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Tetraaquabis[2-(3-benzoylphenyl)-propanoato-κ*O*]nickel(II) monohydrate

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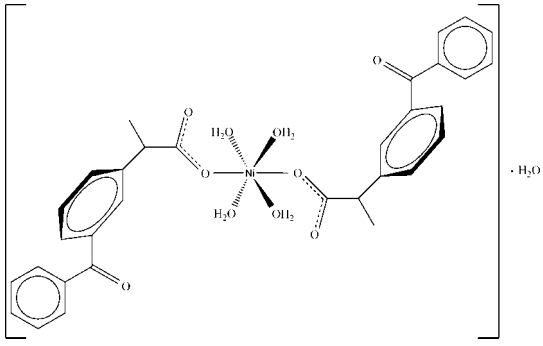
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.007 Å; R factor = 0.053; wR factor = 0.174; data-to-parameter ratio = 16.3.

The title complex, $[Ni(C_{16}H_{13}O_3)_2(H_2O)_4]\cdot H_2O$, is a transition metal complex of ketoprofen [2-(3-benzoylphenyl)propanoic acid], a well known non-steroidal anti-inflammatory drug. The asymmetric unit consists of half each of two complex molecules and one solvent water molecule. Both Ni^{II} atoms are located on crystallographic centres of inversion, each exhibiting an octahedral coordination geometry involving two carboxylate O atoms of different ketoprofenate ligands and four water molecules. A three-dimensional hydrogen-bonding framework is formed by O—H···O hydrogen-bonding interactions.

Related literature

For the crystal structures of the ketoprofen complexes (ketoprofenato)trimethyltin(IV) and tetraaquabis[2-(3-benzoylphenyl)propanoato-κ*O*]nickel(II) monohydrate, see: Tahir *et al.* (1997); Zhang *et al.* (2007).



Experimental

Crystal data

$[Ni(C_{16}H_{13}O_3)_2(H_2O)_4]\cdot H_2O$
M_r = 655.32
Triclinic, $P\overline{1}$
a = 8.559 (2) Å
b = 8.851 (2) Å
c = 21.875 (4) Å
α = 92.05 (3)°
β = 99.78 (3)°
γ = 108.90 (3)°
V = 1537.7 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.69 mm^{-1}
T = 295 (2) K
0.34 × 0.25 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.799, T_{max} = 0.880
15351 measured reflections
7032 independent reflections
4192 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.174
S = 1.14
7032 reflections
432 parameters
15 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W1···O2	0.85 (3)	1.82 (3)	2.640 (4)	162 (4)
O2W—H2W1···O4W^{i}	0.85 (3)	1.92 (3)	2.766 (4)	177 (4)
O2W—H2W2···O5W^{ii}	0.84 (3)	1.99 (3)	2.809 (4)	162 (4)
O3W—H3W1···O5W^{iii}	0.85 (4)	1.88 (4)	2.669 (4)	155 (4)
O3W—H3W2···O1^{iii}	0.85 (4)	1.812 (17)	2.647 (4)	169 (4)
O4W—H4W1···O5	0.84 (3)	1.82 (3)	2.645 (4)	166 (4)
O4W—H4W2···O5W^{iii}	0.84 (3)	1.97 (3)	2.795 (4)	166 (3)
O5W—H5W1···O3	0.85 (2)	1.98 (3)	2.755 (4)	152 (5)
O5W—H5W2···O2^{iv}	0.85 (5)	1.82 (5)	2.651 (4)	168 (6)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*-II (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2040).

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Zhang, Z.-Y., Chen, P.-G., Deng, Z.-P., Yu, N. & Liu, B.-Y. (2007). *Acta Cryst.* E**63**, m1900–m1901.

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supplementary materials

Acta Cryst. (2007). E**63**, m2883 [**doi:10.1107/S1600536807052762**]

Tetraaquabis[2-(3-benzoylphenyl)propanoato-*κO*]nickel(II) monohydrate

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Comment

2-(3-benzoylphenyl)propanoic acid is known as a nonsteroidal anti-inflamatory drug, but there is little information on the structure of its metal complexes (Tahir *et al.*, 1997). The preceeding report (Zhang *et al.*, 2007) details the synthesis and crystal structure of tetraaquabis[2-(3-benzoylphenyl)propanoato-κ*O*] cobalt(II) monohydrate. Replacing cobalt by nickel in a similar reaction leads to the formation of the title complex, (I), (Fig. 1). Similar structural descriptions of the Co^{II} apply to the isomorphous complex.

As illustrated in Fig. 1, the crystal structure of the title compound consists of two crystallographically independent neutral mononuclear Ni^{II} complex molecules and one water of crystallization. The Ni^{II} atoms of the two molecules are situated at crystallographic centers of inversion and display an octahedral geometry defined by two carboxylate O atoms of two ketoprofenato ligands and four water molecules. A three-dimensional supramolecular network structure is formed through the extended hydrogen bonding interactions between water molecules and carboxylate O atoms (Table 1).

Experimental

The title complex was prepared by the addition of nickel diacetate tetrahydrate (2.49 g, 10 mmol) to a hot aqueous solution of racemic 2-(3-benzoylphenyl)propanoic acid (2.54 g, 10 mmol); the pH was adjusted to 6 with 0.1*M* sodium hydroxide. The solution was allowed to evaporate at room temperature. Green prismatic crystals separated from the filtered solution after several days. Elemental analysis: calcd. for $C_{32}H_{36}NiO_{11}$: C 58.63, H 5.54%; found: C 58.52, H 5.69%.

Refinement

The H atoms were placed in calculated positions [C—H = 0.93 and 0.97 Å and U_{iso}(H) = 1.2U_{eq} (C) for aromatic H atoms and methine H atoms, respectively, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq} (C) for methyl group H atoms] and were included in the refinement in the riding-model approximation. The H atoms of water molecules were located in difference Fourier maps and refined with the O—H distance restrained to 0.85 (1) Å and U_{iso}(H) = 1.5U_{eq}(O).

Figures

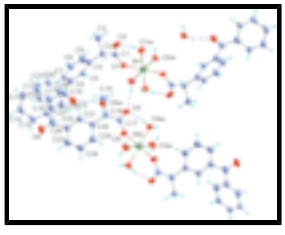


Fig. 1. Molecular structure of (I) with 30% probability ellipsoids for the non-H atoms.

Tetraaquabis[2-(3-benzoylphenyl)propanoato-κ*O*]nickel(II) monohydrate

Crystal data

$[Ni(C_{16}H_{13}O_3)_2(H_2O)_4]\cdot H_2O$	$Z = 2$
$M_r = 655.32$	$F_{000} = 688$
Triclinic, $P\overline{1}$	$D_x = 1.415$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.559$ (2) Å	Cell parameters from 7166 reflections
$b = 8.851$ (2) Å	$\theta = 3.2$–27.5º
$c = 21.875$ (4) Å	$\mu = 0.69$ mm^{-1}
$\alpha = 92.05$ (3)º	$T = 295$ (2) K
$\beta = 99.78$ (3)º	Sheet, pale green
$\gamma = 108.90$ (3)º	0.34 × 0.25 × 0.19 mm
$V = 1537.7$ (6) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	7032 independent reflections
Radiation source: fine-focus sealed tube	4192 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.049$
Detector resolution: 10.000 pixels mm^{-1}	$\theta_{max} = 27.6$º
$T = 295(2)$ K	$\theta_{min} = 3.2$º
ω scans	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -11 \rightarrow 11$
$T_{min} = 0.799$, $T_{max} = 0.880$	$l = -28 \rightarrow 28$
15351 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.053$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.174$	$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{max} < 0.001$
7032 reflections	$\Delta\rho_{max} = 0.78$ e Å^{-3}
432 parameters	$\Delta\rho_{min} = -0.74$ e Å^{-3}
15 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.0000	0.5000	0.5000	0.02982 (19)
Ni2	0.5000	0.0000	0.5000	0.02956 (19)
O1W	0.0183 (3)	0.7333 (3)	0.48051 (12)	0.0373 (6)
H1W1	−0.054 (4)	0.715 (5)	0.4469 (11)	0.056*
H1W2	0.104 (3)	0.813 (3)	0.4787 (17)	0.056*
O2W	−0.1773 (3)	0.5045 (3)	0.55378 (13)	0.0374 (6)
H2W1	−0.266 (3)	0.423 (3)	0.548 (2)	0.056*
H2W2	−0.203 (5)	0.588 (2)	0.550 (2)	0.056*
O3W	0.3556 (4)	−0.0891 (3)	0.56486 (13)	0.0425 (7)
H3W1	0.329 (5)	−0.021 (3)	0.5854 (19)	0.064*
H3W2	0.295 (5)	−0.1834 (18)	0.569 (2)	0.064*
O4W	0.5285 (3)	0.2437 (3)	0.53314 (12)	0.0333 (6)
H4W1	0.449 (3)	0.251 (5)	0.5061 (12)	0.050*
H4W2	0.497 (4)	0.218 (5)	0.5668 (10)	0.050*
O5W	−0.3714 (5)	−0.1696 (4)	0.36447 (13)	0.0526 (8)
H5W1	−0.420 (6)	−0.176 (6)	0.3269 (9)	0.079*
H5W2	−0.313 (6)	−0.231 (5)	0.3679 (19)	0.079*
O1	−0.2010 (3)	0.3951 (3)	0.42308 (11)	0.0307 (6)
O2	−0.2224 (4)	0.6112 (3)	0.38158 (13)	0.0524 (8)
O3	−0.4238 (5)	−0.1841 (4)	0.23630 (14)	0.0631 (10)
O4	0.2831 (3)	−0.0170 (3)	0.43855 (12)	0.0388 (6)
O5	0.2942 (4)	0.2352 (3)	0.43586 (12)	0.0402 (6)
O6	0.1073 (5)	−0.1320 (4)	0.08882 (14)	0.0726 (11)
C1	−0.2702 (5)	0.4638 (4)	0.38376 (16)	0.0315 (8)
C2	−0.4235 (5)	0.3574 (4)	0.33630 (15)	0.0315 (8)
H2	−0.4809	0.2635	0.3564	0.038*
C3	−0.5476 (5)	0.4426 (5)	0.31537 (18)	0.0419 (10)
H3A	−0.5800	0.4813	0.3511	0.063*
H3B	−0.6454	0.3693	0.2885	0.063*
H3C	−0.4963	0.5314	0.2932	0.063*
C4	−0.3642 (5)	0.2982 (4)	0.28058 (16)	0.0332 (8)
C5	−0.4119 (5)	0.1371 (4)	0.26247 (16)	0.0327 (8)
H5	−0.4775	0.0633	0.2850	0.039*
C6	−0.3628 (5)	0.0826 (5)	0.21024 (17)	0.0385 (9)
C7	−0.2675 (6)	0.1917 (5)	0.17658 (19)	0.0492 (11)
H7	−0.2376	0.1563	0.1412	0.059*
C8	−0.2167 (7)	0.3521 (5)	0.1949 (2)	0.0621 (14)
H8	−0.1492	0.4258	0.1728	0.074*
C9	−0.2661 (6)	0.4049 (5)	0.2466 (2)	0.0501 (11)
H9	−0.2324	0.5143	0.2585	0.060*
C10	−0.3988 (5)	−0.0920 (5)	0.19615 (18)	0.0408 (10)
C11	−0.3997 (6)	−0.1565 (5)	0.13194 (19)	0.0445 (10)
C12	−0.4931 (7)	−0.1245 (6)	0.0799 (2)	0.0632 (14)
H12	−0.5546	−0.0563	0.0837	0.076*
C13	−0.4960 (9)	−0.1930 (7)	0.0220 (2)	0.0818 (19)

H13	−0.5623	−0.1734	−0.0130	0.098*
C14	−0.4026 (10)	−0.2894 (7)	0.0156 (3)	0.087 (2)
H14	−0.4022	−0.3323	−0.0238	0.105*
C15	−0.3110 (9)	−0.3221 (7)	0.0665 (3)	0.0822 (19)
H15	−0.2477	−0.3882	0.0620	0.099*
C16	−0.3097 (7)	−0.2590 (6)	0.1254 (2)	0.0602 (13)
H16	−0.2489	−0.2851	0.1604	0.072*
C17	0.2332 (5)	0.0918 (4)	0.41577 (16)	0.0326 (8)
C18	0.0788 (5)	0.0336 (5)	0.36172 (17)	0.0417 (10)
H18	−0.0072	−0.0492	0.3779	0.050*
C19	0.0037 (6)	0.1592 (6)	0.3454 (2)	0.0547 (12)
H19A	−0.0273	0.1974	0.3816	0.082*
H19B	−0.0945	0.1151	0.3132	0.082*
H19C	0.0842	0.2467	0.3309	0.082*
C20	0.1152 (5)	−0.0518 (5)	0.30667 (17)	0.0385 (9)
C21	0.1131 (5)	0.0027 (5)	0.24809 (17)	0.0388 (9)
H21	0.0941	0.0992	0.2421	0.047*
C22	0.1386 (5)	−0.0820 (5)	0.19768 (18)	0.0394 (9)
C23	0.1690 (6)	−0.2258 (5)	0.2073 (2)	0.0513 (12)
H23	0.1865	−0.2847	0.1747	0.062*
C24	0.1727 (7)	−0.2792 (5)	0.2655 (2)	0.0572 (13)
H24	0.1937	−0.3747	0.2718	0.069*
C25	0.1461 (6)	−0.1956 (5)	0.3148 (2)	0.0495 (11)
H25	0.1489	−0.2352	0.3536	0.059*
C26	0.1263 (6)	−0.0346 (5)	0.13258 (19)	0.0492 (11)
C27	0.1325 (6)	0.1312 (5)	0.11962 (17)	0.0441 (10)
C28	0.2545 (6)	0.2649 (5)	0.15325 (19)	0.0510 (11)
H28	0.3303	0.2525	0.1869	0.061*
C29	0.2646 (7)	0.4157 (6)	0.1374 (2)	0.0633 (14)
H29	0.3485	0.5050	0.1597	0.076*
C30	0.1523 (8)	0.4347 (7)	0.0891 (2)	0.0710 (16)
H30	0.1579	0.5374	0.0792	0.085*
C31	0.0293 (7)	0.3025 (6)	0.0546 (2)	0.0621 (14)
H31	−0.0473	0.3160	0.0215	0.074*
C32	0.0214 (6)	0.1525 (6)	0.06945 (18)	0.0545 (12)
H32	−0.0594	0.0634	0.0457	0.065*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0269 (4)	0.0246 (3)	0.0340 (4)	0.0048 (3)	0.0027 (3)	0.0027 (3)
Ni2	0.0262 (4)	0.0252 (3)	0.0336 (4)	0.0051 (3)	0.0029 (3)	0.0031 (3)
O1W	0.0306 (16)	0.0254 (12)	0.0461 (15)	−0.0004 (11)	−0.0008 (12)	0.0090 (11)
O2W	0.0320 (15)	0.0278 (13)	0.0512 (16)	0.0068 (11)	0.0119 (12)	−0.0005 (12)
O3W	0.0550 (19)	0.0256 (13)	0.0459 (16)	0.0042 (13)	0.0253 (13)	0.0047 (12)
O4W	0.0323 (15)	0.0258 (12)	0.0385 (14)	0.0057 (11)	0.0063 (11)	0.0013 (11)
O5W	0.079 (3)	0.0448 (17)	0.0418 (16)	0.0346 (17)	0.0068 (15)	−0.0009 (14)
O1	0.0274 (14)	0.0263 (12)	0.0338 (13)	0.0072 (10)	−0.0031 (10)	0.0036 (10)

O2	0.053 (2)	0.0290 (14)	0.0599 (18)	0.0078 (13)	−0.0205 (15)	0.0109 (13)
O3	0.108 (3)	0.0454 (17)	0.0456 (18)	0.0350 (19)	0.0218 (18)	0.0069 (15)
O4	0.0310 (15)	0.0298 (13)	0.0482 (15)	0.0096 (11)	−0.0108 (12)	0.0042 (12)
O5	0.0396 (16)	0.0349 (14)	0.0448 (15)	0.0143 (12)	0.0005 (12)	0.0057 (12)
O6	0.111 (3)	0.068 (2)	0.0395 (17)	0.036 (2)	0.0074 (18)	−0.0136 (17)
C1	0.031 (2)	0.0272 (17)	0.0331 (18)	0.0061 (15)	0.0053 (15)	0.0014 (15)
C2	0.030 (2)	0.0313 (18)	0.0288 (17)	0.0076 (15)	0.0005 (14)	0.0008 (15)
C3	0.036 (2)	0.043 (2)	0.043 (2)	0.0148 (18)	−0.0045 (17)	−0.0045 (18)
C4	0.031 (2)	0.0343 (19)	0.0303 (18)	0.0085 (16)	0.0012 (15)	0.0004 (16)
C5	0.034 (2)	0.0369 (19)	0.0276 (17)	0.0139 (17)	0.0031 (15)	0.0030 (15)
C6	0.036 (2)	0.041 (2)	0.036 (2)	0.0106 (18)	0.0050 (16)	0.0010 (17)
C7	0.061 (3)	0.048 (2)	0.040 (2)	0.016 (2)	0.020 (2)	0.0010 (19)
C8	0.078 (4)	0.046 (3)	0.055 (3)	0.001 (2)	0.032 (3)	0.003 (2)
C9	0.057 (3)	0.036 (2)	0.050 (2)	0.004 (2)	0.015 (2)	−0.0025 (19)
C10	0.048 (3)	0.040 (2)	0.037 (2)	0.0189 (19)	0.0065 (18)	−0.0008 (18)
C11	0.052 (3)	0.034 (2)	0.046 (2)	0.0088 (19)	0.017 (2)	−0.0015 (18)
C12	0.082 (4)	0.064 (3)	0.045 (3)	0.027 (3)	0.011 (2)	0.005 (2)
C13	0.114 (6)	0.075 (4)	0.042 (3)	0.019 (4)	0.004 (3)	0.002 (3)
C14	0.132 (6)	0.058 (3)	0.057 (3)	0.002 (4)	0.040 (4)	−0.017 (3)
C15	0.100 (5)	0.065 (3)	0.086 (4)	0.023 (3)	0.046 (4)	−0.015 (3)
C16	0.069 (4)	0.050 (3)	0.065 (3)	0.023 (3)	0.017 (3)	−0.005 (2)
C17	0.028 (2)	0.039 (2)	0.0315 (18)	0.0110 (17)	0.0079 (15)	0.0015 (16)
C18	0.046 (3)	0.047 (2)	0.0324 (19)	0.021 (2)	−0.0003 (17)	0.0012 (18)
C19	0.045 (3)	0.070 (3)	0.051 (3)	0.028 (2)	−0.002 (2)	−0.005 (2)
C20	0.039 (2)	0.041 (2)	0.035 (2)	0.0182 (18)	−0.0020 (16)	−0.0017 (17)
C21	0.038 (2)	0.039 (2)	0.038 (2)	0.0160 (18)	−0.0001 (17)	−0.0057 (17)
C22	0.037 (2)	0.039 (2)	0.039 (2)	0.0131 (18)	0.0003 (17)	−0.0053 (18)
C23	0.057 (3)	0.051 (3)	0.048 (2)	0.026 (2)	0.002 (2)	−0.008 (2)
C24	0.073 (4)	0.045 (2)	0.058 (3)	0.031 (2)	0.002 (2)	−0.007 (2)
C25	0.059 (3)	0.046 (2)	0.044 (2)	0.023 (2)	0.002 (2)	0.003 (2)
C26	0.049 (3)	0.054 (3)	0.040 (2)	0.013 (2)	0.0041 (19)	−0.009 (2)
C27	0.048 (3)	0.051 (2)	0.031 (2)	0.016 (2)	0.0046 (17)	−0.0027 (18)
C28	0.054 (3)	0.056 (3)	0.037 (2)	0.016 (2)	−0.0021 (19)	−0.002 (2)
C29	0.081 (4)	0.053 (3)	0.048 (3)	0.014 (3)	0.011 (2)	0.000 (2)
C30	0.118 (5)	0.064 (3)	0.047 (3)	0.045 (3)	0.028 (3)	0.015 (2)
C31	0.081 (4)	0.077 (3)	0.036 (2)	0.040 (3)	0.004 (2)	0.004 (2)
C32	0.059 (3)	0.071 (3)	0.031 (2)	0.023 (3)	0.0002 (19)	−0.006 (2)

Geometric parameters (Å, °)

Ni1—O2W	2.081 (3)	C8—H8	0.9300
Ni1—O1W	2.085 (2)	C9—H9	0.9300
Ni1—O1	2.127 (2)	C10—C11	1.495 (5)
Ni2—O3W	2.051 (3)	C11—C12	1.370 (6)
Ni2—O4	2.057 (2)	C11—C16	1.384 (6)
Ni2—O4W	2.174 (2)	C12—C13	1.377 (7)
Ni1—O2W^{i}	2.081 (3)	C12—H12	0.9300
Ni1—O1W^{i}	2.085 (2)	C13—C14	1.363 (9)

Ni1—O1^{i}	2.127 (2)	C13—H13	0.9300
Ni2—O3W^{ii}	2.051 (3)	C14—C15	1.347 (9)
Ni2—O4^{ii}	2.057 (2)	C14—H14	0.9300
Ni2—O4W^{ii}	2.174 (2)	C15—C16	1.383 (7)
O1W—H1W1	0.85 (3)	C15—H15	0.9300
O1W—H1W2	0.85 (3)	C16—H16	0.9300
O2W—H2W1	0.85 (3)	C17—C18	1.547 (5)
O2W—H2W2	0.84 (3)	C18—C19	1.480 (6)
O3W—H3W1	0.85 (4)	C18—C20	1.528 (5)
O3W—H3W2	0.85 (4)	C18—H18	0.9800
O4W—H4W1	0.84 (3)	C19—H19A	0.9600
O4W—H4W2	0.84 (3)	C19—H19B	0.9600
O5W—H5W1	0.85 (2)	C19—H19C	0.9600
O5W—H5W2	0.85 (5)	C20—C21	1.384 (5)
O1—C1	1.248 (4)	C20—C25	1.393 (6)
O2—C1	1.240 (4)	C21—C22	1.398 (5)
O3—C10	1.218 (5)	C21—H21	0.9300
O4—C17	1.260 (4)	C22—C23	1.395 (6)
O5—C17	1.237 (4)	C22—C26	1.495 (6)
O6—C26	1.218 (5)	C23—C24	1.372 (6)
C1—C2	1.534 (5)	C23—H23	0.9300
C2—C3	1.512 (5)	C24—C25	1.378 (6)
C2—C4	1.534 (5)	C24—H24	0.9300
C2—H2	0.9800	C25—H25	0.9300
C3—H3A	0.9600	C26—C27	1.490 (6)
C3—H3B	0.9600	C27—C28	1.381 (6)
C3—H3C	0.9600	C27—C32	1.386 (6)
C4—C5	1.373 (5)	C28—C29	1.370 (7)
C4—C9	1.378 (6)	C28—H28	0.9300
C5—C6	1.403 (5)	C29—C30	1.357 (7)
C5—H5	0.9300	C29—H29	0.9300
C6—C7	1.372 (6)	C30—C31	1.385 (8)
C6—C10	1.484 (5)	C30—H30	0.9300
C7—C8	1.366 (6)	C31—C32	1.361 (7)
C7—H7	0.9300	C31—H31	0.9300
C8—C9	1.388 (6)	C32—H32	0.9300
O2W—Ni1—O1W	88.29 (11)	C8—C9—H9	119.4
O2W—Ni1—O1W^{i}	91.71 (11)	O3—C10—C6	121.4 (3)
O2W—Ni1—O1	88.56 (10)	O3—C10—C11	118.9 (3)
O1W—Ni1—O1	93.12 (10)	C6—C10—C11	119.7 (4)
O2W—Ni1—O1^{i}	91.44 (10)	C12—C11—C16	119.1 (4)
O1W—Ni1—O1^{i}	86.88 (10)	C12—C11—C10	122.3 (4)
O3W—Ni2—O4^{ii}	91.44 (12)	C16—C11—C10	118.5 (4)
O3W—Ni2—O4	88.56 (12)	C11—C12—C13	120.1 (5)
O3W—Ni2—O4W	90.55 (10)	C11—C12—H12	119.9
O4—Ni2—O4W	90.65 (10)	C13—C12—H12	119.9
O3W—Ni2—O4W^{ii}	89.45 (10)	C14—C13—C12	120.6 (6)

O4—Ni2—O4W^{ii}	89.35 (10)	C14—C13—H13	119.7
O2W^{i}—Ni1—O2W	180.000 (1)	C12—C13—H13	119.7
O2W^{i}—Ni1—O1W	91.71 (11)	C15—C14—C13	119.7 (5)
O2W^{i}—Ni1—O1W^{i}	88.29 (11)	C15—C14—H14	120.2
O1W—Ni1—O1W^{i}	180.000 (1)	C13—C14—H14	120.2
O2W^{i}—Ni1—O1	91.44 (10)	C14—C15—C16	121.0 (6)
O1W^{i}—Ni1—O1	86.88 (10)	C14—C15—H15	119.5
O2W^{i}—Ni1—O1^{i}	88.56 (10)	C16—C15—H15	119.5
O1W^{i}—Ni1—O1^{i}	93.12 (10)	C15—C16—C11	119.4 (5)
O1—Ni1—O1^{i}	180.000 (1)	C15—C16—H16	120.3
O3W^{ii}—Ni2—O3W	180.000 (1)	C11—C16—H16	120.3
O3W^{ii}—Ni2—O4^{ii}	88.56 (12)	O5—C17—O4	124.2 (3)
O3W^{ii}—Ni2—O4	91.44 (12)	O5—C17—C18	120.4 (3)
O4^{ii}—Ni2—O4	180.000 (1)	O4—C17—C18	115.3 (3)
O3W^{ii}—Ni2—O4W	89.45 (10)	C19—C18—C20	115.0 (3)
O4^{ii}—Ni2—O4W	89.35 (10)	C19—C18—C17	113.1 (3)
O3W^{ii}—Ni2—O4W^{ii}	90.55 (10)	C20—C18—C17	111.6 (3)
O4^{ii}—Ni2—O4W^{ii}	90.65 (10)	C19—C18—H18	105.4
O4W—Ni2—O4W^{ii}	180.000 (1)	C20—C18—H18	105.4
Ni1—O1W—H1W1	101 (3)	C17—C18—H18	105.4
Ni1—O1W—H1W2	130 (3)	C18—C19—H19A	109.5
H1W1—O1W—H1W2	111 (4)	C18—C19—H19B	109.5
Ni1—O2W—H2W1	116 (3)	H19A—C19—H19B	109.5
Ni1—O2W—H2W2	111 (3)	C18—C19—H19C	109.5
H2W1—O2W—H2W2	110 (3)	H19A—C19—H19C	109.5
Ni2—O3W—H3W1	116 (3)	H19B—C19—H19C	109.5
Ni2—O3W—H3W2	131 (3)	C21—C20—C25	117.7 (4)
H3W1—O3W—H3W2	110 (4)	C21—C20—C18	123.6 (3)
Ni2—O4W—H4W1	95 (3)	C25—C20—C18	118.6 (4)
Ni2—O4W—H4W2	94 (3)	C20—C21—C22	122.5 (4)
H4W1—O4W—H4W2	111 (4)	C20—C21—H21	118.8
H5W1—O5W—H5W2	110 (4)	C22—C21—H21	118.8
C1—O1—Ni1	128.4 (2)	C23—C22—C21	118.4 (4)
C17—O4—Ni2	130.0 (2)	C23—C22—C26	116.9 (4)
O2—C1—O1	123.7 (3)	C21—C22—C26	124.7 (4)
O2—C1—C2	119.2 (3)	C24—C23—C22	119.3 (4)
O1—C1—C2	117.1 (3)	C24—C23—H23	120.4
C3—C2—C1	112.2 (3)	C22—C23—H23	120.4
C3—C2—C4	111.2 (3)	C23—C24—C25	122.0 (4)
C1—C2—C4	109.4 (3)	C23—C24—H24	119.0
C3—C2—H2	108.0	C25—C24—H24	119.0
C1—C2—H2	108.0	C24—C25—C20	120.2 (4)
C4—C2—H2	108.0	C24—C25—H25	119.9
C2—C3—H3A	109.5	C20—C25—H25	119.9
C2—C3—H3B	109.5	O6—C26—C27	118.6 (4)

H3A—C3—H3B	109.5	O6—C26—C22	120.5 (4)
C2—C3—H3C	109.5	C27—C26—C22	120.9 (3)
H3A—C3—H3C	109.5	C28—C27—C32	118.8 (4)
H3B—C3—H3C	109.5	C28—C27—C26	121.8 (4)
C5—C4—C9	118.5 (3)	C32—C27—C26	119.2 (4)
C5—C4—C2	120.5 (3)	C29—C28—C27	120.5 (4)
C9—C4—C2	121.0 (3)	C29—C28—H28	119.7
C4—C5—C6	120.7 (4)	C27—C28—H28	119.7
C4—C5—H5	119.7	C30—C29—C28	120.0 (5)
C6—C5—H5	119.7	C30—C29—H29	120.0
C7—C6—C5	119.6 (4)	C28—C29—H29	120.0
C7—C6—C10	120.6 (4)	C29—C30—C31	120.5 (5)
C5—C6—C10	119.4 (4)	C29—C30—H30	119.7
C8—C7—C6	120.2 (4)	C31—C30—H30	119.7
C8—C7—H7	119.9	C32—C31—C30	119.5 (5)
C6—C7—H7	119.9	C32—C31—H31	120.2
C7—C8—C9	119.8 (4)	C30—C31—H31	120.2
C7—C8—H8	120.1	C31—C32—C27	120.6 (4)
C9—C8—H8	120.1	C31—C32—H32	119.7
C4—C9—C8	121.3 (4)	C27—C32—H32	119.7
C4—C9—H9	119.4		
O2W^{i}—Ni1—O1—C1	−91.4 (3)	C13—C14—C15—C16	−0.2 (10)
O2W—Ni1—O1—C1	88.6 (3)	C14—C15—C16—C11	−2.1 (9)
O1W—Ni1—O1—C1	0.4 (3)	C12—C11—C16—C15	2.3 (8)
O1W^{i}—Ni1—O1—C1	−179.6 (3)	C10—C11—C16—C15	178.9 (5)
O3W^{ii}—Ni2—O4—C17	−67.2 (3)	Ni2—O4—C17—O5	−14.4 (6)
O3W—Ni2—O4—C17	112.8 (3)	Ni2—O4—C17—C18	169.2 (3)
O4W—Ni2—O4—C17	22.3 (3)	O5—C17—C18—C19	−8.5 (6)
O4W^{ii}—Ni2—O4—C17	−157.7 (3)	O4—C17—C18—C19	168.1 (4)
Ni1—O1—C1—O2	4.7 (6)	O5—C17—C18—C20	123.0 (4)
Ni1—O1—C1—C2	−174.8 (2)	O4—C17—C18—C20	−60.5 (5)
O2—C1—C2—C3	−29.9 (5)	C19—C18—C20—C21	13.1 (6)
O1—C1—C2—C3	149.7 (3)	C17—C18—C20—C21	−117.4 (4)
O2—C1—C2—C4	94.0 (4)	C19—C18—C20—C25	−164.4 (4)
O1—C1—C2—C4	−86.4 (4)	C17—C18—C20—C25	65.1 (5)
C3—C2—C4—C5	−110.6 (4)	C25—C20—C21—C22	0.8 (6)
C1—C2—C4—C5	124.9 (4)	C18—C20—C21—C22	−176.7 (4)
C3—C2—C4—C9	67.6 (5)	C20—C21—C22—C23	−0.7 (6)
C1—C2—C4—C9	−57.0 (5)	C20—C21—C22—C26	175.7 (4)
C9—C4—C5—C6	−0.4 (6)	C21—C22—C23—C24	0.1 (7)
C2—C4—C5—C6	177.8 (3)	C26—C22—C23—C24	−176.6 (4)
C4—C5—C6—C7	−0.7 (6)	C22—C23—C24—C25	0.4 (8)
C4—C5—C6—C10	172.8 (4)	C23—C24—C25—C20	−0.3 (8)
C5—C6—C7—C8	1.9 (7)	C21—C20—C25—C24	−0.3 (7)
C10—C6—C7—C8	−171.5 (4)	C18—C20—C25—C24	177.4 (4)
C6—C7—C8—C9	−2.0 (8)	C23—C22—C26—O6	15.3 (7)
C5—C4—C9—C8	0.3 (7)	C21—C22—C26—O6	−161.2 (4)
C2—C4—C9—C8	−177.9 (4)	C23—C22—C26—C27	−166.2 (4)

C7—C8—C9—C4	0.9 (8)	C21—C22—C26—C27	17.3 (7)
C7—C6—C10—O3	151.8 (5)	O6—C26—C27—C28	−133.4 (5)
C5—C6—C10—O3	−21.6 (6)	C22—C26—C27—C28	48.1 (7)
C7—C6—C10—C11	−26.3 (6)	O6—C26—C27—C32	41.5 (7)
C5—C6—C10—C11	160.3 (4)	C22—C26—C27—C32	−137.0 (4)
O3—C10—C11—C12	129.5 (5)	C32—C27—C28—C29	0.3 (7)
C6—C10—C11—C12	−52.4 (6)	C26—C27—C28—C29	175.3 (5)
O3—C10—C11—C16	−47.0 (6)	C27—C28—C29—C30	1.3 (8)
C6—C10—C11—C16	131.1 (4)	C28—C29—C30—C31	−1.6 (8)
C16—C11—C12—C13	−0.4 (8)	C29—C30—C31—C32	0.1 (8)
C10—C11—C12—C13	−176.8 (5)	C30—C31—C32—C27	1.5 (8)
C11—C12—C13—C14	−1.9 (9)	C28—C27—C32—C31	−1.7 (7)
C12—C13—C14—C15	2.2 (10)	C26—C27—C32—C31	−176.8 (4)

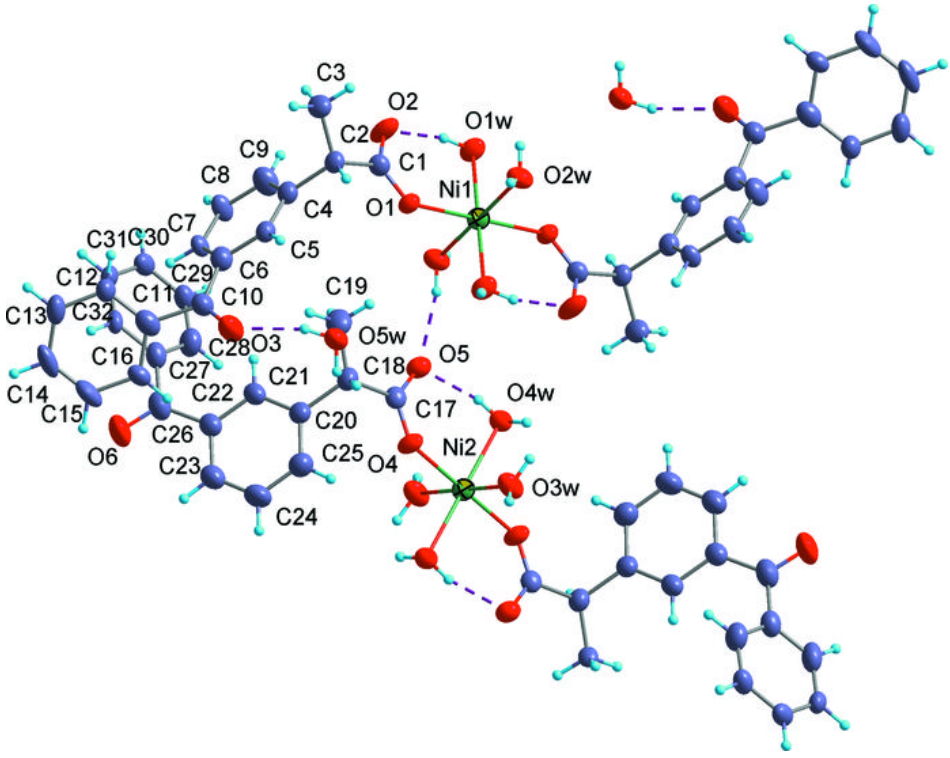
Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W1···O2	0.85 (3)	1.82 (3)	2.640 (4)	162 (4)
O2W—H2W1···O4W^{iii}	0.85 (3)	1.92 (3)	2.766 (4)	177 (4)
O2W—H2W2···O5^{i}	0.84 (3)	1.99 (3)	2.809 (4)	162 (4)
O3W—H3W1···O5W^{iv}	0.85 (4)	1.88 (4)	2.669 (4)	155 (4)
O3W—H3W2···O1^{iv}	0.85 (4)	1.812 (17)	2.647 (4)	169 (4)
O4W—H4W1···O5	0.84 (3)	1.82 (3)	2.645 (4)	166 (4)
O4W—H4W2···O5W^{iv}	0.84 (3)	1.97 (3)	2.795 (4)	166 (3)
O5W—H5W1···O3	0.85 (2)	1.98 (3)	2.755 (4)	152 (5)
O5W—H5W2···O2^{v}	0.85 (5)	1.82 (5)	2.651 (4)	168 (6)

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

Fig. 1


C3
O2
C2
O1w
C1
C9
C8
Ni1
O2w
C4
O1
C7
C31
C30
C5
C12
C29
C6
C11
C10
C19
C13
C32
O5w
C28
O3
C18
O5
C16
C27
C21
C14
C22
O4w
C17
C15
C20
C26
Ni2
C25
O6
C23
O4
O3w
C24