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(5-Aminoisophthalato-κN)triaqua(1,10-phenanthroline-$\kappa^2 N,N'$)nickel(II) trihydrate

Han Huang,[a] Kou-Lin Zhang[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Chemical Engineering, Yangzhou University, Yangzhou 225002, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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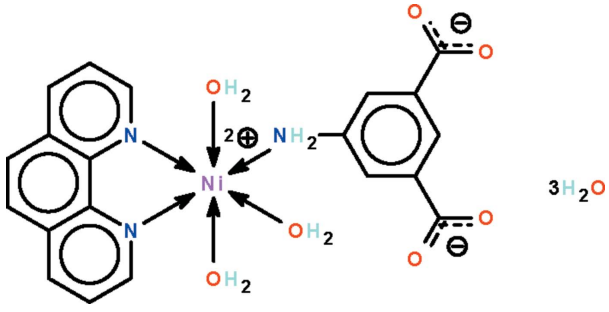
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.081; data-to-parameter ratio = 14.5.

The Ni^{II} atom in the title compound, $[Ni(C_8H_5NO_4)(C_{12}H_8N_2)(H_2O)_3]\cdot 3H_2O$, is six-coordinated in an NiN_3O_3 octahedral geometry. The triply water-coordinated Ni^{II} atom is chelated by the phenantroline ligand and is additionally coordinated by the amino group of the 5-aminoisophtalate anion. The anion, the coordinated and the uncoordinated water molecules interact through an extensive O—H···O and N—H···O hydrogen-bonding network, generating a three-dimensional cage-like network.

Related literature

For the isotypic Co^{II} analog, see: Zhang *et al.* (2010).



Experimental

Crystal data

$[Ni(C_8H_5NO_4)(C_{12}H_8N_2)(H_2O)_3]\cdot 3H_2O$
M_r = 526.14
Monoclinic, $P2_1/n$
a = 10.1039 (10) Å
b = 13.9448 (14) Å
c = 16.4237 (16) Å
β = 95.522 (1)°
V = 2303.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.90 mm^{-1}
T = 295 K
0.13 × 0.12 × 0.10 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.892, T_{max} = 0.915
20080 measured reflections
5258 independent reflections
3702 reflections with $I > 2\sigma(I)$
R_{int} = 0.092

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.081
S = 0.89
5258 reflections
363 parameters
14 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1*w*	2.0408 (16)	Ni1—N3	2.0883 (16)
Ni1—N2	2.0800 (16)	Ni1—O3*w*	2.0965 (15)
Ni1—O2*w*	2.0797 (14)	Ni1—N1	2.1409 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O6*w*^{i}	0.84 (1)	1.91 (1)	2.747 (2)	177 (3)
O1*w*—H1*w*2···O2^{ii}	0.84 (1)	1.82 (1)	2.654 (2)	180 (2)
O2*w*—H2*w*1···O5*w*^{i}	0.85 (1)	1.94 (1)	2.781 (2)	173 (3)
O2*w*—H2*w*2···O4^{iii}	0.85 (1)	1.98 (1)	2.832 (2)	178 (3)
O3*w*—H3*w*1···O5*w*^{iv}	0.84 (1)	2.16 (2)	2.914 (3)	148 (3)
O3*w*—H3*w*2···O3^{ii}	0.85 (1)	1.88 (1)	2.721 (2)	173 (3)
O4*w*—H4*w*1···O6*w*^{v}	0.85 (1)	1.97 (1)	2.817 (3)	171 (3)
O4*w*—H4*w*2···O2^{iv}	0.85 (1)	2.16 (2)	2.915 (3)	149 (3)
O5*w*—H5*w*1···O1	0.85 (1)	1.90 (1)	2.726 (3)	163 (3)
O5*w*—H5*w*2···O3^{vi}	0.84 (1)	1.91 (1)	2.716 (2)	159 (3)
O6*w*—H6*w*1···O1	0.85 (1)	1.83 (1)	2.678 (2)	177 (3)
O6*w*—H6*w*2···O4^{iii}	0.85 (1)	1.95 (1)	2.791 (2)	176 (3)
N1—H1···O4*w*	0.85 (1)	2.08 (1)	2.928 (3)	173 (2)
N1—H2···O4^{iii}	0.85 (1)	2.30 (1)	3.116 (2)	162 (2)

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x-1, y, z$; (v) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from an isotypic structure (Zhang *et al.*, 2010); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2464).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Winsonsin, USA.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zhang, K.-L., Diao, G.-W. & Ng, S. W. (2010). *Acta Cryst.* E**66**, m1421.

supporting information

Acta Cryst. (2011). E**67**, m444–m445 [doi:10.1107/S1600536811008919]

(5-Aminoisophthalato-κN)triaqua(1,10-phenanthroline-$\kappa^2 N,N'$)nickel(II) trihydrate

Han Huang, Kou-Lin Zhang and Seik Weng Ng

S1. Comment

We reported the structure of $[Co(C_8H_5NO_4)(C_{12}H_8N_2)(H_2O)_3]\cdot 3H_2O]$, whose 5-aminoisophthlate dianion binds only through the neutral amino donor site. The coordinated water molecules comprise the *fac* points of the NiN_3O_3 octahedron (Zhang *et al.*, 2010). The nickel analog (Scheme I) is isotypic. The dianion, the coordinated and the lattice water molecules interact through hydrogen bonds (Table 1) to furnish a tightly-held, three-dimensional network. Pairs of phenanthroline units show $\pi\cdots\pi$ interactions about a center-of-inversion at a distance of *ca* 3.5 Å.

S2. Experimental

Nickel nitrate hexahydrate (0.048 g, 0.165 mmol) dissolved in water (5 ml) was added to a mixture of 5-aminoisophthalic acid (0.030 g, 0.165 mmol) and sodium hydroxide (0.013 g, 0.330 mmol) dissolved in water (5 ml). To this solution was added 1,10-phenanthroline (0.033 g, 0.165 mmol) dissolved in methanol (10 ml). The mixture was filtered and set aside for the growth of green crystals.

S3. Refinement

Hydrogen atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The amino and water bound H-atoms were located in difference Fourier maps, and were refined with a distance restraint of N–H = O–H = 0.85 (1) Å. Their temperature factors were freely refined.

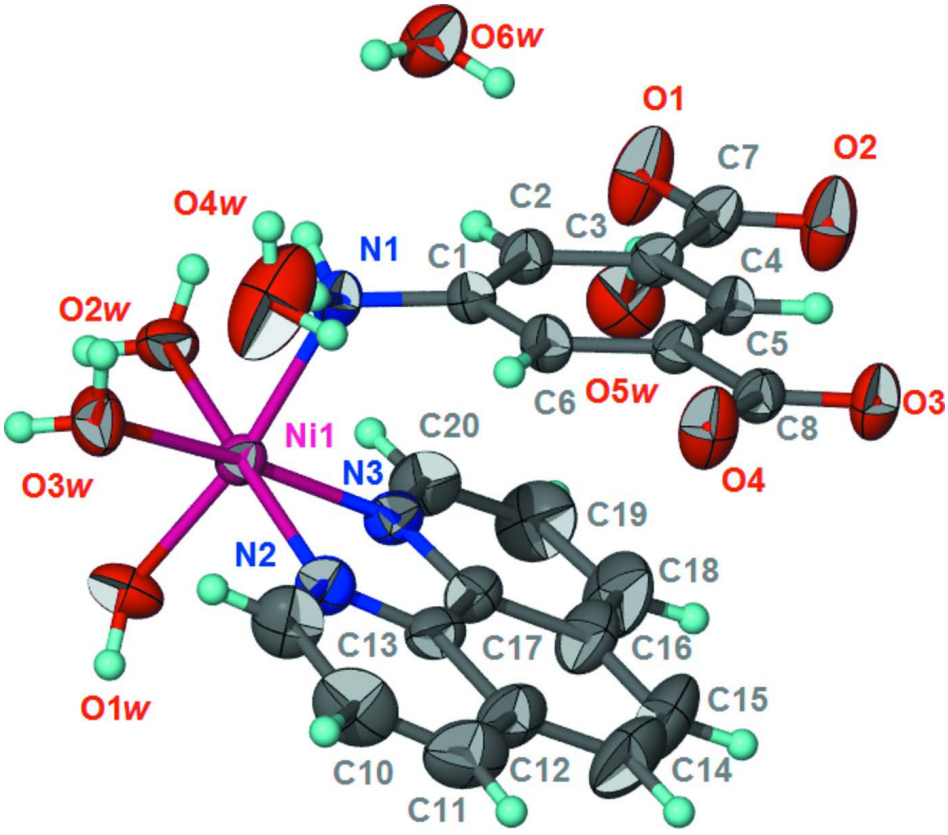

Figure 1

The molecular entities of (I) with atom labelling and displacement parameters at the 70% probability level; hydrogen atoms are shown as spheres of arbitrary radius.

(5-Aminoisophthalato-*κN*)triaqua(1,10-phenanthroline- κ^2N,N')nickel(II) trihydrate

Crystal data

$[Ni(C_8H_5NO_4)(C_{12}H_8N_2)(H_2O)_3]\cdot 3H_2O$
$M_r = 526.14$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.1039\ (10)$ Å
$b = 13.9448\ (14)$ Å
$c = 16.4237\ (16)$ Å
$\beta = 95.522\ (1)°$
$V = 2303.3\ (4)$ Å^3
$Z = 4$
$F(000) = 1096$
$D_x = 1.517$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5121 reflections
$\theta = 2.3–26.3°$
$\mu = 0.90$ mm^{-1}
$T = 295$ K
Block, green
0.13 × 0.12 × 0.10 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.892$, $T_{max} = 0.915$
20080 measured reflections
5258 independent reflections
3702 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -13\rightarrow12$
$k = -18\rightarrow18$
$l = -21\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.081$
$S = 0.89$
5258 reflections
363 parameters
14 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
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.74294 (2)	0.686043 (16)	0.465623 (15)	0.02405 (8)
O1	1.15737 (15)	0.53335 (11)	0.26472 (11)	0.0506 (5)
O2	1.22244 (15)	0.64153 (11)	0.18022 (11)	0.0484 (4)
O3	0.91901 (14)	0.91569 (10)	0.09761 (9)	0.0368 (4)
O4	0.72199 (14)	0.92528 (10)	0.14342 (10)	0.0413 (4)
O1w	0.76412 (16)	0.70612 (11)	0.58928 (10)	0.0360 (4)
H1w1	0.8275 (18)	0.6793 (16)	0.6169 (14)	0.054 (8)*
H1w2	0.751 (2)	0.7541 (11)	0.6177 (12)	0.044 (7)*
O2w	0.79265 (16)	0.54505 (10)	0.49643 (10)	0.0331 (3)
H2w1	0.743 (2)	0.5271 (18)	0.5323 (12)	0.059 (9)*
H2w2	0.787 (3)	0.5091 (17)	0.4543 (11)	0.074 (10)*
O3w	0.53887 (15)	0.66633 (12)	0.47353 (11)	0.0370 (4)
H3w1	0.493 (3)	0.642 (2)	0.4334 (14)	0.097 (12)*
H3w2	0.507 (3)	0.6419 (18)	0.5148 (11)	0.071 (9)*
O4w	0.4426 (2)	0.71882 (19)	0.28820 (16)	0.0763 (7)
H4w1	0.449 (3)	0.7708 (14)	0.2618 (18)	0.090 (12)*
H4w2	0.403 (3)	0.681 (2)	0.2532 (18)	0.111 (16)*
O5w	1.36084 (19)	0.52842 (12)	0.38634 (12)	0.0482 (4)
H5w1	1.309 (2)	0.5233 (18)	0.3428 (10)	0.055 (9)*
H5w2	1.4241 (18)	0.4915 (16)	0.3787 (16)	0.061 (9)*
O6w	1.03103 (17)	0.37826 (11)	0.31434 (11)	0.0426 (4)
H6w1	1.069 (3)	0.4278 (14)	0.2974 (18)	0.088 (11)*
H6w2	0.9539 (14)	0.3894 (18)	0.3281 (16)	0.062 (9)*
N1	0.70709 (18)	0.64237 (12)	0.34047 (11)	0.0268 (4)
H1	0.6280 (12)	0.6600 (15)	0.3259 (14)	0.042 (7)*
H2	0.708 (2)	0.5815 (7)	0.3415 (14)	0.044 (7)*
N2	0.71486 (16)	0.83087 (11)	0.43901 (11)	0.0306 (4)
N3	0.93864 (16)	0.72811 (11)	0.45417 (11)	0.0282 (4)
C1	0.79789 (19)	0.67558 (13)	0.28500 (12)	0.0247 (4)
C2	0.91676 (19)	0.62745 (13)	0.27875 (12)	0.0266 (4)
H2A	0.9348	0.5716	0.3087	0.032*
C3	1.00920 (19)	0.66171 (13)	0.22835 (12)	0.0257 (4)
C4	0.98077 (19)	0.74537 (13)	0.18383 (13)	0.0265 (4)

H4	1.0420	0.7689	0.1501	0.032*
C5	0.86192 (19)	0.79422 (13)	0.18922 (12)	0.0248 (4)
C6	0.77066 (19)	0.75923 (13)	0.24048 (12)	0.0263 (4)
H6	0.6915	0.7919	0.2449	0.032*
C7	1.1392 (2)	0.60854 (14)	0.22376 (14)	0.0301 (5)
C8	0.83267 (19)	0.88458 (13)	0.14050 (13)	0.0260 (4)
C9	0.6034 (2)	0.88175 (16)	0.43450 (14)	0.0418 (6)
H9	0.5250	0.8512	0.4450	0.050*
C10	0.5988 (3)	0.97908 (17)	0.41470 (16)	0.0499 (7)
H10	0.5190	1.0125	0.4130	0.060*
C11	0.7119 (3)	1.02457 (16)	0.39797 (15)	0.0501 (7)
H11	0.7097	1.0895	0.3852	0.060*
C12	0.8322 (2)	0.97357 (15)	0.40000 (15)	0.0415 (6)
C13	0.8284 (2)	0.87628 (14)	0.42274 (13)	0.0302 (5)
C14	0.9557 (3)	1.01401 (18)	0.38102 (17)	0.0592 (8)
H14	0.9587	1.0781	0.3657	0.071*
C15	1.0675 (3)	0.96117 (19)	0.38490 (18)	0.0606 (8)
H15	1.1459	0.9889	0.3708	0.073*
C16	1.0679 (2)	0.86237 (17)	0.41062 (16)	0.0447 (6)
C17	0.9485 (2)	0.82052 (14)	0.42931 (13)	0.0309 (5)
C18	1.1817 (2)	0.80332 (19)	0.41870 (18)	0.0561 (7)
H18	1.2631	0.8269	0.4055	0.067*
C19	1.1720 (2)	0.71140 (19)	0.44591 (17)	0.0518 (7)
H19	1.2472	0.6727	0.4530	0.062*
C20	1.0490 (2)	0.67576 (16)	0.46297 (14)	0.0376 (5)
H20	1.0438	0.6129	0.4812	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02322 (14)	0.02102 (12)	0.02825 (15)	0.00025 (10)	0.00421 (10)	−0.00008 (11)
O1	0.0315 (9)	0.0409 (9)	0.0806 (13)	0.0094 (7)	0.0107 (9)	0.0334 (9)
O2	0.0349 (9)	0.0411 (9)	0.0730 (12)	0.0125 (7)	0.0248 (8)	0.0254 (9)
O3	0.0359 (8)	0.0326 (8)	0.0437 (10)	0.0020 (6)	0.0139 (7)	0.0150 (7)
O4	0.0363 (9)	0.0308 (8)	0.0594 (11)	0.0116 (7)	0.0170 (8)	0.0170 (7)
O1w	0.0397 (9)	0.0367 (9)	0.0307 (9)	0.0105 (7)	−0.0013 (8)	−0.0102 (7)
O2w	0.0453 (10)	0.0239 (7)	0.0302 (9)	−0.0001 (7)	0.0043 (8)	−0.0023 (7)
O3w	0.0277 (8)	0.0503 (10)	0.0339 (10)	−0.0067 (7)	0.0073 (8)	0.0004 (8)
O4w	0.0468 (12)	0.0806 (16)	0.0972 (19)	−0.0116 (12)	−0.0149 (12)	0.0407 (15)
O5w	0.0492 (12)	0.0439 (10)	0.0510 (12)	0.0107 (9)	0.0022 (10)	0.0044 (9)
O6w	0.0340 (9)	0.0325 (8)	0.0620 (12)	0.0006 (7)	0.0077 (9)	0.0046 (8)
N1	0.0275 (10)	0.0246 (9)	0.0289 (10)	−0.0008 (7)	0.0050 (8)	0.0054 (8)
N2	0.0292 (9)	0.0276 (9)	0.0351 (10)	0.0039 (7)	0.0040 (8)	0.0003 (7)
N3	0.0255 (9)	0.0250 (8)	0.0339 (10)	0.0011 (7)	0.0026 (8)	−0.0019 (7)
C1	0.0277 (10)	0.0246 (10)	0.0220 (10)	−0.0023 (8)	0.0035 (8)	0.0005 (8)
C2	0.0298 (11)	0.0225 (9)	0.0272 (12)	0.0005 (8)	0.0020 (9)	0.0038 (8)
C3	0.0247 (11)	0.0233 (9)	0.0291 (12)	0.0007 (8)	0.0023 (9)	0.0010 (8)
C4	0.0255 (10)	0.0250 (9)	0.0297 (11)	−0.0002 (8)	0.0062 (9)	0.0047 (8)

C5	0.0254 (10)	0.0220 (9)	0.0273 (11)	0.0002 (7)	0.0036 (9)	0.0016 (8)
C6	0.0251 (10)	0.0251 (10)	0.0294 (12)	0.0047 (8)	0.0069 (9)	0.0023 (8)
C7	0.0251 (11)	0.0248 (10)	0.0400 (13)	0.0003 (8)	0.0013 (10)	0.0046 (9)
C8	0.0274 (11)	0.0218 (9)	0.0293 (12)	0.0009 (8)	0.0052 (9)	0.0020 (8)
C9	0.0379 (13)	0.0374 (12)	0.0502 (16)	0.0104 (10)	0.0054 (11)	0.0036 (11)
C10	0.0548 (16)	0.0411 (13)	0.0532 (17)	0.0242 (12)	0.0026 (13)	0.0047 (12)
C11	0.0728 (19)	0.0283 (12)	0.0472 (16)	0.0087 (12)	−0.0040 (14)	0.0060 (11)
C12	0.0558 (15)	0.0263 (11)	0.0416 (15)	−0.0030 (10)	−0.0001 (12)	0.0076 (10)
C13	0.0370 (12)	0.0245 (10)	0.0287 (12)	−0.0019 (9)	0.0005 (10)	−0.0008 (9)
C14	0.070 (2)	0.0360 (13)	0.070 (2)	−0.0163 (13)	0.0022 (16)	0.0167 (13)
C15	0.0546 (17)	0.0536 (16)	0.074 (2)	−0.0244 (14)	0.0083 (15)	0.0153 (15)
C16	0.0361 (13)	0.0468 (14)	0.0511 (16)	−0.0122 (11)	0.0034 (12)	0.0046 (12)
C17	0.0296 (11)	0.0293 (10)	0.0336 (12)	−0.0043 (9)	0.0023 (9)	−0.0005 (9)
C18	0.0264 (13)	0.0691 (18)	0.074 (2)	−0.0113 (12)	0.0101 (13)	0.0005 (15)
C19	0.0266 (13)	0.0561 (16)	0.072 (2)	0.0056 (11)	0.0036 (13)	−0.0040 (14)
C20	0.0308 (12)	0.0355 (11)	0.0457 (14)	0.0054 (9)	0.0000 (11)	−0.0018 (10)

Geometric parameters (Å, º)

Ni1—O1w	2.0408 (16)	C1—C2	1.388 (3)
Ni1—N2	2.0800 (16)	C2—C3	1.391 (3)
Ni1—O2w	2.0797 (14)	C2—H2A	0.9300
Ni1—N3	2.0883 (16)	C3—C4	1.392 (3)
Ni1—O3w	2.0965 (15)	C3—C7	1.516 (3)
Ni1—N1	2.1409 (18)	C4—C5	1.391 (3)
O1—C7	1.250 (2)	C4—H4	0.9300
O2—C7	1.244 (2)	C5—C6	1.395 (3)
O3—C8	1.250 (2)	C5—C8	1.507 (3)
O4—C8	1.259 (2)	C6—H6	0.9300
O1w—H1w1	0.84 (1)	C9—C10	1.395 (3)
O1w—H1w2	0.84 (1)	C9—H9	0.9300
O2w—H2w1	0.85 (1)	C10—C11	1.357 (3)
O2w—H2w2	0.85 (1)	C10—H10	0.9300
O3w—H3w1	0.84 (1)	C11—C12	1.406 (3)
O3w—H3w2	0.85 (1)	C11—H11	0.9300
O4w—H4w1	0.85 (1)	C12—C13	1.409 (3)
O4w—H4w2	0.85 (1)	C12—C14	1.431 (3)
O5w—H5w1	0.85 (1)	C13—C17	1.437 (3)
O5w—H5w2	0.84 (1)	C14—C15	1.346 (4)
O6w—H6w1	0.85 (1)	C14—H14	0.9300
O6w—H6w2	0.85 (1)	C15—C16	1.441 (3)
N1—C1	1.431 (2)	C15—H15	0.9300
N1—H1	0.85 (1)	C16—C17	1.401 (3)
N1—H2	0.85 (1)	C16—C18	1.410 (3)
N2—C9	1.327 (3)	C18—C19	1.364 (4)
N2—C13	1.359 (3)	C18—H18	0.9300
N3—C20	1.329 (2)	C19—C20	1.392 (3)
N3—C17	1.358 (2)	C19—H19	0.9300

C1—C6	1.390 (3)	C20—H20	0.9300
O1w—Ni1—N2	94.30 (7)	C5—C4—H4	119.6
O1w—Ni1—O2w	83.56 (6)	C4—C5—C6	119.46 (17)
N2—Ni1—O2w	173.68 (7)	C4—C5—C8	120.15 (17)
O1w—Ni1—N3	92.38 (7)	C6—C5—C8	120.39 (17)
N2—Ni1—N3	79.60 (6)	C1—C6—C5	120.14 (18)
O2w—Ni1—N3	94.52 (6)	C1—C6—H6	119.9
O1w—Ni1—O3w	88.05 (7)	C5—C6—H6	119.9
N2—Ni1—O3w	91.45 (6)	O2—C7—O1	123.19 (19)
O2w—Ni1—O3w	94.41 (6)	O2—C7—C3	118.99 (17)
N3—Ni1—O3w	171.05 (6)	O1—C7—C3	117.82 (19)
O1w—Ni1—N1	170.49 (6)	O3—C8—O4	122.37 (18)
N2—Ni1—N1	93.87 (7)	O3—C8—C5	118.53 (17)
O2w—Ni1—N1	88.81 (6)	O4—C8—C5	119.09 (17)
N3—Ni1—N1	93.88 (7)	N2—C9—C10	122.8 (2)
O3w—Ni1—N1	86.89 (7)	N2—C9—H9	118.6
Ni1—O1w—H1w1	118.7 (18)	C10—C9—H9	118.6
Ni1—O1w—H1w2	131.3 (16)	C11—C10—C9	119.5 (2)
H1w1—O1w—H1w2	102 (2)	C11—C10—H10	120.3
Ni1—O2w—H2w1	107.6 (18)	C9—C10—H10	120.3
Ni1—O2w—H2w2	111.2 (19)	C10—C11—C12	120.1 (2)
H2w1—O2w—H2w2	113 (3)	C10—C11—H11	120.0
Ni1—O3w—H3w1	119 (2)	C12—C11—H11	120.0
Ni1—O3w—H3w2	123.6 (19)	C11—C12—C13	116.6 (2)
H3w1—O3w—H3w2	104 (3)	C11—C12—C14	124.4 (2)
H4w1—O4w—H4w2	104 (3)	C13—C12—C14	118.9 (2)
H5w1—O5w—H5w2	104 (3)	N2—C13—C12	123.1 (2)
H6w1—O6w—H6w2	113 (3)	N2—C13—C17	117.12 (17)
C1—N1—Ni1	117.36 (13)	C12—C13—C17	119.8 (2)
C1—N1—H1	111.8 (16)	C15—C14—C12	121.3 (2)
Ni1—N1—H1	104.8 (16)	C15—C14—H14	119.3
C1—N1—H2	109.2 (16)	C12—C14—H14	119.3
Ni1—N1—H2	105.4 (16)	C14—C15—C16	121.1 (2)
H1—N1—H2	108 (2)	C14—C15—H15	119.5
C9—N2—C13	117.89 (18)	C16—C15—H15	119.5
C9—N2—Ni1	128.95 (15)	C17—C16—C18	116.6 (2)
C13—N2—Ni1	113.14 (13)	C17—C16—C15	119.0 (2)
C20—N3—C17	117.96 (18)	C18—C16—C15	124.4 (2)
C20—N3—Ni1	128.87 (14)	N3—C17—C16	123.43 (19)
C17—N3—Ni1	113.03 (13)	N3—C17—C13	116.75 (18)
C6—C1—C2	119.71 (18)	C16—C17—C13	119.82 (19)
C6—C1—N1	120.00 (17)	C19—C18—C16	119.7 (2)
C2—C1—N1	120.20 (17)	C19—C18—H18	120.1
C1—C2—C3	120.88 (18)	C16—C18—H18	120.1
C1—C2—H2A	119.6	C18—C19—C20	119.7 (2)
C3—C2—H2A	119.6	C18—C19—H19	120.1
C4—C3—C2	118.95 (18)	C20—C19—H19	120.1

C4—C3—C7	121.31 (18)	N3—C20—C19	122.5 (2)
C2—C3—C7	119.74 (17)	N3—C20—H20	118.7
C3—C4—C5	120.86 (18)	C19—C20—H20	118.7
C3—C4—H4	119.6		

N2—Ni1—N1—C1	63.44 (14)	C4—C5—C8—O4	176.82 (19)
O2w—Ni1—N1—C1	−110.83 (14)	C6—C5—C8—O4	−3.6 (3)
N3—Ni1—N1—C1	−16.38 (14)	C13—N2—C9—C10	−0.6 (3)
O3w—Ni1—N1—C1	154.68 (14)	Ni1—N2—C9—C10	−179.05 (18)
O1w—Ni1—N2—C9	−85.7 (2)	N2—C9—C10—C11	1.0 (4)
N3—Ni1—N2—C9	−177.4 (2)	C9—C10—C11—C12	0.5 (4)
O3w—Ni1—N2—C9	2.4 (2)	C10—C11—C12—C13	−2.2 (4)
N1—Ni1—N2—C9	89.4 (2)	C10—C11—C12—C14	178.1 (2)
O1w—Ni1—N2—C13	95.80 (15)	C9—N2—C13—C12	−1.2 (3)
N3—Ni1—N2—C13	4.16 (14)	Ni1—N2—C13—C12	177.41 (17)
O3w—Ni1—N2—C13	−176.05 (15)	C9—N2—C13—C17	178.95 (19)
N1—Ni1—N2—C13	−89.08 (15)	Ni1—N2—C13—C17	−2.4 (2)
O1w—Ni1—N3—C20	85.17 (19)	C11—C12—C13—N2	2.6 (3)
N2—Ni1—N3—C20	179.1 (2)	C14—C12—C13—N2	−177.6 (2)
O2w—Ni1—N3—C20	1.45 (19)	C11—C12—C13—C17	−177.6 (2)
N1—Ni1—N3—C20	−87.66 (19)	C14—C12—C13—C17	2.2 (3)
O1w—Ni1—N3—C17	−99.33 (15)	C11—C12—C14—C15	179.4 (3)
N2—Ni1—N3—C17	−5.39 (14)	C13—C12—C14—C15	−0.3 (4)
O2w—Ni1—N3—C17	176.95 (15)	C12—C14—C15—C16	−1.8 (5)
N1—Ni1—N3—C17	87.83 (15)	C14—C15—C16—C17	2.0 (4)
Ni1—N1—C1—C6	−93.21 (19)	C14—C15—C16—C18	−177.9 (3)
Ni1—N1—C1—C2	83.2 (2)	C20—N3—C17—C16	1.7 (3)
C6—C1—C2—C3	−0.1 (3)	Ni1—N3—C17—C16	−174.38 (18)
N1—C1—C2—C3	−176.57 (17)	C20—N3—C17—C13	−178.14 (19)
C1—C2—C3—C4	−0.1 (3)	Ni1—N3—C17—C13	5.8 (2)
C1—C2—C3—C7	179.01 (18)	C18—C16—C17—N3	0.0 (4)
C2—C3—C4—C5	−0.1 (3)	C15—C16—C17—N3	−179.9 (2)
C7—C3—C4—C5	−179.23 (18)	C18—C16—C17—C13	179.8 (2)
C3—C4—C5—C6	0.6 (3)	C15—C16—C17—C13	−0.1 (4)
C3—C4—C5—C8	−179.89 (18)	N2—C13—C17—N3	−2.3 (3)
C2—C1—C6—C5	0.5 (3)	C12—C13—C17—N3	177.8 (2)
N1—C1—C6—C5	177.01 (17)	N2—C13—C17—C16	177.8 (2)
C4—C5—C6—C1	−0.8 (3)	C12—C13—C17—C16	−2.0 (3)
C8—C5—C6—C1	179.70 (18)	C17—C16—C18—C19	−1.9 (4)
C4—C3—C7—O2	1.1 (3)	C15—C16—C18—C19	178.0 (3)
C2—C3—C7—O2	−177.98 (19)	C16—C18—C19—C20	2.0 (4)
C4—C3—C7—O1	−179.3 (2)	C17—N3—C20—C19	−1.5 (3)
C2—C3—C7—O1	1.7 (3)	Ni1—N3—C20—C19	173.77 (18)
C4—C5—C8—O3	−2.2 (3)	C18—C19—C20—N3	−0.3 (4)
C6—C5—C8—O3	177.34 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O6*w*^{i}	0.84 (1)	1.91 (1)	2.747 (2)	177 (3)
O1*w*—H1*w*2···O2^{ii}	0.84 (1)	1.82 (1)	2.654 (2)	180 (2)
O2*w*—H2*w*1···O5*w*^{i}	0.85 (1)	1.94 (1)	2.781 (2)	173 (3)
O2*w*—H2*w*2···O4^{iii}	0.85 (1)	1.98 (1)	2.832 (2)	178 (3)
O3*w*—H3*w*1···O5*w*^{iv}	0.84 (1)	2.16 (2)	2.914 (3)	148 (3)
O3*w*—H3*w*2···O3^{ii}	0.85 (1)	1.88 (1)	2.721 (2)	173 (3)
O4*w*—H4*w*1···O6*w*^{v}	0.85 (1)	1.97 (1)	2.817 (3)	171 (3)
O4*w*—H4*w*2···O2^{iv}	0.85 (1)	2.16 (2)	2.915 (3)	149 (3)
O5*w*—H5*w*1···O1	0.85 (1)	1.90 (1)	2.726 (3)	163 (3)
O5*w*—H5*w*2···O3^{vi}	0.84 (1)	1.91 (1)	2.716 (2)	159 (3)
O6*w*—H6*w*1···O1	0.85 (1)	1.83 (1)	2.678 (2)	177 (3)
O6*w*—H6*w*2···O4^{iii}	0.85 (1)	1.95 (1)	2.791 (2)	176 (3)
N1—H1···O4*w*	0.85 (1)	2.08 (1)	2.928 (3)	173 (2)
N1—H2···O4^{iii}	0.85 (1)	2.30 (1)	3.116 (2)	162 (2)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) *x*−1/2, −*y*+3/2, *z*+1/2; (iii) −*x*+3/2, *y*−1/2, −*z*+1/2; (iv) *x*−1, *y*, *z*; (v) −*x*+3/2, *y*+1/2, −*z*+1/2; (vi) −*x*+5/2, *y*−1/2, −*z*+1/2.