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Diacetatobis(propane-1,3-diamine)-nickel(II) dihydrate

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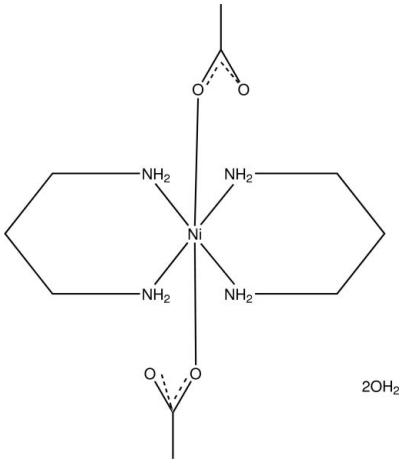
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.024; wR factor = 0.094; data-to-parameter ratio = 17.0.

In the title complex, $[Ni(CH_3COO)_2(C_3H_{10}N_2)_2]\cdot 2H_2O$, the Ni^{II} atom resides on a centre of symmetry and is in an octahedral coordination environment comprising four amino N atoms and two carboxylate O atoms. Intermolecular N—H···O and O—H···O hydrogen bonds produce $R_2^1(6)$, $R_2^2(12)$, $R_3^2(8)$ and $R_5^5(16)$ rings, which generate a two-dimensional polymeric network parallel to (001).

Related literature

For the graph-set analysis of hydrogen-bond patterns, see: Bernstein *et al.* (1995). For details of ring puckering analysis, see: Cremer & Pople (1975). For the effect of hydrogen bonding on the coordination in *trans*-di(salicylato)bis(1,3-diaminopropane-*N,N*')copper(II), see: Sundberg *et al.* (2001).



Experimental

Crystal data

$[Ni(C_2H_3O_2)_2(C_3H_{10}N_2)_2]\cdot 2H_2O$
M_r = 361.09
Triclinic, $P\overline{1}$
a = 6.6268 (3) Å
b = 7.8164 (3) Å
c = 8.9123 (4) Å
α = 73.933 (2)°
β = 80.797 (3)°
γ = 75.323 (2)°
V = 427.12 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.17 mm^{-1}
T = 296 K
0.32 × 0.18 × 0.13 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.775, T_{max} = 0.857
7230 measured reflections
2078 independent reflections
2021 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.094
S = 1.02
2078 reflections
122 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Selected bond lengths (Å).

N1—Ni1	2.1152 (13)	O1—Ni1	2.1031 (10)
N2—Ni1	2.1095 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···O1W	0.82 (3)	2.30 (3)	3.087 (2)	160 (2)
N2—H3···O2	0.82 (2)	2.43 (2)	3.0218 (19)	129.7 (19)
N1—H1···O2^{i}	0.87 (3)	2.51 (3)	3.290 (2)	150 (2)
N2—H4···O2^{ii}	0.83 (2)	2.26 (2)	3.092 (2)	177 (2)
O1W—H2W···O1^{iii}	0.79 (2)	2.02 (2)	2.7999 (18)	173 (2)
O1W—H1W···O2^{iv}	0.78 (2)	2.10 (2)	2.848 (2)	163 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2249).

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Diacetatobis(propane-1,3-diamine)nickel(II) dihydrate

Islam Ullah Khan, Ejaz, Onur Şahin and Orhan Büyükgüngör

S1. Comment

The 1,3-Diaminopropane (tn) ligand behaves as a strong chelator in its metal complexes due to the formation of a stable six-membered ring. At the same time, it is a good H-bond donor due to the existence of amino groups (Sundberg *et al.*, 2001). Herein, we report the synthesis and structure of the title compound.

The molecular structure and atom-numbering scheme are shown in Fig. 1. The compound crystallizes in the space group P-1 with Z′=1/2. The nickel(II) ion is located on a symmetry center, and is coordinated by two O atoms from two identical carboxylate groups and four N atoms from two 1,3-diaminopropane ligands. The geometry around the nickel(II) ion (Table 1) is that of a slightly distorted octahedron, of which the equatorial plane ($N1/N2/N1^{i}/N2^{i}$) is formed by four amino N atoms [symmetry code:(i) -x, 2-y, -z]. The axial positions in the octahedron are occupied by two carboxylate O atoms (O1 and $O1^{i}$). The 1,3-diaminopropane ligand shows chelating coordination behavior and displays a chair conformation [the puckering parameters (Cremer & Pople, 1975) are q_2 = 0.0467 (17)Å , q_3 = -0.5913 (18) Å, Q_T = 0.5930 (19) Å, φ = 349 (2)° and θ = 175.66 (16)°] in the equatorial direction.

The amino atom N1 in the molecule at (x, y, z) acts as a hydrogen-bond donor (Table 2) to atom $O2^{i}$ so forming a C(6) (Bernstein *et al.*, 1995) chain running parallel to the [-100] direction. Amino atom N2 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom $O2^{ii}$ so forming a C(6) chain running parallel to the [100] direction. The combination of N—H···O and O—H···O hydrogen bonds generates $R_2^1(6)$, $R_2^2(12)$, $R_3^2(8)$ and $R_5^5(16)$ rings parallel to the ab plane (Fig. 2).

S2. Experimental

Nickel(II) acetate (0.249 g, 1.0 mmol) was dissolved in methanol (25 ml). 1,3-diaminopropane(0.148 g, 2.0 mmol) were added and the mixture refluxed for 4 hours. The blue solution formed, which was filtered off, kept the filtrate for few days. Blue blocks were obtained from methanol.

S3. Refinement

All H atoms bound to C atoms were refined using a riding model, with C—H = 0.97Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene C atoms and C—H = 0.96Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl C atom. Water H atoms were located in difference maps and refined subject to a DFIX restraint of O—H = 0.83 (2) Å. Amino H atoms were located in difference maps and refined freely.

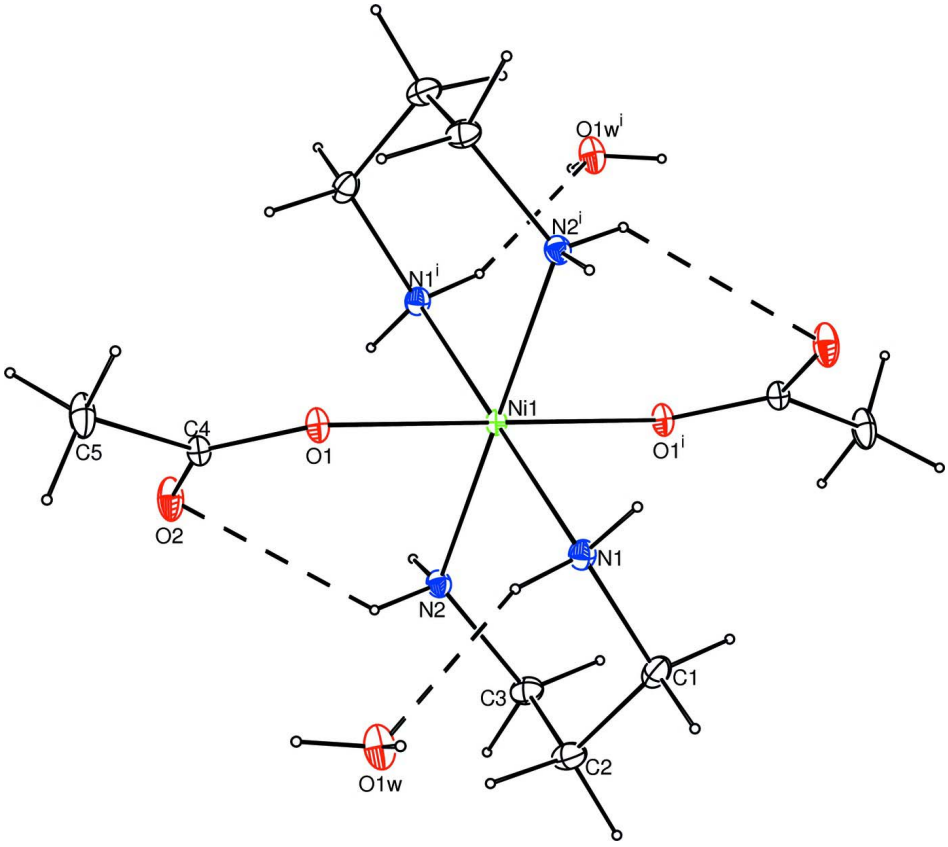

Figure 1

Molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intra- and intermolecular hydrogen bonds are shown as dashed lines. [Symmetry code: (i) -x, 2-y, -z.]

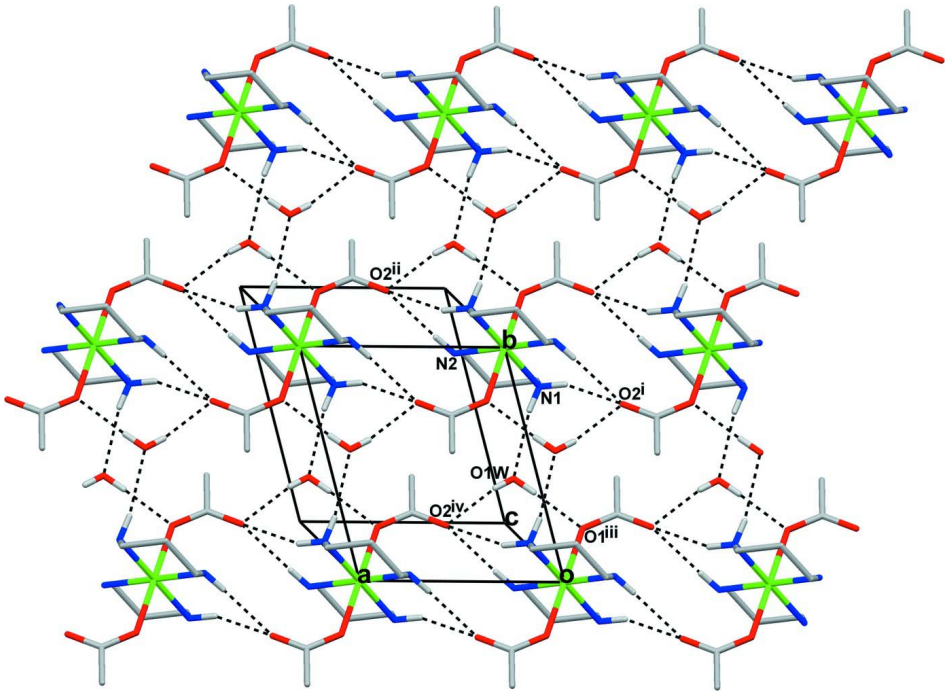

Figure 2

Perspective view of part of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from $R_2^1(6)$, $R_2^2(12)$, $R_3^2(8)$ and $R_5^5(16)$ rings.

Diacetatobis(propane-1,3-diamine)nickel(II) dihydrate

Crystal data

$[Ni(C_2H_3O_2)_2(C_3H_{10}N_2)_2]\cdot 2H_2O$
M_r = 361.09
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.6268 (3) Å
b = 7.8164 (3) Å
c = 8.9123 (4) Å
α = 73.933 (2)°
β = 80.797 (3)°
γ = 75.323 (2)°
V = 427.12 (3) Å^3
Z = 1
F(000) = 194
D_x = 1.404 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5325 reflections
θ = 2.8–28.3°
μ = 1.17 mm^{-1}
T = 296 K
Blocks, blue
0.32 × 0.18 × 0.13 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.775, T_{max} = 0.857
7230 measured reflections
2078 independent reflections
2021 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.3°, θ_{min} = 2.4°
$h = -6 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.094$
$S = 1.02$
2078 reflections
122 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0798P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ 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. 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_{iso}*/U_{eq}
C1	−0.1844 (3)	0.7809 (3)	0.3153 (2)	0.0399 (4)
H1A	−0.2579	0.6840	0.3658	0.048*
H1B	−0.2714	0.8939	0.3353	0.048*
C2	0.0196 (3)	0.7404 (3)	0.3857 (2)	0.0435 (4)
H2A	0.1101	0.6333	0.3572	0.052*
H2B	−0.0085	0.7106	0.4991	0.052*
C3	0.1366 (3)	0.8935 (3)	0.3368 (2)	0.0407 (4)
H3A	0.0437	1.0038	0.3579	0.049*
H3B	0.2537	0.8620	0.3995	0.049*
C4	0.3734 (2)	0.7432 (2)	−0.14011 (19)	0.0288 (3)
C5	0.4393 (3)	0.6191 (3)	−0.2513 (3)	0.0509 (5)
H5A	0.5557	0.6526	−0.3227	0.076*
H5B	0.3242	0.6311	−0.3094	0.076*
H5C	0.4798	0.4948	−0.1924	0.076*
N1	−0.1533 (2)	0.7972 (2)	0.14486 (17)	0.0314 (3)
H1	−0.274 (4)	0.816 (3)	0.110 (3)	0.047 (6)*
H2	−0.073 (4)	0.700 (4)	0.135 (3)	0.047 (6)*
N2	0.2158 (2)	0.9299 (2)	0.16906 (17)	0.0309 (3)
H3	0.293 (3)	0.835 (3)	0.152 (2)	0.036 (5)*
H4	0.293 (3)	1.004 (3)	0.145 (3)	0.038 (6)*
O1	0.17922 (17)	0.79863 (15)	−0.11179 (14)	0.0319 (3)
O2	0.5114 (2)	0.7828 (2)	−0.08414 (19)	0.0492 (4)
O1W	0.0788 (2)	0.3937 (2)	0.1862 (2)	0.0462 (3)
H1W	0.189 (3)	0.364 (4)	0.144 (3)	0.067 (8)*

H2W	0.001 (3)	0.339 (3)	0.173 (3)	0.052 (7)*
Ni1	0.0000	1.0000	0.0000	0.02287 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0449 (9)	0.0370 (8)	0.0357 (9)	−0.0139 (7)	0.0100 (7)	−0.0089 (7)
C2	0.0616 (12)	0.0382 (9)	0.0291 (8)	−0.0117 (8)	−0.0034 (8)	−0.0055 (7)
C3	0.0528 (10)	0.0434 (9)	0.0297 (8)	−0.0125 (8)	−0.0085 (7)	−0.0110 (7)
C4	0.0239 (7)	0.0277 (7)	0.0356 (8)	−0.0050 (5)	0.0005 (6)	−0.0117 (6)
C5	0.0316 (9)	0.0667 (13)	0.0661 (13)	−0.0010 (8)	−0.0004 (8)	−0.0463 (11)
N1	0.0296 (7)	0.0305 (7)	0.0354 (7)	−0.0107 (6)	0.0009 (5)	−0.0087 (5)
N2	0.0276 (6)	0.0339 (7)	0.0330 (7)	−0.0053 (6)	−0.0044 (5)	−0.0118 (6)
O1	0.0227 (5)	0.0329 (6)	0.0449 (7)	−0.0050 (4)	0.0010 (4)	−0.0206 (5)
O2	0.0255 (6)	0.0670 (9)	0.0704 (10)	−0.0107 (6)	0.0005 (5)	−0.0441 (8)
O1W	0.0329 (7)	0.0489 (8)	0.0668 (9)	−0.0088 (6)	−0.0035 (6)	−0.0316 (7)
Ni1	0.01974 (16)	0.02373 (17)	0.02636 (18)	−0.00491 (10)	0.00030 (10)	−0.00948 (11)

Geometric parameters (Å, º)

C1—N1	1.473 (2)	C5—H5B	0.9600
C1—C2	1.506 (3)	C5—H5C	0.9600
C1—H1A	0.9700	N1—Ni1	2.1152 (13)
C1—H1B	0.9700	N1—H1	0.87 (3)
C2—C3	1.515 (3)	N1—H2	0.82 (3)
C2—H2A	0.9700	N2—Ni1	2.1095 (14)
C2—H2B	0.9700	N2—H3	0.82 (2)
C3—N2	1.477 (2)	N2—H4	0.83 (2)
C3—H3A	0.9700	O1—Ni1	2.1031 (10)
C3—H3B	0.9700	O1W—H1W	0.777 (16)
C4—O2	1.2473 (19)	O1W—H2W	0.789 (15)
C4—O1	1.2572 (17)	Ni1—O1^{i}	2.1031 (10)
C4—C5	1.515 (2)	Ni1—N2^{i}	2.1095 (14)
C5—H5A	0.9600	Ni1—N1^{i}	2.1152 (13)
N1—C1—C2	112.28 (14)	C1—N1—H1	109.3 (15)
N1—C1—H1A	109.1	Ni1—N1—H1	106.9 (16)
C2—C1—H1A	109.1	C1—N1—H2	104.5 (16)
N1—C1—H1B	109.1	Ni1—N1—H2	103.9 (16)
C2—C1—H1B	109.1	H1—N1—H2	114 (2)
H1A—C1—H1B	107.9	C3—N2—Ni1	118.74 (11)
C1—C2—C3	115.24 (16)	C3—N2—H3	108.3 (15)
C1—C2—H2A	108.5	Ni1—N2—H3	102.3 (15)
C3—C2—H2A	108.5	C3—N2—H4	112.4 (15)
C1—C2—H2B	108.5	Ni1—N2—H4	109.5 (16)
C3—C2—H2B	108.5	H3—N2—H4	104 (2)
H2A—C2—H2B	107.5	C4—O1—Ni1	132.72 (10)
N2—C3—C2	112.68 (15)	H1W—O1W—H2W	110 (2)

N2—C3—H3A	109.1	O1^{i}—Ni1—O1	180.0
C2—C3—H3A	109.1	O1^{i}—Ni1—N2^{i}	91.58 (5)
N2—C3—H3B	109.1	O1—Ni1—N2^{i}	88.42 (5)
C2—C3—H3B	109.1	O1^{i}—Ni1—N2	88.42 (5)
H3A—C3—H3B	107.8	O1—Ni1—N2	91.58 (5)
O2—C4—O1	125.10 (14)	N2^{i}—Ni1—N2	180.0
O2—C4—C5	118.96 (15)	O1^{i}—Ni1—N1^{i}	87.27 (5)
O1—C4—C5	115.95 (14)	O1—Ni1—N1^{i}	92.73 (5)
C4—C5—H5A	109.5	N2^{i}—Ni1—N1^{i}	88.43 (6)
C4—C5—H5B	109.5	N2—Ni1—N1^{i}	91.57 (6)
H5A—C5—H5B	109.5	O1^{i}—Ni1—N1	92.73 (5)
C4—C5—H5C	109.5	O1—Ni1—N1	87.27 (5)
H5A—C5—H5C	109.5	N2^{i}—Ni1—N1	91.57 (6)
H5B—C5—H5C	109.5	N2—Ni1—N1	88.43 (6)
C1—N1—Ni1	118.49 (10)	N1^{i}—Ni1—N1	180.0

N1—C1—C2—C3	67.9 (2)	C4—O1—Ni1—N1	−127.02 (16)
C1—C2—C3—N2	−67.2 (2)	C3—N2—Ni1—O1^{i}	51.20 (13)
C2—C1—N1—Ni1	−59.62 (18)	C3—N2—Ni1—O1	−128.80 (13)
C2—C3—N2—Ni1	58.12 (19)	C3—N2—Ni1—N1^{i}	138.42 (13)
O2—C4—O1—Ni1	11.3 (3)	C3—N2—Ni1—N1	−41.58 (13)
C5—C4—O1—Ni1	−168.72 (14)	C1—N1—Ni1—O1^{i}	−46.03 (14)
C4—O1—Ni1—N2^{i}	141.34 (15)	C1—N1—Ni1—O1	133.97 (14)
C4—O1—Ni1—N2	−38.66 (15)	C1—N1—Ni1—N2^{i}	−137.69 (14)
C4—O1—Ni1—N1^{i}	52.98 (16)	C1—N1—Ni1—N2	42.31 (14)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···O1*W*	0.82 (3)	2.30 (3)	3.087 (2)	160 (2)
N2—H3···O2	0.82 (2)	2.43 (2)	3.0218 (19)	129.7 (19)
N1—H1···O2^{ii}	0.87 (3)	2.51 (3)	3.290 (2)	150 (2)
N2—H4···O2^{iii}	0.83 (2)	2.26 (2)	3.092 (2)	177 (2)
O1*W*—H2*W*···O1^{iv}	0.79 (2)	2.02 (2)	2.7999 (18)	173 (2)
O1*W*—H1*W*···O2^{v}	0.78 (2)	2.10 (2)	2.848 (2)	163 (3)

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