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Crystal structure and Hirshfeld surface analysis of (succinato-κO)[*N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine-$\kappa^5 O$,*N*,*N*′,*O*′,*O*″]nickel(II) tetrahydrate

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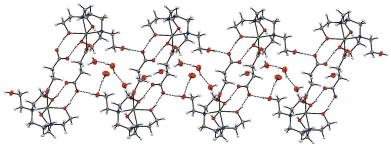
In the title compound, $[Ni(C_{10}H_{24}N_2O_4)(C_4H_4O_4)]\cdot 4H_2O$, the Ni^{II} cation is octahedrally coordinated by one O atom of the succinate anion and three O atoms and two N atoms from an *N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine molecule. In the crystal, molecules are linked by O—H···O and C—H···O hydrogen bonds, forming a three-dimensional supramolecular architecture. Hirshfeld surface analyses and two-dimensional fingerprint plots were used to analyse the intermolecular interactions present in the crystal, indicating that the most important contributions for the crystal packing are from H···H (63.3%) and H···O/O···H (34.5%) interactions.

1. Chemical context

Aliphatic dicarboxylic acid ligands have been utilized consistently in the synthesis of a diverse range of metal complexes. The metal-ion geometries of coordination compounds can easily be identified. Transition metal atoms can be bridged by aliphatic or aromatic dicarboxylate ligands to produce chains, layers and frameworks (Pavlishchuk *et al.*, 2011; Cheng *et al.*, 2013; Şen *et al.*, 2017). In addition, many transition and heavy metal cations play an important role in biological processes in the formation of many vitamins and drug components. An important element for biological systems is nickel. Nickel complexes have biological applications as a result of their antiepileptic, antimicrobial, antibacterial and anticancer activities (Bombicz *et al.*, 2001). Nickel complexes with succinic acid [chemical formula $(CH_2)_2(CO_2H)_2$] are examples containing a dicarboxylic acid. The carboxyl O atoms ligate to transition metals and thus the succinic acid can bridge between nickel metal centres to form one-, two- and three-dimensional structures as polymeric chains, layers and frameworks, respectively. We describe herein the synthesis and structural features of a new Ni^{II} complex, namely (succinato-κO)[*N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine-$\kappa^5 O$,*N*,*N*′,*O*′,*O*″]nickel(II) tetrahydrate. In addition, to understand the intermolecular interactions in the crystal structure, Hirshfeld surface analysis was performed.



2. Structural commentary

The molecular structure of the asymmetric unit of the title compound is illustrated in Fig. 1. The Ni^{II} ion is octahedrally

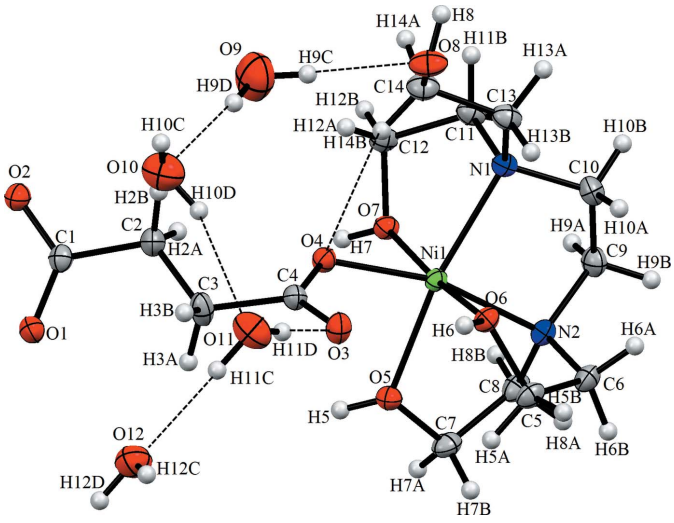
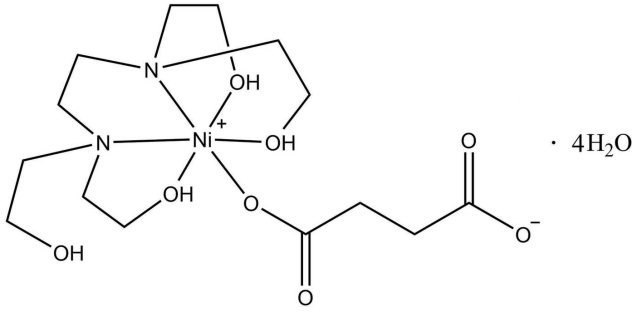


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 20% probability level.

coordinated by three O atoms and two N atoms of *N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine molecule and one O atom of the succinate anion. The Ni1—O4, Ni1—O5 and Ni1—N1 bond lengths are 2.0172 (16), 2.114 (2) and 2.145 (2) Å, respectively (Table 1). The C—O bond lengths in the deprotonated carboxylic groups differ noticeably [C1—O1 = 1.250 (3) Å and C4—O4 = 1.263 (3) Å], which is typical for monodentately coordinated carboxylates (Gumienna-Kontecka *et al.*, 2007; Pavlishchuk *et al.*, 2010; Penkova *et al.*, 2010). In the same way, the C5—O6, C7—O5 and C12—O7 bonds [1.431 (3), 1.440 (3) and 1.434 (3) Å, respectively] show single-bond character. The C10—N1 and C11—N1 bond lengths are similar [1.490 (3) and 1.497 (3) Å, respectively], while the C6—N2 and C9—N2 bonds are also not significantly different [1.500 (3) and 1.484 (4) Å, respectively]. An intramolecular C14—H14*B*···O4 hydrogen bond occurs while the complex molecule and water molecules are linked by O—H···O hydrogen bonds (O9—H9*C*···O8, O9—H9*D*···O10, O10—H10*D*···O11, O11—H11*C*···O12, O11—H11*D*···O3; Fig. 1 and Table 2).



3. Supramolecular features

The crystal packing of the title compound (Fig. 2) features intermolecular hydrogen bonds (O5—H5···O2^{i}, O7—H7···O1^{i}, O8—H8···O2^{ii}, O10—H10*C*···O11^{iii}, O12—H12*C*···O1^{iv} and C6—H6*A*···O10^{v}; symmetry codes as in Table 2), which connect the molecules into a three-dimensional supramolecular architecture. All four O atoms of the water molecules are involved in intra or intermolecular hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

Ni1—O4	2.0172 (16)	Ni1—O5	2.114 (2)
Ni1—O6	2.0622 (18)	Ni1—N1	2.145 (2)
Ni1—N2	2.069 (2)	O4—C4	1.263 (3)
Ni1—O7	2.0768 (17)	O1—C1	1.250 (3)
O4—Ni1—N2	165.11 (9)	O4—Ni1—N1	108.35 (8)
O6—Ni1—O7	174.18 (7)	N2—Ni1—N1	85.82 (9)
O6—Ni1—O5	95.59 (8)	O5—Ni1—N1	162.10 (8)
Ni1—O4—C4—O3	29.5 (4)	Ni1—N1—C10—C9	36.8 (3)
Ni1—O4—C4—C3	−147.4 (2)	Ni1—O7—C12—C11	56.1 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2^{i}	0.86	1.76	2.585 (3)	158
O6—H6···O3	0.87	2.03	2.581 (2)	121
O7—H7···O1^{i}	0.87	1.80	2.603 (3)	152
O8—H8···O2^{ii}	0.82	1.87	2.687 (3)	175
O9—H9*C*···O8	0.85	1.98	2.803 (4)	162
O9—H9*D*···O10	0.85	1.94	2.767 (6)	164
O10—H10*C*···O11^{iii}	0.85	2.09	2.892 (5)	156
O10—H10*D*···O11	0.85	2.10	2.913 (5)	160
O11—H11*C*···O12	0.85	1.99	2.836 (4)	178
O11—H11*D*···O3	0.85	2.02	2.865 (4)	172
O12—H12*C*···O1^{iv}	0.82 (4)	2.38 (5)	2.915 (4)	124 (5)
C6—H6*A*···O10^{v}	0.97	2.57	3.458 (5)	152
C14—H14*B*···O4	0.97	2.39	3.313 (4)	158

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

4. Database survey

A search of the Cambridge Structural database (CSD, version 5.39, update May 2018; Groom *et al.*, 2016) revealed that there are several precendents for *catena*-{[[*N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine-κ^2N^1,N^2]nickel(II)]-μ-succinato-κO^4} tetrahydrate, including the structures of hexaaquanickel(II) bis{aqua[*N*-(2-{bis[(carboxy)methyl]amino}ethyl)-glycinato]nickel(II)} dihydrate (NELMUO; Belošević *et al.*, 2013), hexaaquanickel(II) (μ^2-triethylenetetra-aminehexa-

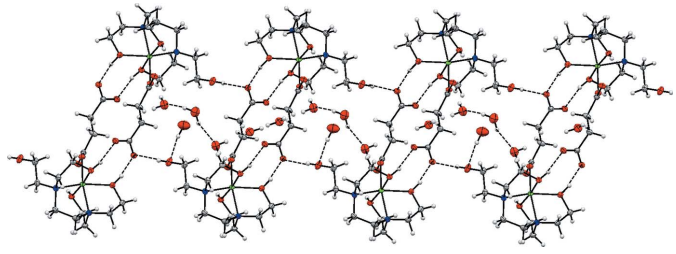


Figure 2
A view of the crystal packing of the title compound along the *c* axis. Dashed lines denote the intramolecular and intermolecular hydrogen bonds.

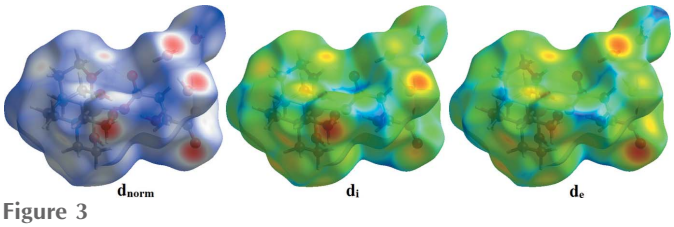


Figure 3
The Hirshfeld surfaces of the title compound mapped over d_{norm}, d_i and d_e.

acetato)diaquadinickel(II) dihydrate (UCAWEB; Shi *et al.*, 2006) and sodium aqua{hydrogen 2,2′,2″,2‴-[ethane-1,2-diylbis(nitrilo)]tetraacetato}nickel(II) trihydrate (WAPHAY; Crouse *et al.*, 2012). In addition, tetraaquabis(isonicotinamide-κN^1)nickel(II) bis(4-formylbenzoate) dihydrate (HUCLAT; Hökelek *et al.*, 2009), *trans*-tetraaquabis(isonicotinamide)-nickel(II) bis(3-hydroxybenzoate) tetrahydrate (GANZAY; Zaman *et al.*, 2012) and tetraaquabis(isonicotinamide)-nickel(II) thiophene-2,5-dicarboxylate dihydrate (NETQIO; Liu *et al.*, 2012) have been reported. In these three complexes, the Ni—N bond lengths vary from 1.999 to 2.118 Å. In the title complex, the Ni—N bond lengths [2.145 (2) and 2.069 (2) Å] fall within these limits.

5. Hirshfeld surface analysis

Hirshfeld surface analysis was used to investigate the presence of hydrogen bonds and intermolecular interactions in the crystal structure and two-dimensional fingerprint plots were calculated using *CrystalExplorer* (Turner *et al.*, 2017). The molecular Hirshfeld surfaces were performed using a standard (high) surface resolution with the three-dimensional d_{norm} surfaces mapped over a fixed colour scale of −0.7407 (red) to 1.6068 (blue) a.u. The red spots on the surface indicate the intermolecular contacts involved in the hydrogen bonds. The red spots identified in Figs. 3 and 4 correspond to the near-type H···O contacts resulting from O—H···O and C—H···O hydrogen bonds (Table 2).

Fig. 5 shows the two-dimensional fingerprint plot for the sum of the contacts contributing to the Hirshfeld surface represented in normal mode. The graph shown in Fig. 6 represents the O···H/H···O contacts (34.5%) between the oxygen atoms inside the surface and the hydrogen atoms outside the surface, $d_e + d_i$ = 1.7 Å, and has two symmetrical points at the top, bottom left and right. These data are characteristic of O—H···O and C—H···O hydrogen bonds (Table 2). The top plot shown in Fig. 6 shows the two-dimensional fingerprint of the (d_i, d_e) points associated with hydrogen atoms. It is characterized by an end point that points to the origin and corresponds to $d_i = d_e$ = 1.0 Å, which indicates the presence of the H···H contacts (63.3% contribution). The graph for C···H/H···C represents the contacts ((1.4% contribution) between the carbon atoms inside the Hirshfeld surface and the hydrogen atoms outside it and *vice versa*. It has two symmetrical wings on the left and right sides.

In the view of the three-dimensional Hirshfeld surface of the title compound plotted over electrostatic potential energy

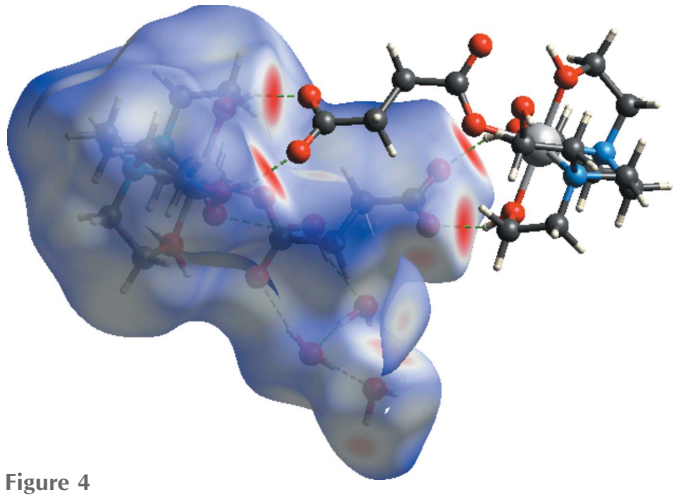


Figure 4
Hirshfeld surface mapped over d_{norm} to visualize the intermolecular interactions.

in the range −0.308 to 0.257 a.u. using the STO-3G basis set at the Hartree–Fock level of theory, Fig. 7, the C—H···O and O—H···O hydrogen-bond donors and acceptors are shown as blue and red areas around the atoms with positive (hydrogen-bond donors) and negative (hydrogen-bond acceptors) electrostatic potentials, respectively.

6. Synthesis and crystallization

A solution of NaOH (50 mmol, 2.0 g) was added to an aqueous solution of succinic acid (25 mmol, 3 g) under stirring. A solution of $NiCl_2·6H_2O$ (25 mmol, 6.14 g) in methanol was added. The mixture was heated at 353 K for one h and then the blue mixture was filtered and left to dry at room temperature. The product (0.88 mmol, 0.20 g) was dissolved in ethanol and added to a ethanol solution of *N*,*N*,*N*′,*N*′-tetra-

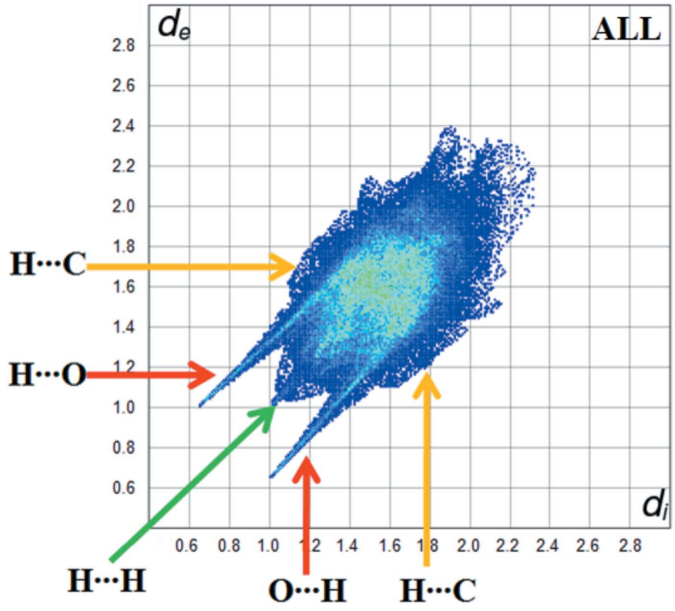


Figure 5
The fingerprint plot for all interactions.

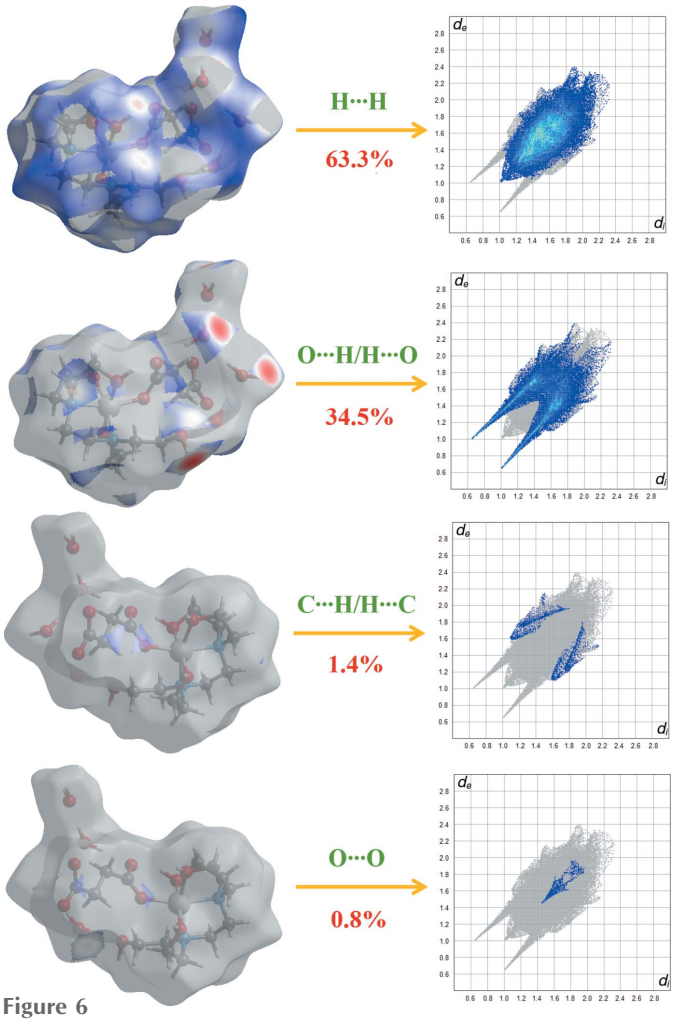


Figure 6
Two-dimensional fingerprint plots with a d_{norm} view of the H···H (63.3%), O···H/H···O (34.5%), C···H/H···C (1.4%) and O···O (0.8%) contacts in the title compound.

kis(2-hydroxyethyl)ethylenediamine (1.75 mmol, 0.41 g). The mixture was heated at 353 K for one h under stirring and the resulting suspension was filtered. It was allowed to crystallize for four weeks at room temperature. Blue prismatic crystals suitable for X-ray diffraction analysis were obtained.

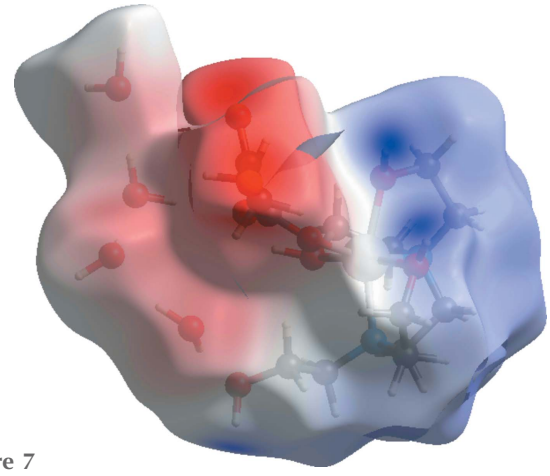


Figure 7
Hirshfeld surface plotted over electrostatic potential energy.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_{10}H_{24}N_2O_4)(C_4H_4O_4)]\cdot 4H_2O$
M_r	483.16
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.1369 (6), 10.8182 (5), 19.7771 (12)
β (°)	90.172 (5)
V (Å^3)	2168.8 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.96
Crystal size (mm)	0.64 × 0.53 × 0.42
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.605, 0.735
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11333, 4472, 3581
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.113, 1.06
No. of reflections	4472
No. of parameters	283
No. of restraints	14
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.53, −0.43

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2002), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were geometrically positioned with C—H distances of 0.93–0.97 Å. and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C). N-bound H atoms were located in difference-Fourier maps and refined isotropically. The water H atoms were located in a difference map and were refined subject to a DFIX restraint of O—H = 0.85 Å. The O12—H12*C* bond length was refined with a DFIX restraint of 0.84 (4) Å. The H atoms bonded to other O atoms (O5, O6, O7 and O8) were located in a difference map and refined freely.

Acknowledgements

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supporting information

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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: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

(Succinato-κO)[*N*,*N*,*N*′,*N*′-tetrakis(2-hydroxyethyl)ethylenediamine-$\kappa^5 O,N,N',O',O''$]nickel(II) tetrahydrate

Crystal data

$[Ni(C_{10}H_{24}N_2O_4)(C_4H_4O_4)]\cdot 4H_2O$
$M_r = 483.16$
Monoclinic, $P2_1/c$
$a = 10.1369$ (6) Å
$b = 10.8182$ (5) Å
$c = 19.7771$ (12) Å
$\beta = 90.172$ (5)°
$V = 2168.8$ (2) Å^3
$Z = 4$
$F(000) = 1032$
$D_x = 1.480$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 18670 reflections
$\theta = 1.9$–27.7°
$\mu = 0.96$ mm^{-1}
$T = 296$ K
Prism, blue
0.64 × 0.53 × 0.42 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)
$T_{min} = 0.605$, $T_{max} = 0.735$
11333 measured reflections
4472 independent reflections
3581 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.1°$
$h = -11\rightarrow12$
$k = -13\rightarrow13$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.113$
$S = 1.06$
4472 reflections
283 parameters
14 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.0617P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.78305 (3)	0.48644 (3)	0.27834 (2)	0.03216 (11)
O6	0.71228 (19)	0.64974 (17)	0.23851 (8)	0.0399 (4)
O7	0.83482 (18)	0.31821 (16)	0.32148 (8)	0.0389 (4)
O4	0.7758 (2)	0.55541 (16)	0.37302 (8)	0.0402 (4)
O5	0.98373 (19)	0.53962 (19)	0.27746 (9)	0.0441 (4)
O1	1.0038 (2)	0.6832 (2)	0.57591 (9)	0.0506 (5)
O3	0.7525 (2)	0.75762 (18)	0.35312 (9)	0.0492 (5)
O2	0.8782 (2)	0.5373 (2)	0.62081 (10)	0.0505 (5)
O8	0.3602 (2)	0.5662 (2)	0.36159 (13)	0.0616 (6)
H8	0.289310	0.530698	0.365865	0.092*
N1	0.6030 (2)	0.3883 (2)	0.25904 (10)	0.0386 (5)
N2	0.8286 (2)	0.4497 (2)	0.17844 (10)	0.0399 (5)
O11	0.6745 (3)	0.9685 (3)	0.43095 (17)	0.0797 (8)
H11C	0.743874	1.002983	0.446171	0.120*
H11D	0.695813	0.910368	0.404223	0.120*
C1	0.9109 (3)	0.6073 (2)	0.57295 (12)	0.0406 (6)
C4	0.7866 (3)	0.6676 (2)	0.38981 (12)	0.0394 (6)
O12	0.9026 (3)	1.0865 (3)	0.48350 (16)	0.0815 (8)
C11	0.6166 (3)	0.2652 (2)	0.29282 (14)	0.0443 (6)
H11A	0.644685	0.204241	0.259896	0.053*
H11B	0.531595	0.239664	0.310360	0.053*
C10	0.6021 (3)	0.3719 (3)	0.18425 (14)	0.0497 (7)
H10A	0.566366	0.445588	0.163106	0.060*
H10B	0.545229	0.302940	0.172542	0.060*
C12	0.7148 (3)	0.2705 (2)	0.34949 (14)	0.0440 (6)
H12A	0.683244	0.324126	0.385255	0.053*
H12B	0.729239	0.188620	0.368117	0.053*
C6	0.8042 (3)	0.5651 (3)	0.13822 (13)	0.0471 (7)
H6A	0.722563	0.556087	0.113029	0.057*
H6B	0.875128	0.575787	0.105906	0.057*
C5	0.7955 (3)	0.6798 (3)	0.18258 (13)	0.0466 (6)
H5A	0.882565	0.703352	0.198508	0.056*
H5B	0.758527	0.748288	0.157150	0.056*
C9	0.7392 (3)	0.3480 (3)	0.15738 (14)	0.0503 (7)
H9A	0.771937	0.269966	0.174776	0.060*
H9B	0.736540	0.342881	0.108427	0.060*
O10	0.5320 (4)	0.8538 (4)	0.54221 (19)	0.1024 (10)
H10C	0.461361	0.887695	0.555905	0.154*
H10D	0.555241	0.886225	0.504954	0.154*

C8	0.9674 (3)	0.4115 (3)	0.17774 (13)	0.0481 (7)
H8A	0.998072	0.406052	0.131423	0.058*
H8B	0.975995	0.330382	0.198239	0.058*
C13	0.4778 (3)	0.4508 (3)	0.27750 (14)	0.0471 (6)
H13A	0.404622	0.399473	0.262823	0.057*
H13B	0.472178	0.528317	0.253025	0.057*
C3	0.8508 (4)	0.6932 (3)	0.45697 (14)	0.0549 (8)
H3A	0.944899	0.702764	0.450027	0.066*
H3B	0.817365	0.771168	0.474064	0.066*
C14	0.4626 (3)	0.4768 (3)	0.35170 (17)	0.0541 (7)
H14A	0.440785	0.401067	0.375403	0.065*
H14B	0.545096	0.508051	0.369923	0.065*
C2	0.8295 (3)	0.5965 (3)	0.50927 (13)	0.0526 (7)
H2A	0.847406	0.516578	0.489003	0.063*
H2B	0.737092	0.597494	0.521824	0.063*
C7	1.0510 (3)	0.5040 (3)	0.21648 (15)	0.0497 (7)
H7A	1.135643	0.467410	0.227709	0.060*
H7B	1.066719	0.576359	0.188659	0.060*
O9	0.4560 (5)	0.6270 (4)	0.49044 (17)	0.1286 (14)
H6	0.691139	0.719062	0.257744	0.193*
H7	0.897531	0.295947	0.348879	0.193*
H5	1.041734	0.531317	0.309305	0.193*
H9C	0.418851	0.624575	0.451870	0.193*
H9D	0.466146	0.701837	0.502467	0.193*
H12C	0.872 (6)	1.154 (3)	0.474 (3)	0.16 (3)*
H12D	0.980 (4)	1.105 (5)	0.497 (5)	0.31 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.03494 (19)	0.03014 (17)	0.03141 (16)	−0.00050 (13)	0.00031 (11)	0.00160 (11)
O6	0.0440 (11)	0.0373 (9)	0.0383 (8)	0.0033 (8)	0.0026 (8)	0.0053 (7)
O7	0.0389 (10)	0.0347 (9)	0.0430 (9)	−0.0009 (8)	−0.0022 (7)	0.0043 (7)
O4	0.0511 (12)	0.0348 (9)	0.0347 (8)	−0.0036 (8)	0.0008 (8)	−0.0006 (7)
O5	0.0371 (10)	0.0524 (11)	0.0426 (9)	−0.0040 (9)	0.0011 (8)	0.0039 (8)
O1	0.0493 (12)	0.0560 (12)	0.0465 (10)	−0.0098 (10)	−0.0097 (9)	0.0051 (9)
O3	0.0676 (14)	0.0370 (10)	0.0430 (10)	0.0029 (10)	−0.0090 (9)	0.0004 (7)
O2	0.0459 (12)	0.0616 (12)	0.0440 (10)	−0.0027 (10)	−0.0038 (9)	0.0104 (9)
O8	0.0498 (13)	0.0501 (12)	0.0850 (15)	0.0042 (11)	0.0207 (12)	0.0023 (11)
N1	0.0355 (12)	0.0367 (11)	0.0436 (11)	−0.0007 (9)	−0.0011 (9)	0.0018 (9)
N2	0.0431 (13)	0.0413 (12)	0.0353 (10)	0.0015 (10)	0.0025 (9)	0.0001 (8)
O11	0.078 (2)	0.0564 (15)	0.105 (2)	0.0042 (14)	−0.0075 (16)	−0.0150 (14)
C1	0.0422 (15)	0.0420 (13)	0.0377 (12)	0.0054 (12)	−0.0007 (11)	−0.0029 (10)
C4	0.0412 (15)	0.0406 (13)	0.0365 (12)	−0.0024 (12)	0.0000 (10)	−0.0012 (10)
O12	0.084 (2)	0.0730 (19)	0.0877 (19)	−0.0127 (17)	−0.0047 (16)	0.0158 (15)
C11	0.0403 (15)	0.0357 (13)	0.0569 (15)	−0.0048 (12)	−0.0007 (12)	0.0024 (11)
C10	0.0470 (17)	0.0567 (17)	0.0454 (14)	−0.0053 (14)	−0.0102 (12)	−0.0041 (12)
C12	0.0466 (17)	0.0352 (13)	0.0502 (14)	−0.0007 (12)	0.0006 (12)	0.0089 (10)

C6	0.0526 (18)	0.0538 (16)	0.0349 (12)	0.0035 (14)	0.0035 (12)	0.0078 (11)
C5	0.0525 (17)	0.0430 (14)	0.0443 (13)	0.0039 (13)	0.0047 (12)	0.0128 (11)
C9	0.0567 (19)	0.0506 (16)	0.0435 (14)	−0.0023 (14)	−0.0016 (13)	−0.0111 (12)
O10	0.092 (3)	0.099 (3)	0.116 (3)	−0.004 (2)	0.0061 (19)	−0.003 (2)
C8	0.0487 (17)	0.0528 (16)	0.0428 (13)	0.0102 (14)	0.0098 (12)	0.0016 (12)
C13	0.0362 (15)	0.0455 (15)	0.0595 (16)	0.0018 (12)	−0.0024 (12)	0.0058 (12)
C3	0.078 (2)	0.0388 (14)	0.0481 (15)	−0.0040 (15)	−0.0195 (15)	−0.0021 (11)
C14	0.0411 (16)	0.0563 (17)	0.0649 (18)	0.0046 (14)	0.0074 (14)	0.0001 (14)
C2	0.0541 (19)	0.0646 (19)	0.0392 (13)	−0.0101 (15)	−0.0058 (12)	0.0018 (12)
C7	0.0404 (15)	0.0591 (18)	0.0498 (15)	0.0021 (14)	0.0105 (12)	0.0077 (13)
O9	0.180 (4)	0.106 (3)	0.100 (2)	−0.014 (3)	−0.013 (2)	−0.017 (2)

Geometric parameters (Å, º)

Ni1—O4	2.0172 (16)	C11—H11B	0.9700
Ni1—O6	2.0622 (18)	C10—C9	1.512 (4)
Ni1—N2	2.069 (2)	C10—H10A	0.9700
Ni1—O7	2.0768 (17)	C10—H10B	0.9700
Ni1—O5	2.114 (2)	C12—H12A	0.9700
Ni1—N1	2.145 (2)	C12—H12B	0.9700
O6—C5	1.431 (3)	C6—C5	1.522 (4)
O6—H6	0.8680	C6—H6A	0.9700
O7—C12	1.434 (3)	C6—H6B	0.9700
O7—H7	0.8681	C5—H5A	0.9700
O4—C4	1.263 (3)	C5—H5B	0.9700
O5—C7	1.440 (3)	C9—H9A	0.9700
O5—H5	0.8650	C9—H9B	0.9700
O1—C1	1.250 (3)	O10—H10C	0.8500
O3—C4	1.262 (3)	O10—H10D	0.8501
O2—C1	1.257 (3)	C8—C7	1.518 (4)
O8—C14	1.433 (4)	C8—H8A	0.9700
O8—H8	0.8200	C8—H8B	0.9700
N1—C13	1.485 (4)	C13—C14	1.502 (4)
N1—C10	1.490 (3)	C13—H13A	0.9700
N1—C11	1.497 (3)	C13—H13B	0.9700
N2—C8	1.467 (4)	C3—C2	1.488 (4)
N2—C9	1.484 (4)	C3—H3A	0.9700
N2—C6	1.500 (3)	C3—H3B	0.9700
O11—H11C	0.8500	C14—H14A	0.9700
O11—H11D	0.8500	C14—H14B	0.9700
C1—C2	1.508 (4)	C2—H2A	0.9700
C4—C3	1.503 (4)	C2—H2B	0.9700
O12—H12C	0.816 (10)	C7—H7A	0.9700
O12—H12D	0.851 (9)	C7—H7B	0.9700
C11—C12	1.498 (4)	O9—H9C	0.8500
C11—H11A	0.9700	O9—H9D	0.8500
O4—Ni1—O6	91.38 (7)	C11—C12—H12A	110.4

O4—Ni1—N2	165.11 (9)	O7—C12—H12B	110.4
O6—Ni1—N2	83.00 (8)	C11—C12—H12B	110.4
O4—Ni1—O7	87.29 (7)	H12A—C12—H12B	108.6
O6—Ni1—O7	174.18 (7)	N2—C6—C5	112.5 (2)
N2—Ni1—O7	99.62 (8)	N2—C6—H6A	109.1
O4—Ni1—O5	86.84 (8)	C5—C6—H6A	109.1
O6—Ni1—O5	95.59 (8)	N2—C6—H6B	109.1
N2—Ni1—O5	80.03 (8)	C5—C6—H6B	109.1
O7—Ni1—O5	90.00 (7)	H6A—C6—H6B	107.8
O4—Ni1—N1	108.35 (8)	O6—C5—C6	107.2 (2)
O6—Ni1—N1	93.50 (8)	O6—C5—H5A	110.3
N2—Ni1—N1	85.82 (9)	C6—C5—H5A	110.3
O7—Ni1—N1	81.56 (8)	O6—C5—H5B	110.3
O5—Ni1—N1	162.10 (8)	C6—C5—H5B	110.3
C5—O6—Ni1	106.55 (15)	H5A—C5—H5B	108.5
C5—O6—H6	106.8	N2—C9—C10	109.6 (2)
Ni1—O6—H6	131.2	N2—C9—H9A	109.7
C12—O7—Ni1	105.11 (15)	C10—C9—H9A	109.7
C12—O7—H7	106.2	N2—C9—H9B	109.7
Ni1—O7—H7	133.1	C10—C9—H9B	109.7
C4—O4—Ni1	126.59 (16)	H9A—C9—H9B	108.2
C7—O5—Ni1	113.10 (17)	H10C—O10—H10D	109.5
C7—O5—H5	105.0	N2—C8—C7	110.1 (2)
Ni1—O5—H5	128.2	N2—C8—H8A	109.6
C14—O8—H8	109.5	C7—C8—H8A	109.6
C13—N1—C10	107.2 (2)	N2—C8—H8B	109.6
C13—N1—C11	111.9 (2)	C7—C8—H8B	109.6
C10—N1—C11	109.7 (2)	H8A—C8—H8B	108.2
C13—N1—Ni1	117.32 (17)	N1—C13—C14	114.5 (2)
C10—N1—Ni1	103.81 (17)	N1—C13—H13A	108.6
C11—N1—Ni1	106.49 (16)	C14—C13—H13A	108.6
C8—N2—C9	111.9 (2)	N1—C13—H13B	108.6
C8—N2—C6	112.7 (2)	C14—C13—H13B	108.6
C9—N2—C6	111.6 (2)	H13A—C13—H13B	107.6
C8—N2—Ni1	106.26 (15)	C2—C3—C4	114.9 (2)
C9—N2—Ni1	105.82 (16)	C2—C3—H3A	108.5
C6—N2—Ni1	108.03 (16)	C4—C3—H3A	108.5
H11C—O11—H11D	109.5	C2—C3—H3B	108.5
O1—C1—O2	124.1 (2)	C4—C3—H3B	108.5
O1—C1—C2	120.0 (2)	H3A—C3—H3B	107.5
O2—C1—C2	116.0 (3)	O8—C14—C13	109.6 (3)
O3—C4—O4	124.6 (2)	O8—C14—H14A	109.7
O3—C4—C3	118.8 (2)	C13—C14—H14A	109.7
O4—C4—C3	116.5 (2)	O8—C14—H14B	109.7
H12C—O12—H12D	101.8 (15)	C13—C14—H14B	109.7
N1—C11—C12	111.1 (2)	H14A—C14—H14B	108.2
N1—C11—H11A	109.4	C3—C2—C1	116.5 (3)
C12—C11—H11A	109.4	C3—C2—H2A	108.2

N1—C11—H11B	109.4	C1—C2—H2A	108.2
C12—C11—H11B	109.4	C3—C2—H2B	108.2
H11A—C11—H11B	108.0	C1—C2—H2B	108.2
N1—C10—C9	111.5 (2)	H2A—C2—H2B	107.3
N1—C10—H10A	109.3	O5—C7—C8	109.5 (2)
C9—C10—H10A	109.3	O5—C7—H7A	109.8
N1—C10—H10B	109.3	C8—C7—H7A	109.8
C9—C10—H10B	109.3	O5—C7—H7B	109.8
H10A—C10—H10B	108.0	C8—C7—H7B	109.8
O7—C12—C11	106.7 (2)	H7A—C7—H7B	108.2
O7—C12—H12A	110.4	H9C—O9—H9D	109.5

Ni1—O4—C4—O3	29.5 (4)	Ni1—N2—C9—C10	42.5 (3)
Ni1—O4—C4—C3	−147.4 (2)	N1—C10—C9—N2	−55.8 (3)
C13—N1—C11—C12	−105.5 (3)	C9—N2—C8—C7	−165.1 (2)
C10—N1—C11—C12	135.7 (3)	C6—N2—C8—C7	68.1 (3)
Ni1—N1—C11—C12	23.9 (3)	Ni1—N2—C8—C7	−50.0 (2)
C13—N1—C10—C9	161.6 (2)	C10—N1—C13—C14	−179.1 (3)
C11—N1—C10—C9	−76.7 (3)	C11—N1—C13—C14	60.6 (3)
Ni1—N1—C10—C9	36.8 (3)	Ni1—N1—C13—C14	−62.9 (3)
Ni1—O7—C12—C11	56.1 (2)	O3—C4—C3—C2	151.7 (3)
N1—C11—C12—O7	−54.5 (3)	O4—C4—C3—C2	−31.2 (4)
C8—N2—C6—C5	−99.8 (3)	N1—C13—C14—O8	163.5 (2)
C9—N2—C6—C5	133.2 (3)	C4—C3—C2—C1	169.7 (3)
Ni1—N2—C6—C5	17.3 (3)	O1—C1—C2—C3	−10.1 (4)
Ni1—O6—C5—C6	50.5 (2)	O2—C1—C2—C3	169.4 (3)
N2—C6—C5—O6	−45.8 (3)	Ni1—O5—C7—C8	−13.7 (3)
C8—N2—C9—C10	157.8 (2)	N2—C8—C7—O5	42.3 (3)
C6—N2—C9—C10	−74.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2^{i}	0.86	1.76	2.585 (3)	158
O6—H6···O3	0.87	2.03	2.581 (2)	121
O7—H7···O1^{i}	0.87	1.80	2.603 (3)	152
O8—H8···O2^{ii}	0.82	1.87	2.687 (3)	175
O9—H9*C*···O8	0.85	1.98	2.803 (4)	162
O9—H9*D*···O10	0.85	1.94	2.767 (6)	164
O10—H10*C*···O11^{iii}	0.85	2.09	2.892 (5)	156
O10—H10*D*···O11	0.85	2.10	2.913 (5)	160
O11—H11*C*···O12	0.85	1.99	2.836 (4)	178
O11—H11*D*···O3	0.85	2.02	2.865 (4)	172
O12—H12*C*···O1^{iv}	0.82 (4)	2.38 (5)	2.915 (4)	124 (5)
C6—H6*A*···O10^{v}	0.97	2.57	3.458 (5)	152
C14—H14*B*···O4	0.97	2.39	3.313 (4)	158

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