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Tetraaquabis[(1-carboxylatomethyl-1,3-benzimidazol-3-ium-3-yl)acetato-κO]palladium(II) dihydrate

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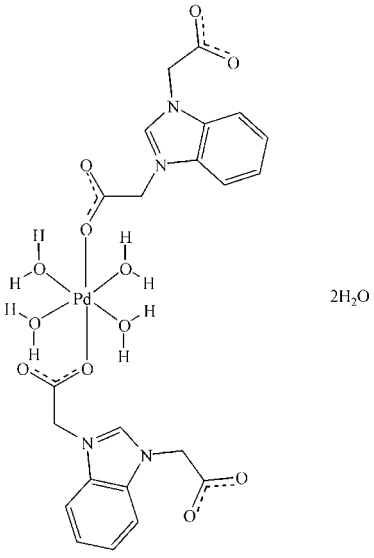
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.073; data-to-parameter ratio = 11.8.

In the title compound, $[Pd(C_{11}H_9N_2O_4)_2(H_2O)_4]\cdot 2H_2O$, the palladium(II) cation lies on an inversion centre and is hexacoordinated by two carboxylate O atoms from two (1-carboxylatomethyl-1,3-benzimidazol-3-ium-3-yl)acetate ligands and four water molecules, with a slightly distorted octahedral geometry. O—H···O hydrogen bonds link the molecules together.

Related literature

For uses of carboxylic acids in materials science, see: Church & Halvorson (1959). For uses in biological systems, see: Chung *et al.* (1971); Okabe & Oya (2000); Serre *et al.* (2005); Pocker & Fong (1980); Scapin *et al.* (1997); Kim *et al.* (2001).



$2H_2O$

Experimental

Crystal data

$[Pd(C_{11}H_9N_2O_4)_2(H_2O)_4]\cdot 2H_2O$
M_r = 680.90
Monoclinic, $P2_1/n$
a = 5.4702 (10) Å
b = 11.794 (2) Å
c = 20.886 (3) Å
β = 95.13 (3)°
V = 1342.1 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 293 (2) K
0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.733, T_{max} = 0.849
7075 measured reflections
2425 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.072
S = 1.00
2425 reflections
205 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H1W···O4^{i}	0.822 (10)	1.985 (14)	2.756 (3)	156 (3)
O7—H2W···O5^{i}	0.821 (10)	1.937 (10)	2.747 (3)	169 (3)

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2196).

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supporting information

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Tetraaquabis[(1-carboxylatomethyl-1,3-benzimidazol-3-ium-3-yl)acetato-*κO*]palladium(II) dihydrate

Lujiang Hao, Chunhua Mu and Ridong Wang

S1. Comment

In recent years, carboxylates have been widely used as polydentate ligands, which can coordinate to transition or rare earth ions yielding complexes with interesting properties that are useful in materials science (Church & Halvorson, 1959; Chung *et al.*, 1971) and in biological systems (Okabe & Oya, 2000; Serre *et al.*, 2005; Pocker & Fong, 1980; Scapin *et al.*, 1997). For example, Kim *et al.* (2001) focused on the syntheses of transition metal complexes containing benzene-carboxylate and rigid aromatic pyridine ligands in order to study their electronic conductivity and magnetic properties. The importance of transition metal dicarboxylate complexes motivated us to pursue synthetic strategies for these compounds, using sodium 1-carboxymethyl-1,3-benzimidazol-3-ium-3-acetate as a polydentate ligand. Here we report the synthesis and X-ray crystal structure analysis of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The palladium(II) cation lies on an inversion center and is hexacoordinated by two carboxylate oxygen atoms from two 1-carboxymethyl-1,3-benzimidazol-3-ium-3-acetato ligands and four water molecules, with a slightly distorted octahedral geometry. The Pd—O bond distances are in the range 2.2608 (19)–2.276 (2) Å. The packing involves hydrogen bonds, shown in Table 1 and Figure 2.

S2. Experimental

A mixture of palladium dichloride (0.5 mmol), imidazole (1.0 mmol), sodium 1-carboxymethyl-1,3-benzimidazol-3-ium-3-acetate (0.5 mmol), water (8 ml) and ethanol (8 ml) in a 25 ml Teflon-lined stainless steel autoclave was kept at 413 K for three days. Colorless crystals were obtained after cooling to room temperature with a yield of 27%. Anal. Calc. for $C_{22}H_{30}N_4O_{14}Pd$: C 38.77, H 4.41, N 8.22%; Found: C 38.68, H 4.37, N 8.14%.

S3. Refinement

The H atoms of the water molecule were located in a difference density map and were refined with distance restraints H···H = 1.38 (2) Å, O—H = 0.88 (2) Å, and with a fixed U_{iso} of 0.80 Å^2. All other H atoms were placed in calculated positions with a C—H bond distance of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

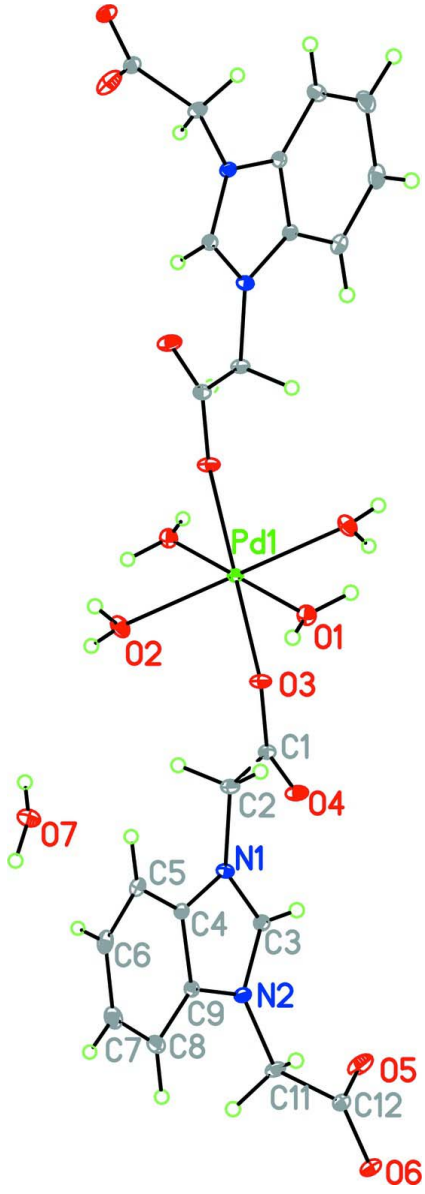

Figure 1

The molecular structure of (I), showing the atomic numbering scheme and 30% probability displacement ellipsoids. [Symmetry code for unlabelled atoms: 1-x, 2-y, -z.]

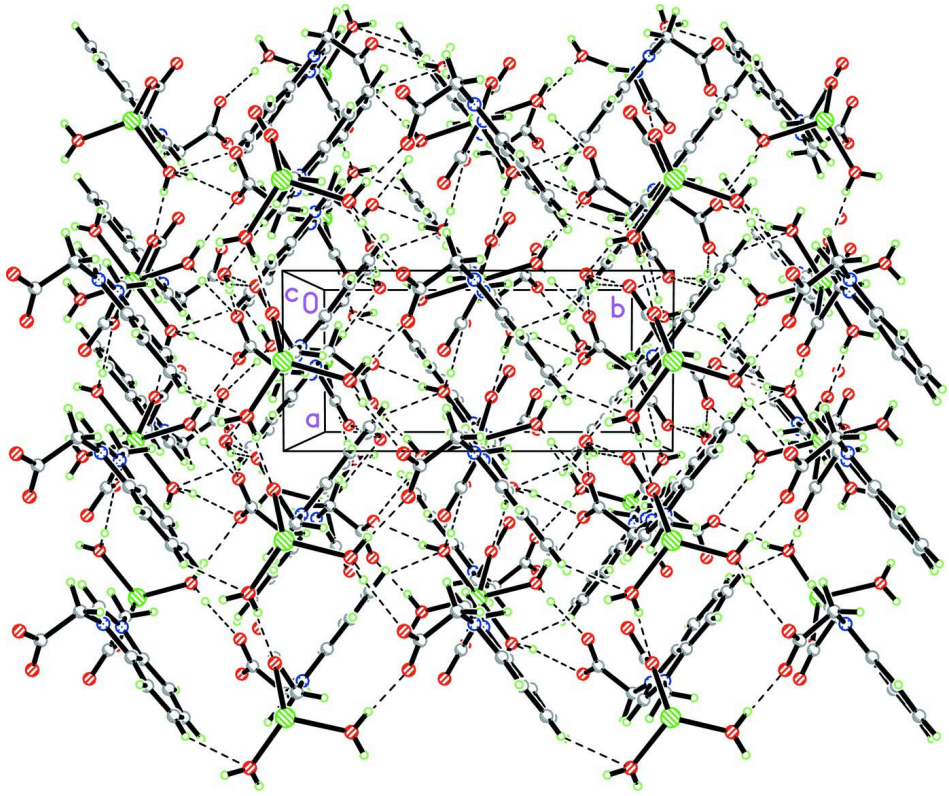

Figure 2

The packing of (I) with hydrogen bonds shown as dashed lines.

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Crystal data

$[Pd(C_{11}H_9N_2O_4)_2(H_2O)_4]\cdot 2H_2O$
$M_r = 680.90$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.4702\ (10)$ Å
$b = 11.794\ (2)$ Å
$c = 20.886\ (3)$ Å
$\beta = 95.13\ (3)°$
$V = 1342.1\ (4)$ Å^3
$Z = 2$
$F(000) = 696$
$D_x = 1.685$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2425 reflections
$\theta = 2.0–25.2°$
$\mu = 0.77$ mm^{-1}
$T = 293$ K
Block, colorless
0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.733$, $T_{max} = 0.849$
7075 measured reflections
2425 independent reflections
1958 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.0°$
$h = -6\rightarrow 6$
$k = -13\rightarrow 14$
$l = -24\rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.072$
$S = 1.00$
2425 reflections
205 parameters
9 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
$w = 1/[\sigma^2(F_o^2) + (0.042P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.5000	1.0000	0.0000	0.02449 (11)
C1	0.7711 (5)	1.0484 (2)	0.14648 (12)	0.0299 (6)
C2	0.5544 (5)	0.9924 (2)	0.17543 (12)	0.0326 (6)
H2A	0.4065	1.0354	0.1631	0.039*
H2B	0.5316	0.9166	0.1578	0.039*
C3	0.4539 (5)	1.0371 (2)	0.28620 (12)	0.0294 (6)
H3	0.3237	1.0858	0.2745	0.035*
C4	0.7676 (4)	0.9208 (2)	0.28035 (12)	0.0279 (6)
C5	0.9568 (5)	0.8536 (2)	0.26202 (14)	0.0365 (7)
H5	0.9834	0.8433	0.2190	0.044*
C6	1.1029 (6)	0.8030 (3)	0.31070 (17)	0.0480 (8)
H6	1.2346	0.7586	0.3006	0.058*
C7	1.0587 (6)	0.8166 (3)	0.37470 (17)	0.0540 (9)
H7	1.1595	0.7793	0.4062	0.065*
C8	0.8726 (6)	0.8828 (3)	0.39307 (15)	0.0449 (7)
H8	0.8444	0.8916	0.4360	0.054*
C9	0.7282 (5)	0.9361 (2)	0.34427 (12)	0.0299 (6)
C11	0.4355 (5)	1.0558 (3)	0.40327 (13)	0.0369 (7)
H11A	0.4276	0.9955	0.4346	0.044*
H11B	0.2696	1.0831	0.3923	0.044*
C12	0.5910 (5)	1.1527 (2)	0.43369 (13)	0.0347 (6)
H1W	0.545 (5)	0.681 (2)	0.2365 (7)	0.042*
H2W	0.571 (4)	0.693 (2)	0.1719 (9)	0.042*
H3W	0.160 (5)	1.1619 (14)	0.0196 (13)	0.042*

H4W	0.074 (4)	1.078 (2)	0.0568 (12)	0.042*
H5W	0.503 (3)	0.7896 (18)	0.0589 (13)	0.042*
H6W	0.270 (3)	0.799 (2)	0.0290 (13)	0.042*
N1	0.5889 (4)	0.98516 (17)	0.24523 (11)	0.0290 (5)
N2	0.5306 (4)	1.01001 (17)	0.34575 (11)	0.0301 (5)
O1	0.1897 (3)	1.09974 (17)	0.03739 (10)	0.0394 (5)
O2	0.3926 (4)	0.83292 (17)	0.04423 (10)	0.0451 (5)
O3	0.7682 (3)	1.03890 (18)	0.08641 (9)	0.0384 (5)
O4	0.9339 (4)	1.09448 (18)	0.18150 (9)	0.0460 (5)
O5	0.7726 (4)	1.1842 (2)	0.40763 (11)	0.0650 (7)
O6	0.5172 (3)	1.19231 (18)	0.48365 (9)	0.0439 (5)
O7	0.4821 (4)	0.7010 (2)	0.20114 (10)	0.0485 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02495 (17)	0.02873 (18)	0.01952 (16)	−0.00361 (11)	0.00047 (11)	0.00121 (11)
C1	0.0280 (14)	0.0368 (15)	0.0252 (15)	0.0003 (12)	0.0035 (11)	0.0001 (12)
C2	0.0323 (15)	0.0429 (17)	0.0225 (14)	−0.0058 (12)	0.0013 (12)	−0.0005 (11)
C3	0.0301 (14)	0.0302 (14)	0.0285 (15)	−0.0017 (11)	0.0057 (12)	−0.0005 (11)
C4	0.0297 (14)	0.0259 (14)	0.0280 (14)	−0.0057 (11)	0.0025 (11)	−0.0017 (11)
C5	0.0348 (15)	0.0316 (16)	0.0433 (18)	−0.0014 (12)	0.0053 (13)	−0.0082 (13)
C6	0.0394 (17)	0.0315 (17)	0.072 (2)	0.0073 (13)	−0.0003 (16)	−0.0058 (15)
C7	0.055 (2)	0.042 (2)	0.061 (2)	0.0064 (16)	−0.0148 (17)	0.0110 (16)
C8	0.0555 (19)	0.0411 (18)	0.0369 (17)	−0.0030 (15)	−0.0020 (14)	0.0067 (13)
C9	0.0336 (15)	0.0261 (15)	0.0295 (15)	−0.0037 (12)	0.0007 (11)	0.0001 (11)
C11	0.0397 (16)	0.0461 (18)	0.0264 (15)	−0.0038 (14)	0.0115 (12)	−0.0088 (13)
C12	0.0360 (15)	0.0362 (16)	0.0322 (16)	0.0023 (12)	0.0049 (12)	−0.0056 (12)
N1	0.0282 (12)	0.0365 (14)	0.0227 (12)	−0.0006 (9)	0.0046 (9)	0.0024 (9)
N2	0.0342 (13)	0.0327 (13)	0.0239 (12)	−0.0027 (9)	0.0058 (10)	−0.0037 (9)
O1	0.0368 (11)	0.0391 (12)	0.0445 (13)	−0.0023 (9)	0.0158 (9)	0.0021 (9)
O2	0.0349 (12)	0.0429 (13)	0.0558 (14)	−0.0055 (9)	−0.0058 (10)	0.0164 (10)
O3	0.0353 (11)	0.0585 (13)	0.0212 (11)	−0.0095 (9)	0.0023 (8)	−0.0004 (9)
O4	0.0425 (12)	0.0675 (15)	0.0279 (11)	−0.0240 (10)	0.0021 (9)	−0.0049 (9)
O5	0.0650 (16)	0.0750 (17)	0.0596 (15)	−0.0360 (13)	0.0314 (13)	−0.0339 (13)
O6	0.0475 (13)	0.0510 (14)	0.0339 (11)	0.0038 (9)	0.0067 (9)	−0.0152 (9)
O7	0.0458 (13)	0.0635 (15)	0.0366 (13)	0.0001 (11)	0.0067 (10)	0.0083 (11)

Geometric parameters (Å, º)

Pd1—O1	2.2608 (19)	C6—C7	1.389 (4)
Pd1—O1^{i}	2.2608 (19)	C6—H6	0.930
Pd1—O3	2.2687 (19)	C7—C8	1.364 (5)
Pd1—O3^{i}	2.2687 (19)	C7—H7	0.930
Pd1—O2^{i}	2.276 (2)	C8—C9	1.383 (4)
Pd1—O2	2.276 (2)	C8—H8	0.930
C1—O4	1.227 (3)	C9—N2	1.392 (3)
C1—O3	1.258 (3)	C11—N2	1.455 (3)

C1—C2	1.529 (4)	C11—C12	1.528 (4)
C2—N1	1.456 (3)	C11—H11A	0.970
C2—H2A	0.970	C11—H11B	0.970
C2—H2B	0.970	C12—O5	1.232 (3)
C3—N2	1.316 (4)	C12—O6	1.243 (3)
C3—N1	1.329 (3)	O1—H3W	0.832 (10)
C3—H3	0.930	O1—H4W	0.823 (10)
C4—C9	1.383 (3)	O2—H5W	0.828 (10)
C4—C5	1.384 (4)	O2—H6W	0.824 (10)
C4—N1	1.394 (3)	O7—H1W	0.822 (10)
C5—C6	1.373 (4)	O7—H2W	0.821 (10)
C5—H5	0.930		

O1—Pd1—O1^{i}	180.00 (9)	C5—C6—H6	119.2
O1—Pd1—O3	94.14 (7)	C7—C6—H6	119.2
O1^{i}—Pd1—O3	85.86 (8)	C8—C7—C6	122.4 (3)
O1—Pd1—O3^{i}	85.86 (8)	C8—C7—H7	118.8
O1^{i}—Pd1—O3^{i}	94.14 (7)	C6—C7—H7	118.8
O3—Pd1—O3^{i}	180.0	C7—C8—C9	116.3 (3)
O1—Pd1—O2^{i}	85.34 (7)	C7—C8—H8	121.8
O1^{i}—Pd1—O2^{i}	94.66 (7)	C9—C8—H8	121.8
O3—Pd1—O2^{i}	88.61 (7)	C8—C9—C4	121.6 (3)
O3^{i}—Pd1—O2^{i}	91.39 (7)	C8—C9—N2	131.4 (3)
O1—Pd1—O2	94.66 (7)	C4—C9—N2	106.9 (2)
O1^{i}—Pd1—O2	85.34 (7)	N2—C11—C12	113.2 (2)
O3—Pd1—O2	91.39 (7)	N2—C11—H11A	108.9
O3^{i}—Pd1—O2	88.61 (7)	C12—C11—H11A	108.9
O2^{i}—Pd1—O2	180.00 (10)	N2—C11—H11B	108.9
O4—C1—O3	125.3 (2)	C12—C11—H11B	108.9
O4—C1—C2	120.2 (2)	H11A—C11—H11B	107.7
O3—C1—C2	114.5 (2)	O5—C12—O6	126.3 (3)
N1—C2—C1	112.7 (2)	O5—C12—C11	118.9 (2)
N1—C2—H2A	109.1	O6—C12—C11	114.8 (2)
C1—C2—H2A	109.1	C3—N1—C4	108.4 (2)
N1—C2—H2B	109.1	C3—N1—C2	126.0 (2)
C1—C2—H2B	109.1	C4—N1—C2	125.5 (2)
H2A—C2—H2B	107.8	C3—N2—C9	108.3 (2)
N2—C3—N1	110.3 (2)	C3—N2—C11	125.6 (2)
N2—C3—H3	124.8	C9—N2—C11	125.9 (2)
N1—C3—H3	124.8	Pd1—O1—H3W	115.2 (18)
C9—C4—C5	121.8 (2)	Pd1—O1—H4W	130.4 (18)
C9—C4—N1	106.0 (2)	H3W—O1—H4W	111 (2)
C5—C4—N1	132.3 (2)	Pd1—O2—H5W	118.6 (19)
C6—C5—C4	116.4 (3)	Pd1—O2—H6W	120.1 (19)
C6—C5—H5	121.8	H5W—O2—H6W	112 (2)

C4—C5—H5	121.8	C1—O3—Pd1	139.53 (17)
C5—C6—C7	121.5 (3)	H1W—O7—H2W	114 (2)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H1*W*···O4^{ii}	0.82 (1)	1.99 (1)	2.756 (3)	156 (3)
O7—H2*W*···O5^{ii}	0.82 (1)	1.94 (1)	2.747 (3)	169 (3)

Symmetry code: (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.