

Diaqua{2,2'-dimethoxy-6,6'-[(1*E*,1'*E*)-propane-1,3-diylbis(azanylylidene)bis(methanylylidene)]diphenolato}nickel(II)

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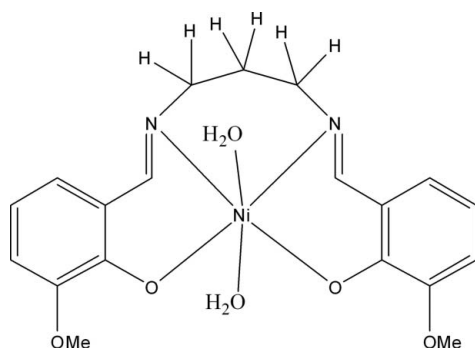
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.025; wR factor = 0.073; data-to-parameter ratio = 12.3.

In the molecule of the title compound, $[\text{Ni}(\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_4)(\text{H}_2\text{O})_2]$, the central Ni^{II} ion lies on a mirror plane and is surrounded by an N_2O_4 coordination set in the form of a distorted octahedron defined by the O atoms of two water molecules and by two phenolic O and two imine N atoms of the tetradentate Schiff base ligand. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the water molecules and the phenolic and methoxy O atoms of neighbouring molecules lead to the formation of rods propagating parallel to $[100]$.

Related literature

For related complexes with similar ligands, see: Sen *et al.* (2006); Thakurta *et al.* (2009*a,b*, 2010*a,b*).



Experimental

Crystal data

$[\text{Ni}(\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_4)(\text{H}_2\text{O})_2]$
 $M_r = 435.11$
 Orthorhombic, $Pnma$

$a = 7.4920$ (2) Å
 $b = 22.1442$ (6) Å
 $c = 11.6045$ (3) Å

$V = 1925.24$ (9) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 1.05$ mm⁻¹
 $T = 295$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
 $T_{\text{min}} = 0.782$, $T_{\text{max}} = 1.000$

20064 measured reflections
 1745 independent reflections
 1578 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.073$
 $S = 1.06$
 1745 reflections
 142 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.80$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1
Selected bond lengths (Å).

Ni1—O1 ⁱ	2.0131 (13)	Ni1—N1 ⁱ	2.0684 (16)
Ni1—O1	2.0131 (13)	Ni1—O4	2.1048 (19)
Ni1—N1	2.0684 (16)	Ni1—O3	2.113 (2)

Symmetry code: (i) $x, -y + \frac{1}{2}, z$.

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O4—H4O \cdots O2 ⁱⁱ	0.83 (1)	2.49 (2)	3.1433 (19)	136 (2)
O4—H4O \cdots O1 ⁱⁱⁱ	0.83 (1)	2.11 (2)	2.823 (2)	143 (2)
O3—H3O \cdots O1 ⁱⁱⁱ	0.83 (1)	2.37 (2)	3.059 (2)	140 (2)
O3—H3O \cdots O2 ⁱⁱⁱ	0.83 (1)	2.21 (2)	2.9432 (18)	147 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2747).

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supporting information

Acta Cryst. (2013). E69, m399 [https://doi.org/10.1107/S1600536813016188]

Diaqua{2,2'-dimethoxy-6,6'-[(1*E*,1'*E*)-propane-1,3-diylbis(azan-1-yl-1-ylidene)bis(methan-1-yl-1-ylidene)]diphenolato}nickel(II)

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S1. Comment

In the title compound, $[\text{Ni}(\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_4)(\text{H}_2\text{O})_2]$, the Ni(II) ion has site symmetry *m* and exhibits a distorted octahedral coordination environment defined by two water molecules and the tetradentate ligand, 6,6'-(*1E*,1'*E*)-(propane-1,3-diylbis(azan-1-yl-1-ylidene)bis(methan-1-yl-1-ylidene)bis(2-methoxyphenol) (*L*) that coordinates *via* two phenolic O and two imine N atoms (Fig. 1). The bond angles around the Ni(II) ion are slightly distorted from those of a regular octahedron and range from 85.57 (7)° to 175.62 (8)°. The two O_{water} molecules lie at the *trans* position of the octahedron. The *cis* Ni— $\text{O}_{\text{phenolic}}$ bond lengths [2.0131 (13) Å] is considerably smaller than the *trans* Ni— O_{water} bond lengths [2.113 (2) and 2.1048 (19) Å] (Table 1). In the molecule, the dihedral angle between the (C1—C7, N1) plane and its symmetry-related counterpart (C1A—C7A, N1A) is 27.58 (7)° [A] *x*, 1/2 - *y*, *z*].

The resulting coordination geometry around the metal cation is comparable to that of complexes with similar Schiff-bases. See, for example: Thakurta *et al.* (2009*a,b*,2010*a,b*).

In the crystal structure of the title compound, intermolecular O—H...O hydrogen bonds between water molecules as donor groups and phenolic O and methoxy O atoms of neighbouring molecules as acceptor groups are observed (Table 2). The hydrogen bonding interactions lead to the formation of rods propagating parallel to [100] (Fig. 2).

S2. Experimental

The tetradentate Schiff base precursor was prepared according to the literature procedure (Sen *et al.*, 2006). To a hot methanolic solution (20 ml) of $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ (0.248 g, 1.0 mmol), the ligand (1.0 mmol) was added, which produced immediately an intensely brown solution. The mixture was then kept at room temperature. After slow evaporation of the brown solution, dark chocolate-brown single crystals with a rectangular form were separated out in 5 days. The crystals were filtered off and washed with water and dried in air.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation. The H atoms of the water molecules were located in a difference map and were refined with an O—H distances restraint of 0.85 (1) Å.

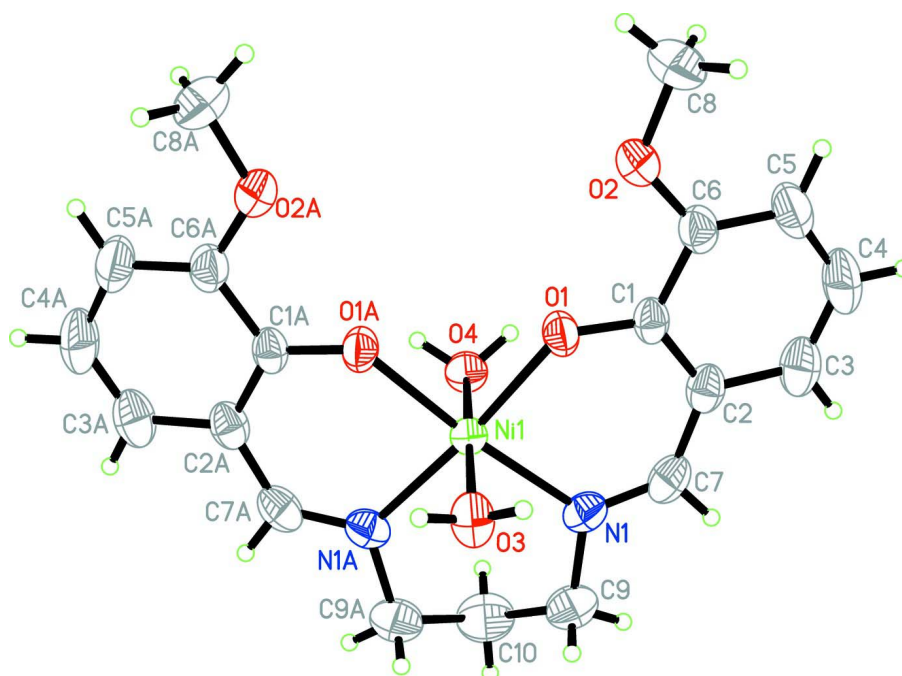


Figure 1

The molecular structure of the title complex, showing displacement ellipsoids at the 50% probability level. [Symmetry code: (A) $x, 1/2 - y, z$].

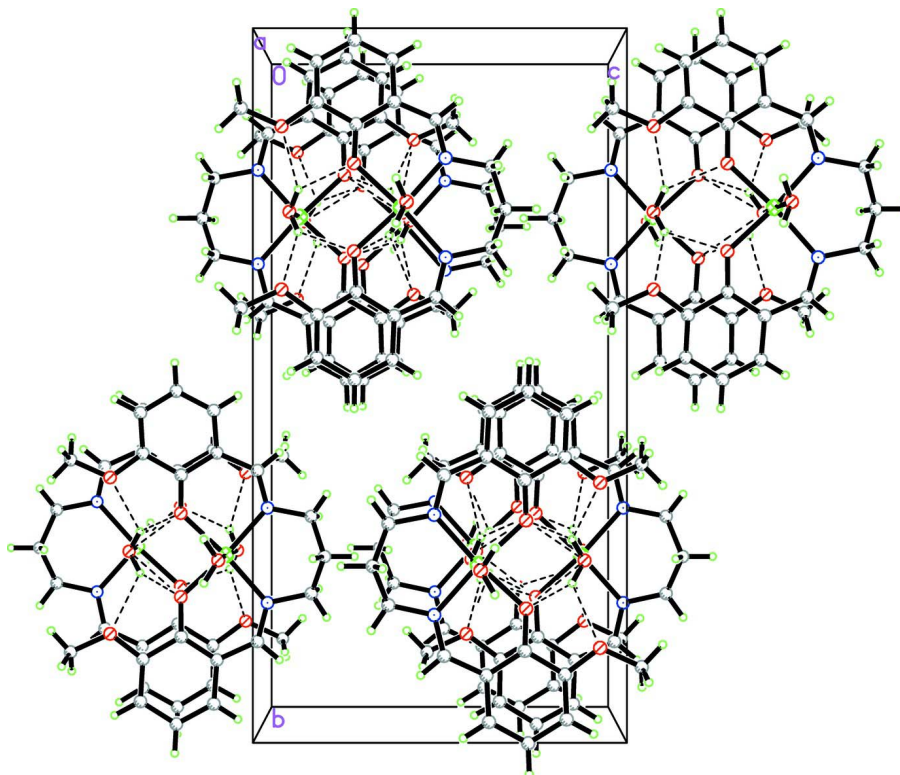


Figure 2

Packing diagram of the title compound as viewed down the a axis. Intermolecular O—H...O hydrogen bonds are shown as dashed lines.

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Crystal data

[Ni(C₁₉H₂₀N₂O₄)(H₂O)₂]

$M_r = 435.11$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 7.4920$ (2) Å

$b = 22.1442$ (6) Å

$c = 11.6045$ (3) Å

$V = 1925.24$ (9) Å³

$Z = 4$

$F(000) = 912$

$D_x = 1.501$ Mg m⁻³

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

Cell parameters from 8168 reflections

$\theta = 3.4$ – 29.2°

$\mu = 1.05$ mm⁻¹

$T = 295$ K

Rectangular, brown

$0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2007)

$T_{\min} = 0.782$, $T_{\max} = 1.000$

20064 measured reflections

1745 independent reflections

1578 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -8 \rightarrow 8$

$k = -26 \rightarrow 26$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.073$

$S = 1.06$

1745 reflections

142 parameters

2 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

$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.966P]$

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

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

$\Delta\rho_{\max} = 0.80 \text{ e } \text{Å}^{-3}$

$\Delta\rho_{\min} = -0.31 \text{ e } \text{Å}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.47233 (4)	0.2500	0.10284 (3)	0.02817 (13)
N1	0.4635 (2)	0.31989 (7)	-0.01527 (14)	0.0375 (4)
O1	0.48285 (18)	0.31175 (5)	0.22998 (11)	0.0359 (3)
O2	0.4497 (2)	0.36489 (7)	0.42580 (13)	0.0531 (4)
O3	0.7532 (3)	0.2500	0.08662 (18)	0.0406 (5)
O4	0.1914 (3)	0.2500	0.10516 (17)	0.0376 (4)
C1	0.4402 (2)	0.36858 (8)	0.22558 (17)	0.0324 (4)
C2	0.4123 (3)	0.40152 (8)	0.12334 (18)	0.0378 (4)
C3	0.3675 (3)	0.46364 (9)	0.1292 (2)	0.0512 (6)
H3	0.3515	0.4853	0.0613	0.061*
C4	0.3474 (3)	0.49203 (10)	0.2318 (2)	0.0605 (7)
H4	0.3166	0.5327	0.2339	0.073*
C5	0.3728 (3)	0.46043 (9)	0.3339 (2)	0.0527 (6)
H5	0.3581	0.4799	0.4042	0.063*
C6	0.4197 (3)	0.40052 (8)	0.33109 (18)	0.0396 (5)
C7	0.4323 (3)	0.37529 (9)	0.01098 (18)	0.0423 (5)
H7	0.4212	0.4018	-0.0508	0.051*
C8	0.4391 (4)	0.39102 (12)	0.5356 (2)	0.0696 (8)
H8A	0.5251	0.4230	0.5417	0.104*
H8B	0.4636	0.3609	0.5930	0.104*
H8C	0.3214	0.4070	0.5474	0.104*
C9	0.4892 (4)	0.30703 (12)	-0.1376 (2)	0.0594 (6)
H9A	0.6162	0.3042	-0.1531	0.071*
H9B	0.4428	0.3406	-0.1822	0.071*

C10	0.4007 (5)	0.2500	-0.1775 (3)	0.0624 (9)
H10A	0.2782	0.2500	-0.1504	0.075*
H10B	0.3976	0.2500	-0.2610	0.075*
H4O	0.150 (3)	0.2810 (7)	0.1357 (19)	0.059 (7)*
H3O	0.802 (3)	0.2808 (8)	0.113 (2)	0.067 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0280 (2)	0.0281 (2)	0.0284 (2)	0.000	-0.00017 (12)	0.000
N1	0.0365 (9)	0.0418 (9)	0.0342 (9)	0.0008 (7)	0.0009 (7)	0.0074 (7)
O1	0.0469 (8)	0.0247 (6)	0.0360 (7)	0.0037 (5)	-0.0038 (6)	-0.0011 (5)
O2	0.0838 (12)	0.0355 (8)	0.0401 (8)	-0.0063 (7)	0.0054 (8)	-0.0076 (6)
O3	0.0287 (10)	0.0336 (11)	0.0596 (13)	0.000	-0.0046 (9)	0.000
O4	0.0288 (10)	0.0396 (12)	0.0443 (11)	0.000	0.0044 (8)	0.000
C1	0.0260 (9)	0.0251 (8)	0.0461 (11)	-0.0024 (7)	0.0001 (8)	-0.0005 (7)
C2	0.0306 (10)	0.0315 (10)	0.0514 (12)	0.0004 (8)	-0.0014 (8)	0.0058 (8)
C3	0.0481 (13)	0.0344 (11)	0.0711 (16)	0.0043 (9)	-0.0079 (11)	0.0100 (10)
C4	0.0610 (15)	0.0280 (10)	0.093 (2)	0.0089 (10)	-0.0026 (13)	-0.0029 (12)
C5	0.0551 (13)	0.0333 (11)	0.0698 (16)	-0.0011 (9)	0.0075 (11)	-0.0142 (10)
C6	0.0383 (10)	0.0310 (10)	0.0494 (12)	-0.0057 (8)	0.0040 (9)	-0.0036 (8)
C7	0.0406 (11)	0.0406 (11)	0.0457 (12)	0.0008 (9)	-0.0037 (9)	0.0154 (9)
C8	0.099 (2)	0.0630 (16)	0.0469 (14)	-0.0099 (15)	0.0072 (13)	-0.0182 (12)
C9	0.0818 (18)	0.0607 (16)	0.0357 (12)	0.0025 (13)	0.0073 (11)	0.0096 (11)
C10	0.076 (2)	0.077 (2)	0.0342 (17)	0.000	-0.0113 (16)	0.000

Geometric parameters (Å, °)

Ni1—O1 ⁱ	2.0131 (13)	C3—C4	1.355 (3)
Ni1—O1	2.0131 (13)	C3—H3	0.9300
Ni1—N1	2.0684 (16)	C4—C5	1.389 (3)
Ni1—N1 ⁱ	2.0684 (16)	C4—H4	0.9300
Ni1—O4	2.1048 (19)	C5—C6	1.373 (3)
Ni1—O3	2.113 (2)	C5—H5	0.9300
N1—C7	1.286 (3)	C7—H7	0.9300
N1—C9	1.461 (3)	C8—H8A	0.9600
O1—C1	1.299 (2)	C8—H8B	0.9600
O2—C6	1.372 (3)	C8—H8C	0.9600
O2—C8	1.402 (3)	C9—C10	1.500 (3)
O3—H3O	0.830 (10)	C9—H9A	0.9700
O4—H4O	0.833 (9)	C9—H9B	0.9700
C1—C2	1.408 (3)	C10—C9 ⁱ	1.500 (3)
C1—C6	1.422 (3)	C10—H10A	0.9700
C2—C3	1.418 (3)	C10—H10B	0.9700
C2—C7	1.435 (3)		
O1 ⁱ —Ni1—O1	85.57 (7)	C3—C4—C5	120.0 (2)
O1 ⁱ —Ni1—N1	174.34 (6)	C3—C4—H4	120.0

O1—Ni1—N1	88.78 (6)	C5—C4—H4	120.0
O1 ⁱ —Ni1—N1 ⁱ	88.78 (6)	C6—C5—C4	120.1 (2)
O1—Ni1—N1 ⁱ	174.34 (6)	C6—C5—H5	119.9
N1—Ni1—N1 ⁱ	96.88 (9)	C4—C5—H5	119.9
O1 ⁱ —Ni1—O4	91.71 (6)	O2—C6—C5	125.4 (2)
O1—Ni1—O4	91.71 (6)	O2—C6—C1	112.71 (16)
N1—Ni1—O4	88.65 (6)	C5—C6—C1	121.9 (2)
N1 ⁱ —Ni1—O4	88.65 (6)	N1—C7—C2	128.35 (18)
O1 ⁱ —Ni1—O3	91.51 (6)	N1—C7—H7	115.8
O1—Ni1—O3	91.51 (6)	C2—C7—H7	115.8
N1—Ni1—O3	88.44 (6)	O2—C8—H8A	109.5
N1 ⁱ —Ni1—O3	88.44 (6)	O2—C8—H8B	109.5
O4—Ni1—O3	175.62 (8)	H8A—C8—H8B	109.5
C7—N1—C9	116.12 (18)	O2—C8—H8C	109.5
C7—N1—Ni1	124.25 (14)	H8A—C8—H8C	109.5
C9—N1—Ni1	119.60 (15)	H8B—C8—H8C	109.5
C1—O1—Ni1	128.27 (12)	N1—C9—C10	113.9 (2)
C6—O2—C8	118.81 (18)	N1—C9—H9A	108.8
Ni1—O3—H3O	113.9 (19)	C10—C9—H9A	108.8
Ni1—O4—H4O	112.4 (17)	N1—C9—H9B	108.8
O1—C1—C2	124.84 (17)	C10—C9—H9B	108.8
O1—C1—C6	118.32 (17)	H9A—C9—H9B	107.7
C2—C1—C6	116.85 (16)	C9 ⁱ —C10—C9	114.7 (3)
C1—C2—C3	119.82 (19)	C9 ⁱ —C10—H10A	108.6
C1—C2—C7	122.70 (17)	C9—C10—H10A	108.6
C3—C2—C7	117.45 (19)	C9 ⁱ —C10—H10B	108.6
C4—C3—C2	121.2 (2)	C9—C10—H10B	108.6
C4—C3—H3	119.4	H10A—C10—H10B	107.6
C2—C3—H3	119.4		

Symmetry code: (i) $x, -y+1/2, z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4O \cdots O2 ⁱⁱ	0.83 (1)	2.49 (2)	3.1433 (19)	136 (2)
O4—H4O \cdots O1 ⁱⁱ	0.83 (1)	2.11 (2)	2.823 (2)	143 (2)
O3—H3O \cdots O1 ⁱⁱⁱ	0.83 (1)	2.37 (2)	3.059 (2)	140 (2)
O3—H3O \cdots O2 ⁱⁱⁱ	0.83 (1)	2.21 (2)	2.9432 (18)	147 (2)

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