O3—C1—C2	8.6 (5)	N1A—C5A—C6A—C7A	2 (5)
Ni1—N4—C2—C1	18.7 (4)	C5A—N1A—C9A—C8A	1 (4)
Ni1—N4—C3—C4	−26.9 (5)	C5A—C6A—C7A—C8A	−2 (5)
Ni1—N1—C5—C6	175.7 (14)	C5A—C6A—C7A—N2A	−175 (3)
Ni1—N1—C9—C8	−176.5 (8)	C6A—C7A—C8A—C9A	2 (4)
Ni1—N1A—C5A—C6A	−177 (2)	C6A—C7A—N2A—C12A	−170 (3)
Ni1—N1A—C9A—C8A	176.5 (12)	C7A—C8A—C9A—N1A	−1 (3)

Ni1^{ii}—N3—C10—C11	175.2 (7)	C7A—N2A—C12A—C11A	−171 (2)
Ni1^{ii}—N3—C14—C13	−175.3 (7)	C7A—N2A—C12A—C13A	13 (5)
Ni1^{ii}—N3A—C10A—C11A	−171.5 (11)	C8A—C7A—N2A—C12A	18 (5)
Ni1^{ii}—N3A—C14A—C13A	174.3 (11)	C9A—N1A—C5A—C6A	−1 (5)
O3—C1—C2—N4	−19.1 (6)	N2A—C7A—C8A—C9A	174 (2)
O4—C1—C2—N4	162.5 (4)	N2A—C12A—C13A—C14A	−173 (2)
N4—C3—C4—O1	−163.7 (4)	N3—C10—C11—C12	−0.6 (17)
N4—C3—C4—O2	17.6 (6)	C10—N3—C14—C13	3.0 (17)
C2—N4—C3—C4	90.7 (5)	C10—C11—C12—N2	−173.7 (13)
C3—N4—C2—C1	−97.9 (4)	C10—C11—C12—C13	4.1 (18)
N1—C5—C6—C7	−1 (3)	C11—C12—C13—C14	−4.1 (18)
C5—N1—C9—C8	0 (3)	C12—C13—C14—N3	0.5 (16)
C5—C6—C7—C8	3 (3)	C14—N3—C10—C11	−3.0 (17)
C5—C6—C7—N2	177.8 (17)	N3A—C10A—C11A—C12A	0 (3)
C6—C7—C8—C9	−4 (3)	C10A—N3A—C14A—C13A	−3 (3)
C6—C7—N2—C12	157 (2)	C10A—C11A—C12A—N2A	175.1 (19)
C7—C8—C9—N1	2.4 (17)	C10A—C11A—C12A—C13A	−8 (3)
C7—N2—C12—C11	171.1 (16)	C11A—C12A—C13A—C14A	11 (3)
C7—N2—C12—C13	−7 (3)	C12A—C13A—C14A—N3A	−6 (2)
C8—C7—N2—C12	−29 (3)	C14A—N3A—C10A—C11A	5 (3)

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*
O5—H5*A*···O1^{iii}	0.81 (2)	1.99 (2)	2.774 (4)	161 (5)
O5—H5*B*···O1^{iv}	0.86 (2)	1.86 (2)	2.706 (5)	172 (5)
N4—H4···O2^{iv}	1.00	2.28	3.233 (4)	158
C2—H2*A*···O1^{iv}	0.99	2.58	3.374 (6)	137
C6—H6···O3^{v}	0.95	2.32	3.259 (14)	170
C9—H9···N3^{i}	0.95	2.56	3.037 (16)	112
N2—H2···O4^{v}	0.88	1.86	2.718 (16)	166
C6*A*—H6*A*···O3^{v}	0.95	2.50	3.37 (2)	152
C9*A*—H9*A*···O5	0.95	2.47	2.952 (13)	111
N2*A*—H2*AA*···O4^{v}	0.88	1.99	2.82 (2)	156
C10*A*—H10*A*···O5^{ii}	0.95	2.52	3.131 (15)	123
C14*A*—H14*A*···O2^{ii}	0.95	2.28	2.904 (9)	122

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