



Crystal structure and Hirshfeld surface analysis of tetraaquabis(isonicotinamide- κN^1)nickel(II) fumarate

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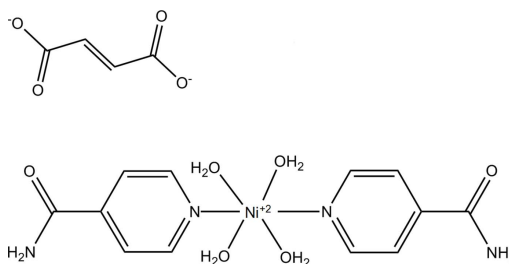
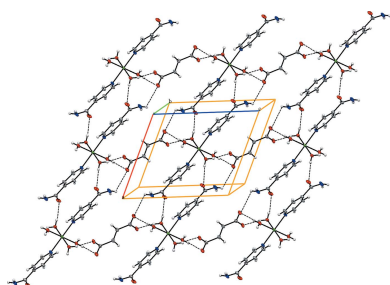
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Keywords: crystal structure; fumaric acid; isonicotinamide; nickel(II); Hirshfeld surface.**CCDC reference:** 1579677**Supporting information:** this article has supporting information at journals.iucr.org/e

The reaction of NiCl₂ with fumaric acid and isonicotinamide in a basic solution produces the title complex, [Ni(C₆H₆N₂O)₂(H₂O)₄](C₄H₂O₄). The nickel(II) ion of the complex cation and the fumarate anion are each located on an inversion centre. The Ni^{II} ion is coordinated octahedrally by four water O atoms and two N atoms of isonicotinamide molecules. The fumarate anion is linked to neighbouring complex cations *via* O_{water}—H···O_{fumarate} hydrogen bonds. In the crystal, the complex cations are further linked by O—H···O, N—H···O and C—H···O hydrogen bonds, forming a three-dimensional supramolecular architecture. Hirshfeld surface analysis and two-dimensional fingerprint plots were used to analyse the intermolecular interactions present in the crystal and indicate that the most important contributions for the crystal packing are from H···O/O···H (41.8%), H···H (35.3%) and H···C/C···H (10.2%) interactions.

1. Chemical context

Metal complexes of biologically important ligands are sometimes more effective than the free ligands. Many transition and heavy metal cations play an important role in the biological processes involved in the formation of vitamins and drug components. An important element for biological systems is nickel and nickel complexes have biological activities including antiepileptic, antimicrobial, antibacterial and anti-cancer activities (Bombicz *et al.*, 2001). Dicarboxylic acid ligands have been utilized primarily in the synthesis of a range of metal complexes. Dicarboxylic acids such as fumaric acid and amides have been particularly useful in creating many supramolecular structures (Pavlishchuk *et al.*, 2011; Ostrowska *et al.*, 2016), in particular isonicotinamide with a variety of carboxylic acids (Vishweshwar *et al.*, 2003; Aakeröy *et al.*, 2002).



We have prepared a new Ni^{II} complex, tetraaquabis(isonicotinamide- κN^1)nickel(II) fumarate, and determined its structure by single crystal X-ray diffraction. In addition, Hirshfeld surface analysis and fingerprint plots were used to

Table 1
 Selected geometric parameters (Å, °).

Ni1—O3	2.0537 (16)	Ni1—N1	2.1075 (18)
Ni1—O2	2.0812 (15)		
O3—Ni1—O2	92.00 (7)	O3—Ni1—N1	86.97 (7)
O3—Ni1—O2 ⁱ	88.00 (7)	O2—Ni1—N1	92.05 (6)
O3 ⁱ —Ni1—N1	93.03 (7)	O2 ⁱ —Ni1—N1	87.95 (7)

 Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

understand the intermolecular interactions in the crystal structure.

2. Structural commentary

The molecular structure of the title complex is illustrated in Fig. 1. The nickel(II) ion is octahedrally coordinated to four water O atoms and two N_{pyridine} atoms of isonicotinamide molecules. The values of the Ni—O_{water} and Ni—N_{pyridine} bond lengths and the bond angles involving atom Ni1 (Table 1) are close to those reported for similar nickel(II) complexes (Krämer *et al.*, 2002; Bora & Das, 2011; Moroz *et al.*, 2012).

3. Supramolecular features

In the crystal, each O atom of the fumarate dianion is linked to a water H atom *via* O—H···O hydrogen bonds, forming chains along the *c*-axis direction (Table 2, Fig. 2).

The fumarate anions and complex cations are linked by O—H···O hydrogen bonds; the complex cations also interact with each other through O—H···O, N—H···O and C—H···O hydrogen bonds, forming a three-dimensional supramolecular architecture.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.39, update of May 2018; Groom *et al.*, 2016) revealed the structures of four similar tetraaquabis(isonicotinamide- κN^1)nickel(II) complexes with different counter-anions *viz.*

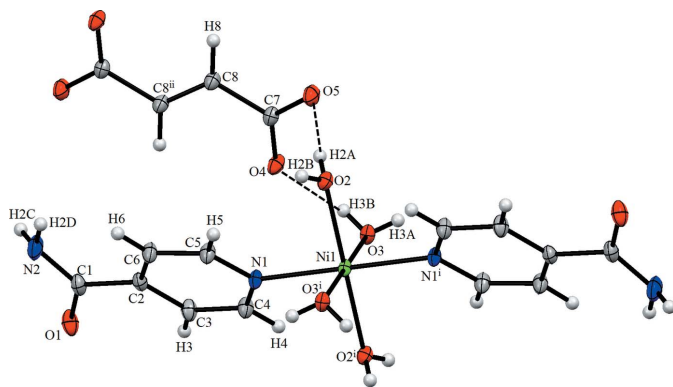


Figure 1
 The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 20% probability level. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z$.]

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O3—H3B···O4	0.80 (3)	1.89 (3)	2.678 (2)	169 (3)
O2—H2A···O5	0.78 (3)	2.01 (3)	2.791 (3)	175 (3)
C6—H6···O1 ⁱⁱⁱ	0.93	2.31	3.230 (3)	172
N2—H2D···O1 ⁱⁱⁱ	0.93 (5)	2.31 (5)	3.230 (3)	172 (4)
C5—H5···O4 ^{iv}	0.93	2.38	3.282 (3)	165
O2—H2B···O4 ^{iv}	0.73 (3)	2.02 (3)	2.739 (2)	172 (3)
O3—H3A···O1 ^v	0.73 (3)	2.07 (3)	2.798 (3)	175 (3)

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

bis(4-formylbenzoate) dihydrate (HUCLAT; Hökelek *et al.*, 2009), bis(3-hydroxybenzoate) tetrahydrate (GANZAY; Zaman *et al.*, 2012), bis(thiophene-2,5-dicarboxylate) dihydrate (NETQIO; Liu *et al.*, 2012) and naphthalene-1,5-disulfonate tetrahydrate (TESDEC; Lian, 2012). In all four complexes, the cation possesses inversion symmetry with the nickel ion being located on a centre of symmetry. The Ni—O_{water} bond lengths vary from 2.044 to 2.086 Å, while the Ni—N_{pyridine} bond lengths vary from 2.075 to 2.098 Å. In the title complex, the cation also possesses inversion symmetry and the Ni—O_{water} bond lengths [2.0812 (15) and 2.0537 (16) Å] and the Ni—N_{pyridine} bond length [2.1075 (18) Å] fall within these limits.

5. Hirshfeld surface analysis

*Crystal Explorer*17.5 (Turner *et al.*, 2017) was used to investigate the Hirshfeld surfaces and to analyse the interactions in the crystal. The Hirshfeld surfaces mapped over d_{norm} , d_i and d_e are shown in Fig. 3. Red spots indicate the contacts involved in strong hydrogen bonds and interatomic contacts (Gümüş *et al.*, 2018; Sen *et al.*, 2018; Kansız & Dege, 2018); those in Fig. 3 correspond to the near-type H···O contacts resulting from C—H···O, O—H···O and N—H···O hydrogen bonds. The Hirshfeld surfaces were obtained using a standard surface

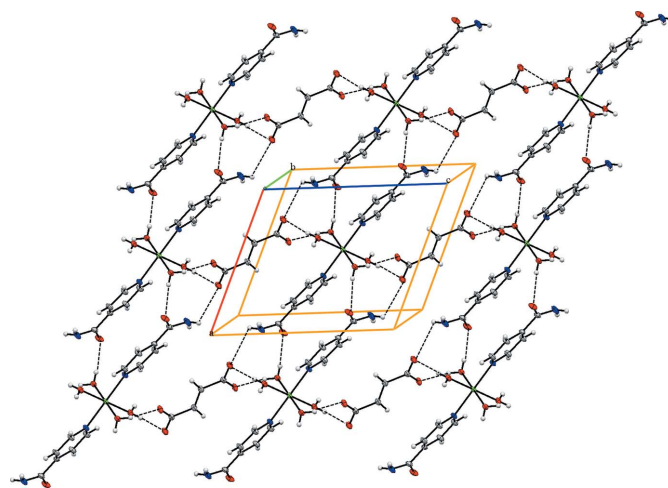


Figure 2
 A view of the crystal packing of the title compound. Dashed lines indicate hydrogen bonds.

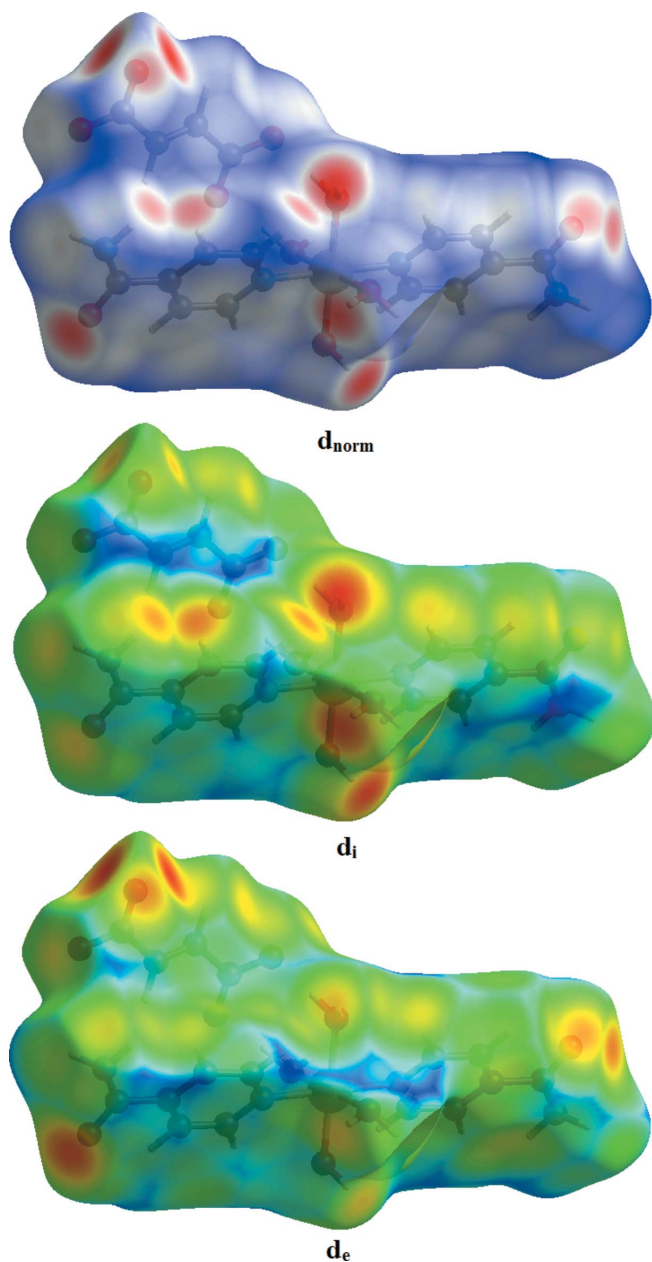


Figure 3
The Hirshfeld surface of the title compound mapped over d_{norm} , d_i and d_e .

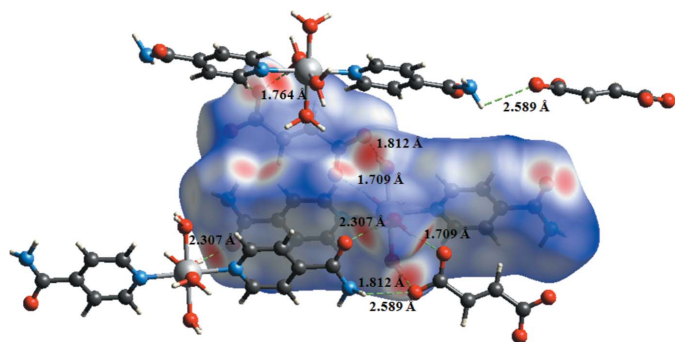


Figure 4
The Hirshfeld surface mapped over d_{norm} to visualize the intramolecular and intermolecular interactions in the title compound.

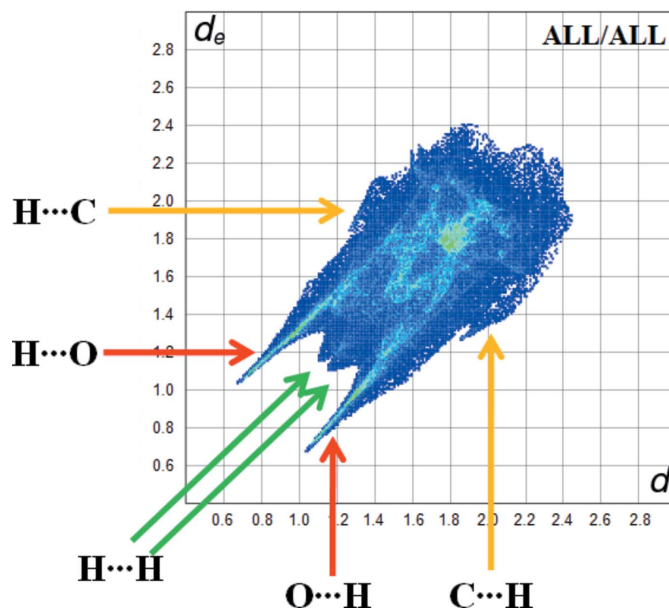


Figure 5
A fingerprint plot of the title compound.

(high) resolution with the three-dimensional d_{norm} surfaces mapped over a fixed colour scale of -0.701 (red) to 1.286 (blue) a.u. The red spots in Fig. 4 correspond to the near-type $H \cdots O$ contacts resulting from $O-H \cdots O$ and $N-H \cdots O$ hydrogen bonds. Fig. 5 shows the two-dimensional fingerprint plot of the sum of the contacts contributing to the Hirshfeld surface represented in normal mode. In Fig. 6a, the two symmetrical points at the top, bottom left and right with $d_e + d_i = 1.7 \text{ \AA}$ indicate the presence of $H \cdots O/O \cdots H$ (41.8%) contacts. Fig. 6b shows the two-dimensional fingerprint plot of the (d_i, d_e) points associated with hydrogen atoms and is characterized by an end point that points to the origin and corresponds to $d_i = d_e = 1.08 \text{ \AA}$, which indicates the presence of the $H \cdots H$ contacts (35.3%). Fig. 6c shows the contacts

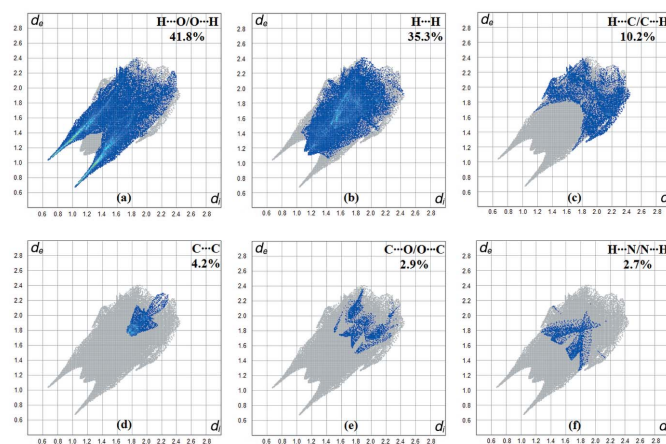


Figure 6
(a) $H \cdots O/O \cdots H$, (b) $H \cdots H$, (c) $H \cdots C/C \cdots H$, (d) $C \cdots C$, (e) $C \cdots O/O \cdots C$ and (f) $H \cdots N/N \cdots H$ contacts in the title complex, showing their percentage contributions to the Hirshfeld surface.

between the carbon atoms inside the surface and the hydrogen atoms outside the surface of Hirshfeld and *vice versa* (H...C/C...H) and has two symmetrical wings on the left and right sides (10.2%), C...C (4.2%), C...O/O...C (2.9%) and H...N/N...H (2.7%) contacts also contribute to the Hirshfeld surface.

6. Synthesis and crystallization

A solution of NaOH (52 mmol, 2.07 g) was added to an aqueous solution of fumaric acid (26 mmol, 3 g) under stirring. A solution of NiCl₂·6H₂O (25 mmol, 6.14 g) in methanol was then added. The mixture was heated at 353 K for 30 min. and then the blue mixture was filtered and left to dry at room temperature. The reaction mixture (0.88 mmol, 0.20 g) was dissolved in methanol and added to a ethanol solution of isonicotinamide (1.76 mmol, 0.21 g). The mixture was heated at 353 K for 60 min. under stirring and the resulting suspension was filtered and left to crystallize for three weeks at room temperature. The title compound was obtained as a blue solid and contained crystals suitable for X-ray diffraction analysis.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The water and NH₂ hydrogen atoms were located from difference-Fourier maps and freely refined. The C-bound H atoms were positioned geometrically and refined using a riding model: C–H = 0.93–0.97 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Acknowledgements

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Table 3

Experimental details.

Crystal data	
Chemical formula	[Ni(C ₆ H ₆ N ₂ O) ₂ (H ₂ O) ₄](C ₄ H ₂ O ₄)
<i>M_r</i>	489.08
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.6140 (8), 9.9819 (9), 11.3874 (10)
β (°)	113.157 (7)
<i>V</i> (Å ³)	1004.76 (16)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.03
Crystal size (mm)	0.58 × 0.50 × 0.39
Data collection	
Diffractometer	STOE IPDS 2
Absorption correction	Integration (<i>X-RED32</i> ; Stoe & Cie, 2002)
<i>T</i> _{min} , <i>T</i> _{max}	0.527, 0.593
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	5175, 2075, 1777
<i>R</i> _{int}	0.045
(sin θ/λ) _{max} (Å ⁻¹)	0.628
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.104, 1.05
No. of reflections	2075
No. of parameters	171
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.39, −0.83

Computer programs: *X-AREA* (Stoe & Cie, 2002), *X-RED* (Stoe & Cie, 2002), *SHELXL2017* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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supporting information

Acta Cryst. (2018). E74, 1536-1539 [https://doi.org/10.1107/S2056989018013580]

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *WinGX* (Farrugia, 2012); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Tetraaquabis(isonicotinamide- κN^1)nickel(II) fumarate

Crystal data

$[\text{Ni}(\text{C}_6\text{H}_6\text{N}_2\text{O})_2(\text{H}_2\text{O})_4](\text{C}_4\text{H}_2\text{O}_4)$

$M_r = 489.08$

Monoclinic, $P2_1/c$

$a = 9.6140$ (8) Å

$b = 9.9819$ (9) Å

$c = 11.3874$ (10) Å

$\beta = 113.157$ (7)°

$V = 1004.76$ (16) Å³

$Z = 2$

$F(000) = 508$

$D_x = 1.617$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8294 reflections

$\theta = 2.0\text{--}28.5^\circ$

$\mu = 1.03$ mm⁻¹

$T = 296$ K

Prism, blue

$0.58 \times 0.50 \times 0.39$ mm

Data collection

STOE IPDS 2

diffractometer

Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus

Detector resolution: 6.67 pixels mm⁻¹

rotation method scans

Absorption correction: integration

(X-RED32; Stoe & Cie, 2002)

$T_{\min} = 0.527$, $T_{\max} = 0.593$

5175 measured reflections

2075 independent reflections

1777 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -12 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.104$

$S = 1.05$

2075 reflections

171 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 0.0362P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.39$ e Å⁻³

$\Delta\rho_{\min} = -0.82$ 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. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.500000	0.500000	0.500000	0.02691 (15)
O2	0.39639 (19)	0.36038 (17)	0.35626 (16)	0.0352 (3)
O3	0.3976 (2)	0.65707 (16)	0.38085 (15)	0.0345 (3)
O4	0.45031 (18)	0.63766 (14)	0.16741 (14)	0.0399 (4)
O5	0.2761 (2)	0.47778 (18)	0.11437 (18)	0.0473 (4)
O1	1.0888 (2)	0.70001 (16)	0.31656 (18)	0.0506 (4)
N1	0.6822 (2)	0.52490 (16)	0.44312 (18)	0.0315 (4)
C7	0.3828 (2)	0.5393 (2)	0.10101 (19)	0.0328 (4)
C8	0.4341 (3)	0.4863 (2)	0.0015 (2)	0.0357 (5)
N2	1.0342 (3)	0.5042 (3)	0.2095 (3)	0.0605 (7)
C2	0.8968 (2)	0.5673 (2)	0.33931 (19)	0.0344 (4)
C3	0.8503 (3)	0.6699 (2)	0.3963 (2)	0.0402 (5)
H3	0.891089	0.755195	0.401464	0.048*
C4	0.7431 (3)	0.6452 (2)	0.4456 (2)	0.0392 (5)
H4	0.711583	0.715976	0.482316	0.047*
C6	0.8353 (3)	0.4416 (2)	0.3374 (2)	0.0423 (5)
H6	0.864249	0.369442	0.300562	0.051*
C1	1.0136 (3)	0.5951 (2)	0.2856 (2)	0.0402 (5)
C5	0.7303 (3)	0.4249 (2)	0.3910 (2)	0.0401 (5)
H5	0.691168	0.339653	0.390746	0.048*
H3B	0.402 (3)	0.655 (3)	0.312 (3)	0.048 (8)*
H3A	0.318 (3)	0.670 (3)	0.369 (2)	0.038 (7)*
H2B	0.434 (4)	0.297 (3)	0.354 (3)	0.051 (9)*
H2A	0.364 (4)	0.389 (3)	0.287 (3)	0.056 (9)*
H2C	1.030 (6)	0.554 (6)	0.139 (5)	0.133 (17)*
H2D	0.989 (5)	0.420 (5)	0.199 (4)	0.104 (14)*
H8	0.367 (3)	0.424 (3)	-0.058 (3)	0.053 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0289 (2)	0.0249 (2)	0.0340 (2)	0.00018 (13)	0.01994 (16)	-0.00068 (12)
O2	0.0414 (9)	0.0306 (8)	0.0386 (8)	0.0005 (7)	0.0210 (7)	-0.0031 (6)
O3	0.0366 (9)	0.0352 (8)	0.0389 (8)	0.0044 (7)	0.0226 (7)	0.0032 (6)
O4	0.0537 (10)	0.0327 (8)	0.0455 (8)	-0.0069 (7)	0.0328 (7)	-0.0049 (6)
O5	0.0479 (10)	0.0543 (10)	0.0529 (10)	-0.0138 (8)	0.0341 (8)	-0.0076 (7)
O1	0.0464 (10)	0.0421 (9)	0.0781 (12)	-0.0012 (7)	0.0405 (9)	0.0097 (8)
N1	0.0330 (9)	0.0291 (8)	0.0412 (9)	-0.0003 (7)	0.0241 (8)	0.0002 (7)

C7	0.0389 (11)	0.0291 (9)	0.0366 (9)	0.0038 (9)	0.0216 (9)	0.0031 (8)
C8	0.0425 (13)	0.0329 (11)	0.0393 (11)	-0.0024 (9)	0.0244 (10)	-0.0040 (8)
N2	0.0581 (16)	0.0683 (18)	0.0789 (18)	-0.0089 (12)	0.0526 (15)	-0.0146 (12)
C2	0.0274 (10)	0.0407 (12)	0.0397 (9)	0.0013 (9)	0.0182 (8)	0.0042 (9)
C3	0.0419 (12)	0.0314 (10)	0.0578 (12)	-0.0019 (9)	0.0309 (11)	0.0025 (9)
C4	0.0429 (12)	0.0305 (10)	0.0544 (12)	-0.0005 (9)	0.0302 (10)	-0.0024 (9)
C6	0.0422 (13)	0.0372 (12)	0.0592 (13)	-0.0031 (10)	0.0325 (11)	-0.0112 (10)
C1	0.0333 (11)	0.0469 (13)	0.0485 (11)	0.0040 (9)	0.0245 (10)	0.0086 (10)
C5	0.0424 (12)	0.0313 (11)	0.0584 (12)	-0.0042 (9)	0.0325 (11)	-0.0069 (9)

Geometric parameters (Å, °)

Ni1—O3 ⁱ	2.0536 (15)	C7—C8	1.499 (3)
Ni1—O3	2.0537 (16)	C8—C8 ⁱⁱ	1.309 (5)
Ni1—O2	2.0812 (15)	C8—H8	0.95 (3)
Ni1—O2 ⁱ	2.0812 (15)	N2—C1	1.322 (4)
Ni1—N1	2.1075 (18)	N2—H2C	0.93 (6)
Ni1—N1 ⁱ	2.1075 (18)	N2—H2D	0.93 (5)
O2—H2B	0.73 (3)	C2—C3	1.378 (3)
O2—H2A	0.78 (3)	C2—C6	1.384 (3)
O3—H3B	0.80 (3)	C2—C1	1.501 (3)
O3—H3A	0.73 (3)	C3—C4	1.376 (3)
O4—C7	1.253 (3)	C3—H3	0.9300
O5—C7	1.255 (3)	C4—H4	0.9300
O1—C1	1.242 (3)	C6—C5	1.379 (3)
N1—C4	1.332 (3)	C6—H6	0.9300
N1—C5	1.334 (3)	C5—H5	0.9300
O3 ⁱ —Ni1—O3	180.0	O5—C7—C8	116.5 (2)
O3 ⁱ —Ni1—O2	88.00 (7)	C8 ⁱⁱ —C8—C7	123.7 (3)
O3—Ni1—O2	92.00 (7)	C8 ⁱⁱ —C8—H8	120.6 (17)
O3 ⁱ —Ni1—O2 ⁱ	92.00 (7)	C7—C8—H8	115.6 (16)
O3—Ni1—O2 ⁱ	88.00 (7)	C1—N2—H2C	104 (4)
O2—Ni1—O2 ⁱ	180.0	C1—N2—H2D	121 (3)
O3 ⁱ —Ni1—N1	93.03 (7)	H2C—N2—H2D	121 (4)
O3—Ni1—N1	86.97 (7)	C3—C2—C6	117.72 (19)
O2—Ni1—N1	92.05 (6)	C3—C2—C1	119.23 (19)
O2 ⁱ —Ni1—N1	87.95 (7)	C6—C2—C1	123.0 (2)
O3 ⁱ —Ni1—N1 ⁱ	86.97 (7)	C4—C3—C2	119.6 (2)
O3—Ni1—N1 ⁱ	93.03 (7)	C4—C3—H3	120.2
O2—Ni1—N1 ⁱ	87.95 (7)	C2—C3—H3	120.2
O2 ⁱ —Ni1—N1 ⁱ	92.05 (6)	N1—C4—C3	123.1 (2)
N1—Ni1—N1 ⁱ	180.0	N1—C4—H4	118.4
Ni1—O2—H2B	121 (2)	C3—C4—H4	118.4
Ni1—O2—H2A	115 (2)	C5—C6—C2	119.0 (2)
H2B—O2—H2A	107 (3)	C5—C6—H6	120.5
Ni1—O3—H3B	116 (2)	C2—C6—H6	120.5
Ni1—O3—H3A	117 (2)	O1—C1—N2	122.9 (2)

H3B—O3—H3A	106 (3)	O1—C1—C2	119.0 (2)
C4—N1—C5	117.25 (18)	N2—C1—C2	118.0 (2)
C4—N1—Ni1	120.76 (14)	N1—C5—C6	123.3 (2)
C5—N1—Ni1	121.62 (14)	N1—C5—H5	118.4
O4—C7—O5	124.33 (18)	C6—C5—H5	118.4
O4—C7—C8	119.12 (18)		
O4—C7—C8—C8 ⁱⁱ	17.1 (4)	C1—C2—C6—C5	178.9 (2)
O5—C7—C8—C8 ⁱⁱ	-160.8 (3)	C3—C2—C1—O1	15.0 (3)
C6—C2—C3—C4	-1.7 (3)	C6—C2—C1—O1	-163.2 (2)
C1—C2—C3—C4	179.9 (2)	C3—C2—C1—N2	-166.6 (3)
C5—N1—C4—C3	0.6 (4)	C6—C2—C1—N2	15.1 (4)
Ni1—N1—C4—C3	-172.50 (19)	C4—N1—C5—C6	-1.9 (4)
C2—C3—C4—N1	1.2 (4)	Ni1—N1—C5—C6	171.21 (19)
C3—C2—C6—C5	0.6 (3)	C2—C6—C5—N1	1.3 (4)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+1, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3B \cdots O4	0.80 (3)	1.89 (3)	2.678 (2)	169 (3)
O2—H2A \cdots O5	0.78 (3)	2.01 (3)	2.791 (3)	175 (3)
C6—H6 \cdots O1 ⁱⁱⁱ	0.93	2.31	3.230 (3)	172
N2—H2D \cdots O1 ⁱⁱⁱ	0.93 (5)	2.31 (5)	3.230 (3)	172 (4)
C5—H5 \cdots O4 ^{iv}	0.93	2.38	3.282 (3)	165
O2—H2B \cdots O4 ^{iv}	0.73 (3)	2.02 (3)	2.739 (2)	172 (3)
O3—H3A \cdots O1 ^v	0.73 (3)	2.07 (3)	2.798 (3)	175 (3)

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