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Bis[2-(2-hydroxybenzoylhydrazono)-propionato]nickel(II) trihydrate

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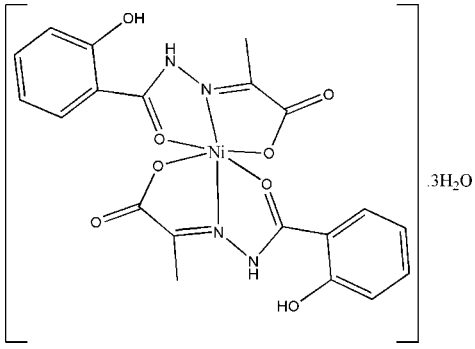
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.116; data-to-parameter ratio = 13.0.

In the title compound, $[Ni(C_{10}H_9N_2O_4)_2]\cdot 3H_2O$, the Ni^{2+} ion is octahedrally coordinated by carboxyl and acyl O atoms and azomethine N atoms of two tridentate 2-(2-hydroxybenzoylhydrazono)propionate ligands, each of which forms two five-membered chelate rings sharing the N—Ni bond. The occurrence of O—H···N and O—H···O hydrogen bonds between water molecules and ligands results in the formation of an intricate three-dimensional network which stabilizes the packing.

Related literature

For related literature, see: Buss *et al.* (2003); He *et al.* (2002); Rodriguez-Argelles *et al.* (2004).



Experimental

Crystal data

$[Ni(C_{10}H_9N_2O_4)_2]\cdot 3H_2O$
M_r = 555.14
Triclinic, $P\overline{1}$
a = 9.3787 (12) Å
b = 10.7935 (14) Å
c = 11.8795 (15) Å
α = 86.447 (2)°
β = 81.805 (2)°
γ = 79.847 (2)°
V = 1170.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.90 mm^{-1}
T = 298 (2) K
0.32 × 0.27 × 0.14 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.762, T_{max} = 0.883
5974 measured reflections
4271 independent reflections
3173 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.116
S = 1.03
4271 reflections
329 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O5^{i}	0.82	1.81	2.606 (3)	162
O8—H8···O1^{ii}	0.82	1.77	2.584 (3)	170
N2—H2···O3	0.86	2.02	2.622 (3)	126
N2—H2···O11^{iii}	0.86	2.18	2.826 (4)	132
N4—H4···O8	0.86	1.91	2.571 (3)	133
O9—H9*A*···O10	0.87	1.87	2.741 (6)	178
O9—H9*B*···O6^{iv}	0.86	1.98	2.838 (4)	171
O10—H10*A*···O6^{v}	0.86	2.05	2.882 (5)	163
O10—H10*B*···O4	0.86	2.16	2.978 (5)	160
O11—H11*A*···O9	0.86	1.88	2.741 (4)	173
O11—H11*B*···O2^{vi}	0.86	2.13	2.890 (4)	148

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2235).

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supplementary materials

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Bis[2-(2-hydroxybenzoylhydrazono)propionato]nickel(II) trihydrate

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Comment

Hydrazones have attracted considerable interest due to their complicated coordination behavior and pharmacological activity. Many of physiologically active hydrazone-metal complexes find application in the treatment of several diseases such as tuberculosis, tumour, cancer and so on (Rodriguez-Argelles *et al.*, 2004; Buss *et al.*, 2003). This paper reports the crystal structure of a novel Ni(II) complexe with N-(2-propionic acid)-salicyloyl hydrazone.

In complex (I), the Ni^{2+} ion is octahedrally surrounded by two tridentate N-(2-propionacid)-salicyloyl hydrazone ligands(Fig.1). The linkage of two tridentate ligands to Ni^{2+} ion is accomplished through the acyl oxygen, carboxyl oxygen and imido nitrogen, resulting in the formation of two five-membered chelate rings sharing the same edge. The atoms O1, N1, O4 and N3 are nearly coplanar and located in the equatorial plane, while two oxygen atoms of another ligand occupy the axial sites, the angle of the axial O5—Ni—O7 is 150° which deviates significantly from the linear angle of 180°. Those data indicate that the Ni atom is in distorted octahedron geometry·Comparing with the distances of C—O(1.42 Å) and C=O(1.23 Å), the bond lengths of O7—C14 and O4—C4 are 1.228Å and 1.246 Å, respectively, indicating that these bonds are double linkage and the ligand functions as a keto form.

The occurrence of O—H···N and O—H···O hydrogen bondings between water molecules and ligands results in the formation of an intricated three dimensionnal network which stabilizes the packing (Table 1).

Experimental

The ligand was prepared according to the literature(He *et al.* 2002). Pyruvic acid is biochemical reagent and all other chemicals used were of analytical grade.

The ligand H_3L(25.1 mg, 0.12 mmol))and $NiSO_4$ (11.8 mg, 0.05 mmol), were added in a mixed solvent of ethanol and acetonitrile, the mixture was heated for five hours under reflux. during the process stirring and influx were required. The resultant was then filtered to give a pure solution which was infiltrated by diethyl ether freely in a closed vessel, a weeks later some single crystals of the size suitable for X-Ray diffraction analysis.

Refinement

The water H atoms were located in a difference Fourier map and they were refined freely with a distance restraint of O—H=0.85 (1) Å. The others H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances of 0.93Å (pyridine ring), 0.86 Å (amine group), and with U_{iso}(H) 1.2U_{eq}(C).

Figures

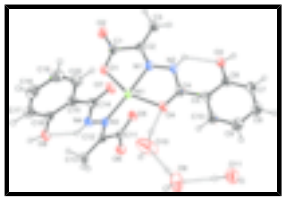


Fig. 1. The asymmetric unit of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

Bis[2-(2-hydroxybenzoylhydrazono)propionato]nickel(II) trihydrate

Crystal data

$[Ni(C_{10}H_9N_2O_4)_2]\cdot 3H_2O$	$Z = 2$
$M_r = 555.14$	$F_{000} = 576$
Triclinic, $P\overline{1}$	$D_x = 1.575$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.3787$ (12) Å	Cell parameters from 4104 reflections
$b = 10.7935$ (14) Å	$\theta = 1.9$–25.1°
$c = 11.8795$ (15) Å	$\mu = 0.90$ mm^{-1}
$\alpha = 86.447$ (2)°	$T = 298$ (2) K
$\beta = 81.805$ (2)°	Block, green
$\gamma = 79.847$ (2)°	0.32 × 0.27 × 0.14 mm
$V = 1170.8$ (3) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	4271 independent reflections
Radiation source: fine-focus sealed tube	3173 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.017$
$T = 298$(2) K	$\theta_{max} = 25.1^\circ$
φ and ω scans	$\theta_{min} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 11$
$T_{min} = 0.762$, $T_{max} = 0.883$	$k = -12 \rightarrow 12$
5974 measured reflections	$l = -14 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.9257P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.03	$(\Delta/\sigma)_{max}$ = 0.026
4271 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
329 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.03633 (5)	0.24821 (4)	0.73989 (4)	0.04088 (16)
O1	−0.1184 (3)	0.4062 (2)	0.72823 (19)	0.0515 (6)
O2	−0.2371 (3)	0.5249 (3)	0.6003 (2)	0.0682 (8)
O3	0.1561 (3)	−0.0084 (2)	0.3587 (2)	0.0526 (6)
H3	0.1593	−0.0388	0.2966	0.079*
O4	0.1846 (3)	0.0895 (2)	0.6819 (2)	0.0542 (7)
O5	−0.1161 (3)	0.1231 (2)	0.8180 (2)	0.0520 (6)
O6	−0.1891 (3)	0.0383 (2)	0.9881 (2)	0.0557 (7)
O7	0.2082 (3)	0.3690 (2)	0.75660 (19)	0.0535 (7)
O8	0.1911 (3)	0.4664 (2)	1.08922 (19)	0.0479 (6)
H8	0.1781	0.5064	1.1474	0.072*
N1	0.0160 (3)	0.2538 (2)	0.5790 (2)	0.0401 (6)
N2	0.1001 (3)	0.1580 (3)	0.5177 (2)	0.0443 (7)
H2	0.0984	0.1519	0.4461	0.053*
N3	0.0438 (3)	0.2508 (2)	0.9057 (2)	0.0385 (6)
N4	0.1266 (3)	0.3302 (2)	0.9400 (2)	0.0423 (7)
H4	0.1284	0.3409	1.0109	0.051*
C1	−0.1541 (4)	0.4325 (3)	0.6284 (3)	0.0432 (8)
C2	−0.0796 (4)	0.3390 (3)	0.5391 (3)	0.0408 (8)
C3	−0.1218 (4)	0.3499 (4)	0.4230 (3)	0.0558 (10)
H3A	−0.0469	0.3006	0.3726	0.084*
H3B	−0.1334	0.4365	0.3967	0.084*
H3C	−0.2123	0.3196	0.4244	0.084*
C4	0.1869 (4)	0.0730 (3)	0.5787 (3)	0.0418 (8)
C5	0.2799 (4)	−0.0343 (3)	0.5215 (3)	0.0405 (8)
C6	0.2656 (4)	−0.0715 (3)	0.4127 (3)	0.0431 (8)
C7	0.3629 (4)	−0.1724 (4)	0.3644 (3)	0.0558 (10)

H7	0.3541	−0.1969	0.2924	0.067*
C8	0.4727 (4)	−0.2367 (4)	0.4224 (4)	0.0656 (11)
H8A	0.5379	−0.3035	0.3886	0.079*
C9	0.4865 (5)	−0.2027 (4)	0.5299 (4)	0.0662 (11)
H9	0.5597	−0.2470	0.5692	0.079*
C10	0.3911 (4)	−0.1030 (4)	0.5781 (3)	0.0557 (10)
H10	0.4006	−0.0804	0.6506	0.067*
C11	−0.1220 (4)	0.1103 (3)	0.9248 (3)	0.0421 (8)
C12	−0.0357 (4)	0.1912 (3)	0.9795 (3)	0.0396 (8)
C13	−0.0482 (4)	0.1976 (3)	1.1052 (3)	0.0467 (9)
H13A	0.0477	0.1870	1.1276	0.070*
H13B	−0.0980	0.1320	1.1408	0.070*
H13C	−0.1024	0.2780	1.1284	0.070*
C14	0.2061 (4)	0.3916 (3)	0.8568 (3)	0.0408 (8)
C15	0.2854 (4)	0.4859 (3)	0.8933 (3)	0.0407 (8)
C16	0.2739 (3)	0.5245 (3)	1.0053 (3)	0.0384 (7)
C17	0.3467 (4)	0.6181 (3)	1.0295 (3)	0.0463 (8)
H17	0.3391	0.6428	1.1040	0.056*
C18	0.4302 (4)	0.6752 (4)	0.9443 (4)	0.0568 (10)
H18	0.4774	0.7393	0.9612	0.068*
C19	0.4446 (4)	0.6377 (4)	0.8334 (4)	0.0588 (10)
H19	0.5024	0.6755	0.7760	0.071*
C20	0.3733 (4)	0.5445 (3)	0.8085 (3)	0.0515 (9)
H20	0.3834	0.5197	0.7337	0.062*
O9	0.6479 (4)	−0.0977 (3)	0.8717 (3)	0.0988 (12)
H9A	0.5683	−0.0451	0.8631	0.148*
H9B	0.6945	−0.0611	0.9137	0.148*
O10	0.3963 (5)	0.0695 (5)	0.8494 (4)	0.155 (2)
H10A	0.3389	0.0475	0.9073	0.233*
H10B	0.3468	0.0894	0.7935	0.233*
O11	0.8003 (4)	−0.2489 (3)	0.7025 (3)	0.0849 (10)
H11A	0.7474	−0.2063	0.7572	0.127*
H11B	0.7640	−0.3156	0.6966	0.127*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0518 (3)	0.0373 (3)	0.0334 (3)	−0.00219 (19)	−0.00861 (19)	−0.00908 (18)
O1	0.0721 (17)	0.0454 (14)	0.0345 (13)	0.0031 (12)	−0.0114 (12)	−0.0091 (11)
O2	0.084 (2)	0.0558 (16)	0.0591 (17)	0.0200 (15)	−0.0234 (16)	−0.0124 (14)
O3	0.0710 (17)	0.0503 (15)	0.0379 (14)	−0.0022 (13)	−0.0172 (13)	−0.0141 (11)
O4	0.0695 (17)	0.0569 (15)	0.0348 (13)	0.0005 (13)	−0.0116 (12)	−0.0121 (12)
O5	0.0742 (17)	0.0474 (14)	0.0402 (14)	−0.0170 (13)	−0.0164 (13)	−0.0086 (11)
O6	0.0670 (17)	0.0522 (15)	0.0513 (16)	−0.0209 (13)	−0.0062 (13)	−0.0007 (13)
O7	0.0672 (17)	0.0638 (16)	0.0318 (13)	−0.0149 (13)	−0.0063 (12)	−0.0102 (12)
O8	0.0635 (16)	0.0489 (14)	0.0335 (13)	−0.0163 (12)	−0.0008 (12)	−0.0127 (11)
N1	0.0469 (16)	0.0391 (15)	0.0344 (15)	−0.0065 (13)	−0.0028 (13)	−0.0108 (12)
N2	0.0549 (18)	0.0443 (16)	0.0309 (15)	0.0018 (14)	−0.0054 (13)	−0.0100 (13)

N3	0.0439 (16)	0.0330 (14)	0.0407 (16)	−0.0057 (12)	−0.0113 (13)	−0.0084 (12)
N4	0.0534 (17)	0.0422 (15)	0.0340 (15)	−0.0108 (13)	−0.0097 (13)	−0.0066 (12)
C1	0.052 (2)	0.0374 (18)	0.041 (2)	−0.0045 (16)	−0.0120 (17)	−0.0051 (15)
C2	0.050 (2)	0.0374 (18)	0.0356 (18)	−0.0074 (15)	−0.0084 (16)	−0.0029 (14)
C3	0.068 (3)	0.058 (2)	0.041 (2)	−0.003 (2)	−0.0129 (19)	−0.0069 (18)
C4	0.047 (2)	0.0449 (19)	0.0343 (18)	−0.0084 (16)	−0.0054 (15)	−0.0088 (15)
C5	0.0432 (19)	0.0435 (18)	0.0346 (18)	−0.0076 (15)	−0.0037 (15)	−0.0033 (15)
C6	0.047 (2)	0.0422 (19)	0.0405 (19)	−0.0086 (16)	−0.0054 (16)	−0.0044 (15)
C7	0.063 (3)	0.055 (2)	0.046 (2)	−0.0040 (19)	0.0023 (19)	−0.0160 (18)
C8	0.059 (3)	0.062 (3)	0.068 (3)	0.010 (2)	0.001 (2)	−0.016 (2)
C9	0.057 (2)	0.069 (3)	0.066 (3)	0.008 (2)	−0.009 (2)	−0.007 (2)
C10	0.059 (2)	0.064 (2)	0.044 (2)	0.000 (2)	−0.0144 (19)	−0.0079 (18)
C11	0.050 (2)	0.0358 (18)	0.040 (2)	−0.0028 (16)	−0.0080 (16)	−0.0046 (15)
C12	0.0442 (19)	0.0343 (17)	0.0395 (19)	0.0014 (15)	−0.0111 (16)	−0.0048 (15)
C13	0.057 (2)	0.046 (2)	0.0378 (19)	−0.0022 (17)	−0.0145 (17)	−0.0056 (16)
C14	0.0428 (19)	0.0427 (18)	0.0353 (19)	−0.0010 (15)	−0.0063 (15)	−0.0048 (15)
C15	0.0409 (18)	0.0411 (18)	0.0384 (19)	−0.0020 (15)	−0.0051 (15)	−0.0037 (15)
C16	0.0370 (18)	0.0366 (17)	0.0390 (18)	0.0010 (14)	−0.0061 (15)	−0.0003 (14)
C17	0.044 (2)	0.0427 (19)	0.053 (2)	−0.0035 (16)	−0.0104 (17)	−0.0076 (17)
C18	0.050 (2)	0.047 (2)	0.077 (3)	−0.0114 (18)	−0.015 (2)	−0.002 (2)
C19	0.052 (2)	0.057 (2)	0.066 (3)	−0.0160 (19)	−0.001 (2)	0.010 (2)
C20	0.052 (2)	0.057 (2)	0.043 (2)	−0.0045 (18)	−0.0066 (18)	0.0032 (18)
O9	0.092 (2)	0.102 (3)	0.108 (3)	−0.033 (2)	0.002 (2)	−0.040 (2)
O10	0.111 (3)	0.260 (6)	0.082 (3)	−0.012 (4)	−0.008 (3)	0.023 (3)
O11	0.108 (3)	0.073 (2)	0.074 (2)	−0.0278 (19)	0.0084 (19)	−0.0113 (17)

Geometric parameters (Å, °)

Ni1—N1	1.943 (3)	C5—C6	1.408 (4)
Ni1—N3	1.983 (3)	C6—C7	1.387 (5)
Ni1—O1	2.046 (2)	C7—C8	1.381 (5)
Ni1—O4	2.092 (2)	C7—H7	0.9300
Ni1—O5	2.207 (3)	C8—C9	1.381 (6)
Ni1—O7	2.281 (3)	C8—H8A	0.9300
O1—C1	1.280 (4)	C9—C10	1.370 (5)
O2—C1	1.213 (4)	C9—H9	0.9300
O3—C6	1.348 (4)	C10—H10	0.9300
O3—H3	0.8200	C11—C12	1.519 (5)
O4—C4	1.247 (4)	C12—C13	1.487 (4)
O5—C11	1.262 (4)	C13—H13A	0.9600
O6—C11	1.237 (4)	C13—H13B	0.9600
O7—C14	1.226 (4)	C13—H13C	0.9600
O8—C16	1.365 (4)	C14—C15	1.480 (5)
O8—H8	0.8200	C15—C20	1.401 (5)
N1—C2	1.285 (4)	C15—C16	1.403 (4)
N1—N2	1.365 (3)	C16—C17	1.380 (5)
N2—C4	1.360 (4)	C17—C18	1.373 (5)
N2—H2	0.8600	C17—H17	0.9300
N3—C12	1.285 (4)	C18—C19	1.383 (6)

N3—N4	1.368 (4)	C18—H18	0.9300
N4—C14	1.362 (4)	C19—C20	1.370 (5)
N4—H4	0.8600	C19—H19	0.9300
C1—C2	1.518 (4)	C20—H20	0.9300
C2—C3	1.480 (5)	O9—H9A	0.8690
C3—H3A	0.9600	O9—H9B	0.8622
C3—H3B	0.9600	O10—H10A	0.8594
C3—H3C	0.9600	O10—H10B	0.8587
C4—C5	1.462 (4)	O11—H11A	0.8629
C5—C10	1.398 (5)	O11—H11B	0.8594
N1—Ni1—N3	175.96 (11)	O3—C6—C7	121.8 (3)
N1—Ni1—O1	78.86 (10)	O3—C6—C5	118.8 (3)
N3—Ni1—O1	97.27 (10)	C7—C6—C5	119.5 (3)
N1—Ni1—O4	77.80 (10)	C8—C7—C6	120.6 (4)
N3—Ni1—O4	106.07 (10)	C8—C7—H7	119.7
O1—Ni1—O4	156.66 (9)	C6—C7—H7	119.7
N1—Ni1—O5	103.83 (10)	C9—C8—C7	120.6 (4)
N3—Ni1—O5	75.31 (10)	C9—C8—H8A	119.7
O1—Ni1—O5	96.27 (10)	C7—C8—H8A	119.7
O4—Ni1—O5	89.12 (10)	C10—C9—C8	119.2 (4)
N1—Ni1—O7	106.13 (10)	C10—C9—H9	120.4
N3—Ni1—O7	74.89 (10)	C8—C9—H9	120.4
O1—Ni1—O7	90.70 (10)	C9—C10—C5	122.0 (4)
O4—Ni1—O7	95.93 (10)	C9—C10—H10	119.0
O5—Ni1—O7	150.01 (9)	C5—C10—H10	119.0
C1—O1—Ni1	114.5 (2)	O6—C11—O5	126.7 (3)
C6—O3—H3	109.5	O6—C11—C12	117.5 (3)
C4—O4—Ni1	113.0 (2)	O5—C11—C12	115.8 (3)
C11—O5—Ni1	113.5 (2)	N3—C12—C13	126.1 (3)
C14—O7—Ni1	110.0 (2)	N3—C12—C11	112.6 (3)
C16—O8—H8	109.5	C13—C12—C11	121.3 (3)
C2—N1—N2	124.4 (3)	C12—C13—H13A	109.5
C2—N1—Ni1	119.8 (2)	C12—C13—H13B	109.5
N2—N1—Ni1	115.6 (2)	H13A—C13—H13B	109.5
C4—N2—N1	114.8 (3)	C12—C13—H13C	109.5
C4—N2—H2	122.6	H13A—C13—H13C	109.5
N1—N2—H2	122.6	H13B—C13—H13C	109.5
C12—N3—N4	120.5 (3)	O7—C14—N4	120.1 (3)
C12—N3—Ni1	122.4 (2)	O7—C14—C15	122.8 (3)
N4—N3—Ni1	116.8 (2)	N4—C14—C15	117.0 (3)
C14—N4—N3	117.0 (3)	C20—C15—C16	117.9 (3)
C14—N4—H4	121.5	C20—C15—C14	117.2 (3)
N3—N4—H4	121.5	C16—C15—C14	124.8 (3)
O2—C1—O1	125.9 (3)	O8—C16—C17	121.2 (3)
O2—C1—C2	119.1 (3)	O8—C16—C15	118.6 (3)
O1—C1—C2	115.0 (3)	C17—C16—C15	120.3 (3)
N1—C2—C3	127.4 (3)	C18—C17—C16	120.5 (3)
N1—C2—C1	111.6 (3)	C18—C17—H17	119.7
C3—C2—C1	120.9 (3)	C16—C17—H17	119.7

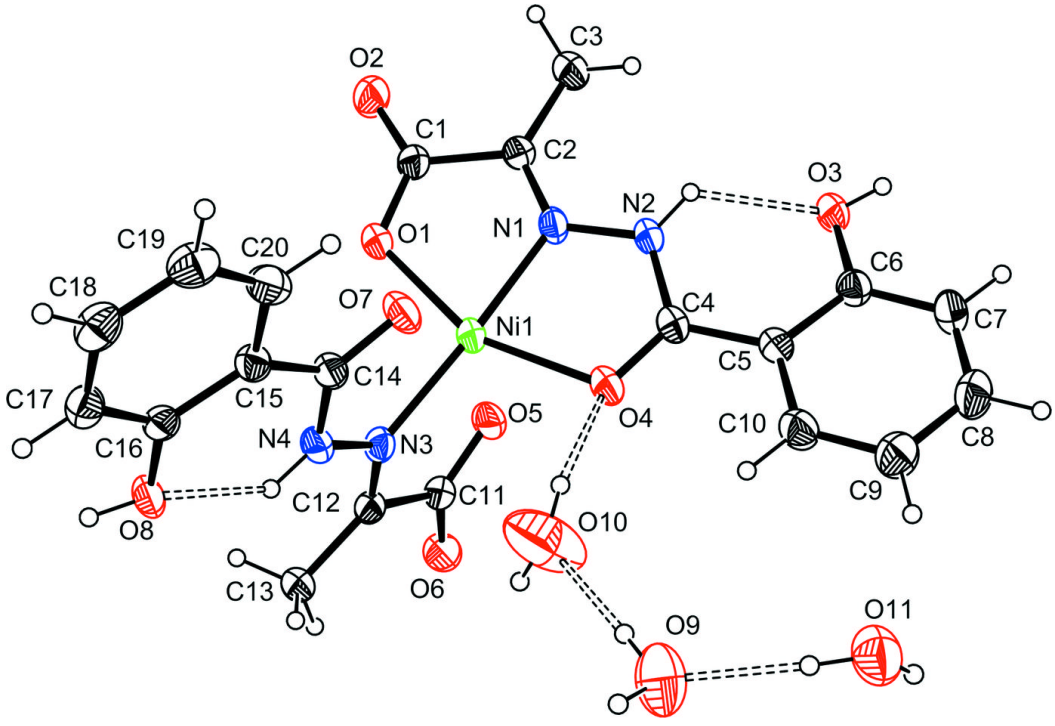
C2—C3—H3A	109.5	C17—C18—C19	120.2 (4)
C2—C3—H3B	109.5	C17—C18—H18	119.9
H3A—C3—H3B	109.5	C19—C18—H18	119.9
C2—C3—H3C	109.5	C20—C19—C18	119.8 (4)
H3A—C3—H3C	109.5	C20—C19—H19	120.1
H3B—C3—H3C	109.5	C18—C19—H19	120.1
O4—C4—N2	118.7 (3)	C19—C20—C15	121.3 (4)
O4—C4—C5	122.0 (3)	C19—C20—H20	119.4
N2—C4—C5	119.3 (3)	C15—C20—H20	119.4
C10—C5—C6	118.2 (3)	H9A—O9—H9B	105.9
C10—C5—C4	117.8 (3)	H10A—O10—H10B	108.5
C6—C5—C4	124.1 (3)	H11A—O11—H11B	108.1

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O5^{i}	0.82	1.81	2.606 (3)	162
O8—H8···O1^{ii}	0.82	1.77	2.584 (3)	170
N2—H2···O3	0.86	2.02	2.622 (3)	126
N2—H2···O11^{iii}	0.86	2.18	2.826 (4)	132
N4—H4···O8	0.86	1.91	2.571 (3)	133
O9—H9A···O10	0.87	1.87	2.741 (6)	178
O9—H9B···O6^{iv}	0.86	1.98	2.838 (4)	171
O10—H10A···O6^{v}	0.86	2.05	2.882 (5)	163
O10—H10B···O4	0.86	2.16	2.978 (5)	160
O11—H11A···O9	0.86	1.88	2.741 (4)	173
O11—H11B···O2^{vi}	0.86	2.13	2.890 (4)	148

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

Fig. 1


C3
O2
C1
C2
O3
N2
N1
O1
C19
C20
C6
C18
O7
Ni1
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C7
C14
C5
C17
C15
O5
O4
C10
C8
N4
N3
C16
C12
C11
O10
C9
O8
C13
O6
O9
O11