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Ethyl α-D-sorboside monohydrate

Tomoko Takashita,[a]* Yoji Horii,[a] Kei Takeshita[b] and Tomohiko Ishii[a]

[a]Graduate School of Science for Creative Emergence, Kagawa University, 2217-20 Hayashi-cho, Takamatsu, Kagawa 761-0396, Japan, and [b]FUSHIMI Pharmaceutical Co., Ltd., 307 Minatomachi, Marugame, Kagawa 763-8605, Japan. *Correspondence e-mail: s25d154@kagawa-u.ac.jp

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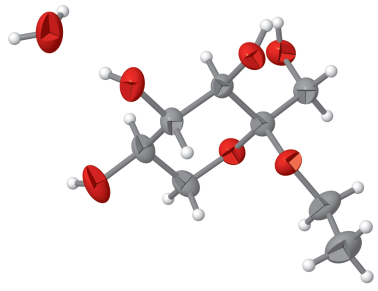
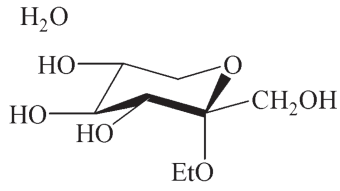
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_{16}O_6\cdot H_2O$, was prepared by Fischer glycosylation of D-sorbose with ethanol. Colorless block-shaped single crystals suitable for single-crystal X-ray diffraction were obtained. The title compound crystallizes in the orthorhombic space group $P2_12_12_1$, with one sorboside molecule and one water molecule in the asymmetric unit. The sorboside molecule adopts an α-pyranose form with a $^5C_2$ chair conformation. In the crystal, the sorboside and water molecules are linked by O—H···O hydrogen bonds, forming an extended hydrogen-bonded network.

3D view



Chemical scheme



Structure description

Rare sugars are monosaccharides and their derivatives that occur only in limited quantities in nature (Izumori, 2002). D-Sorbose is a rare ketohexose, and its alkyl glycosides provide useful examples for examining how substitution at the anomeric centre affects molecular conformation, hydration and crystal packing. The title compound, ethyl α-D-sorboside monohydrate, is an ethyl glycoside of D-sorbose in which the anomeric hydroxy group at C2 is replaced by an ethoxy group. In the present study, its single-crystal structure was determined and compared with the previously reported anhydrous structure of ethyl α-L-sorboside (Nagayama *et al.*, 2020).

The title compound crystallizes in the orthorhombic space group $P2_12_12_1$. The asymmetric unit contains one ethyl α-D-sorboside molecule and one water molecule. The sorboside molecule adopts an α-pyranose form with a $^5C_2$ chair conformation and the ethoxy substituent at the anomeric C2 atom occupying an axial position (Fig. 1). The stereogenic centres C2, C3, C4 and C5 have *S*, *R*, *S* and *R* configurations, respectively. The C2—O2—C7—C8 torsion angle is −173.8 (2)°.

The anhydrous crystal structure of ethyl α-L-sorboside (CSD refcode EJAKAE; Nagayama *et al.*, 2020) also belongs to space group $P2_12_12_1$, with a unit-cell volume of 940.63 (19) Å^3 and Z = 4. The unit-cell volume of the present monohydrate is therefore 151.35 Å^3 (16.1%) larger. This difference is consistent with the inclusion of four water

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.82	2.36	3.018 (2)	137
O1—H1···O4^{i}	0.82	2.25	2.975 (3)	148
O3—H3···O4^{i}	0.82	1.93	2.741 (2)	167
O4—H4···O7	0.82	1.82	2.618 (3)	165
O5—H5···O3^{ii}	0.82	2.08	2.842 (3)	154
O7—H7*C*···O5^{iii}	0.85	1.90	2.738 (3)	168
O7—H7*D*···O1^{iv}	0.85	2.09	2.909 (3)	163

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

molecules of crystallization per unit cell, together with the resulting change in crystal packing. After inversion of the L-sorboside molecule, least-squares fitting of all 14 non-hydrogen atoms to the corresponding atoms of the present D-sorboside molecule gave an r.m.s. deviation of 0.441 Å The principal conformational difference is the orientation of the hydroxymethyl group at C1, as indicated by the O1—C1—C2—C3 torsion angles of −1257.1 (3)° in the title monohydrate and −153.2 (2)° in the inverted EJAKAE molecule. By contrast, the sorbopyranose ring conformations and the *anti* conformations of the ethoxy groups are closely similar.

In the crystal, the sorboside and water molecules are connected by the O1—H1···O3, O1—H1···O4, O3—H3···O4, O4—H4···O7, O5—H5···O3, O7—H7C···O5 and O7—H7D···O1 hydrogen bonds listed in Table 1. The O1—H1 group participates in a bifurcated hydrogen bond, with the O3 and O4 atoms acting as acceptors. The donor···acceptor distances range from 2.618 (3) to 3.018 (2) Å. Taken together, the seven O—H···O hydrogen bonds generate a two-dimensional hydrogen-bonded network extending parallel to the (001) plane. The crystal packing and all seven hydrogen bonds listed in Table 1 are shown in Fig. 2.

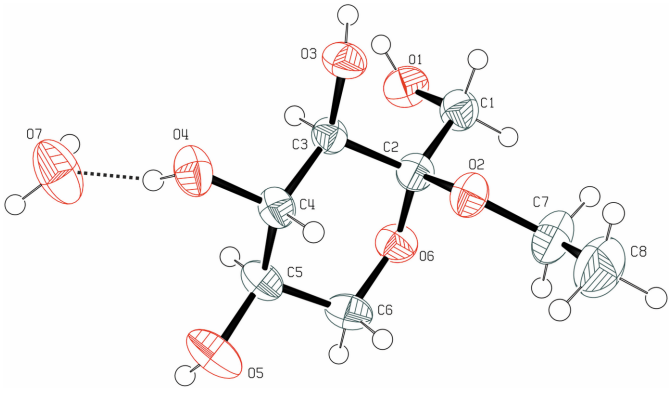


Figure 1
The molecular structure of the asymmetric unit of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius. The O4—H4···O7 hydrogen bond between the sorboside and water molecules is shown as a dashed line.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{16}O_6·H_2O$
M_r	226.22
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	6.6457 (2), 7.5616 (2), 21.7300 (6)
V (Å^3)	1091.98 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.05
Crystal size (mm)	0.1 × 0.1 × 0.1
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min}, T_{max}	0.702, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12255, 1969, 1706
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.076, 1.02
No. of reflections	1969
No. of parameters	144
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.13
Absolute structure	Flack x determined using 626 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.04 (11)

Computer programs: *RAPID-AUTO* (Rigaku, 2009), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

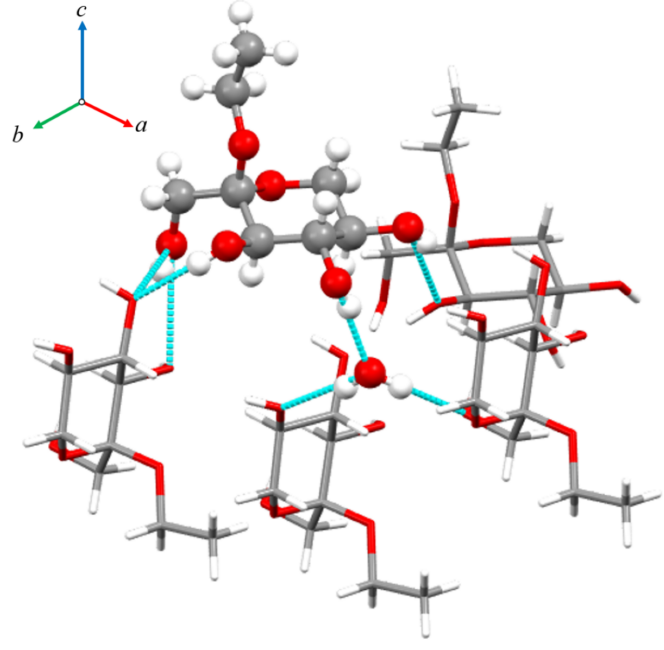


Figure 2
A portion of the crystal packing of the title compound, shown in an oblique view with the unit-cell directions indicated. The central sorboside molecule and the water molecule are shown using a ball-and-stick representation, whereas the surrounding sorboside molecules are shown using a capped-stick representation. The seven crystallographically distinct O—H···O hydrogen bonds listed in Table 1 are shown as cyan dashed lines.

Synthesis and crystallization

Ethyl α-D-sorboside monohydrate was prepared by Fischer glycosylation of D-sorbose with ethanol. Because the reaction produced a mixture of isomeric products, including α- and β-anomers and furanose forms, the reaction mixture was separated by ion-exchange chromatography. Fractions containing the desired product were combined and concentrated to give a syrup, which was allowed to stand at room temperature. Colorless block-shaped single crystals suitable for single-crystal X-ray diffraction were obtained. The absolute configuration was assigned on the basis of the known configuration of the D-sorbose starting material and the synthetic route.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The Flack parameter supports the absolute configuration expected from the use of D-sorbose as the starting material.

Acknowledgements

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References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Izumori, K. (2002). *Naturwissenschaften* **89**, 120–124.
Nagayama, N., Taniguchi, N., Matsumoto, M., Takeshita, K. & Ishii, T. (2020). *IUCrData* **5**, x201625.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Rigaku (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2009). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2026). **11**, x260804 [https://doi.org/10.1107/S2414314626008047]

Ethyl α-D-sorboside monohydrate

Tomoko Takashita, Yoji Horii, Kei Takeshita and Tomohiko Ishii

Ethyl *α*-*D*-sorboside monohydrate

Crystal data

$C_8H_{16}O_6 \cdot H_2O$
$M_r = 226.22$
Orthorhombic, $P2_12_12_1$
$a = 6.6457$ (2) Å
$b = 7.5616$ (2) Å
$c = 21.7300$ (6) Å
$V = 1091.98$ (5) Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.376$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 11105 reflections
$\theta = 4.1$–$68.1°$
$\mu = 1.05$ mm^{-1}
$T = 296$ K
Block, clear light colourless
0.1 × 0.1 × 0.1 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
$T_{min} = 0.702$, $T_{max} = 1.000$
12255 measured reflections
1969 independent reflections
1706 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 68.1°$, $\theta_{min} = 4.1°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -26 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.076$
$S = 1.02$
1969 reflections
144 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 0.1352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Absolute structure: Flack *x* determined using 626 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.04 (11)

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. The structure was solved using SHELXT (Sheldrick, 2015*a*) and refined against F^2 by full-matrix least-squares methods using *SHELXL* (Sheldrick, 2015*b*). All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions and refined using constrained models. The final refinement gave $R_1 = 0.0311$ for reflections with $I > 2\sigma(I)$ and $wR_2 = 0.0759$ for all data. The maximum and minimum residual electron densities were 0.15 and -0.13 e Å^{-3}, respectively.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5536 (3)	0.9834 (2)	0.34665 (8)	0.0487 (5)
H1	0.502283	0.981931	0.312334	0.073*
O2	0.4803 (2)	0.5405 (2)	0.41294 (7)	0.0409 (4)
O3	0.4025 (2)	0.5562 (2)	0.28922 (7)	0.0392 (4)
H3	0.348364	0.643371	0.274062	0.059*
O4	0.7416 (3)	0.3772 (2)	0.24941 (7)	0.0443 (5)
H4	0.840422	0.417639	0.231721	0.066*
O5	1.0724 (3)	0.3576 (2)	0.33542 (10)	0.0572 (5)
H5	1.187162	0.394017	0.329875	0.086*
O6	0.7715 (3)	0.7138 (2)	0.40269 (7)	0.0409 (4)
C1	0.4643 (4)	0.8509 (3)	0.38368 (11)	0.0418 (6)
H1A	0.326162	0.833089	0.370681	0.050*
H1B	0.462452	0.890214	0.426162	0.050*
C2	0.5773 (4)	0.6760 (3)	0.37952 (10)	0.0350 (5)
C3	0.5945 (3)	0.6048 (3)	0.31342 (10)	0.0314 (5)
H3A	0.651070	0.698141	0.287380	0.038*
C4	0.7331 (4)	0.4455 (3)	0.31061 (10)	0.0350 (5)
H4A	0.679010	0.353462	0.337700	0.042*
C5	0.9361 (4)	0.5024 (3)	0.33518 (11)	0.0390 (6)
H5A	0.990207	0.597101	0.309203	0.047*
C6	0.9110 (4)	0.5700 (4)	0.40010 (11)	0.0455 (6)
H6A	0.863529	0.474734	0.426218	0.055*
H6B	1.040394	0.608870	0.415706	0.055*
C7	0.4558 (5)	0.5640 (4)	0.47787 (11)	0.0590 (8)
H7A	0.365283	0.661921	0.485898	0.071*
H7B	0.584626	0.590126	0.496772	0.071*
C8	0.3727 (6)	0.4001 (5)	0.50366 (15)	0.0775 (11)
H8A	0.243423	0.377111	0.485613	0.116*
H8B	0.358345	0.412393	0.547409	0.116*
H8C	0.461880	0.303491	0.494816	0.116*
O7	1.0151 (3)	0.5050 (3)	0.17499 (12)	0.0711 (7)
H7C	1.005187	0.615896	0.169385	0.107*
H7D	1.134246	0.479972	0.163735	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0558 (12)	0.0388 (10)	0.0513 (11)	−0.0007 (9)	−0.0042 (9)	0.0038 (8)
O2	0.0448 (10)	0.0422 (9)	0.0355 (8)	−0.0050 (8)	0.0050 (7)	0.0031 (8)

O3	0.0291 (9)	0.0433 (10)	0.0452 (10)	0.0004 (9)	−0.0072 (7)	−0.0025 (8)
O4	0.0376 (10)	0.0476 (10)	0.0477 (10)	−0.0057 (9)	0.0075 (8)	−0.0148 (8)
O5	0.0296 (10)	0.0428 (10)	0.0992 (15)	0.0029 (9)	0.0004 (10)	−0.0055 (10)
O6	0.0369 (9)	0.0417 (10)	0.0440 (9)	−0.0032 (8)	−0.0075 (8)	−0.0067 (7)
C1	0.0453 (15)	0.0396 (14)	0.0405 (13)	0.0025 (12)	0.0016 (11)	−0.0047 (11)
C2	0.0331 (13)	0.0361 (13)	0.0359 (12)	−0.0046 (11)	−0.0009 (11)	−0.0009 (10)
C3	0.0276 (12)	0.0334 (12)	0.0332 (11)	−0.0029 (11)	0.0009 (9)	−0.0002 (9)
C4	0.0311 (12)	0.0350 (13)	0.0389 (13)	−0.0015 (12)	0.0031 (10)	−0.0042 (11)
C5	0.0275 (13)	0.0353 (14)	0.0543 (14)	−0.0002 (10)	0.0006 (11)	−0.0022 (11)
C6	0.0329 (13)	0.0487 (15)	0.0549 (15)	0.0018 (13)	−0.0119 (11)	−0.0002 (13)
C7	0.070 (2)	0.072 (2)	0.0348 (13)	−0.0081 (19)	0.0069 (13)	0.0024 (14)
C8	0.091 (3)	0.082 (2)	0.060 (2)	0.002 (2)	0.0174 (18)	0.0237 (18)
O7	0.0619 (14)	0.0485 (13)	0.1028 (17)	0.0016 (10)	0.0380 (12)	0.0090 (11)

Geometric parameters (Å, º)

O1—H1	0.8200	C3—H3A	0.9800
O1—C1	1.415 (3)	C3—C4	1.517 (3)
O2—C2	1.412 (3)	C4—H4A	0.9800
O2—C7	1.431 (3)	C4—C5	1.514 (3)
O3—H3	0.8200	C5—H5A	0.9800
O3—C3	1.428 (3)	C5—C6	1.510 (3)
O4—H4	0.8200	C6—H6A	0.9700
O4—C4	1.428 (3)	C6—H6B	0.9700
O5—H5	0.8200	C7—H7A	0.9700
O5—C5	1.421 (3)	C7—H7B	0.9700
O6—C2	1.414 (3)	C7—C8	1.468 (4)
O6—C6	1.430 (3)	C8—H8A	0.9600
C1—H1A	0.9700	C8—H8B	0.9600
C1—H1B	0.9700	C8—H8C	0.9600
C1—C2	1.524 (3)	O7—H7C	0.8501
C2—C3	1.538 (3)	O7—H7D	0.8498
C1—O1—H1	109.5	C5—C4—C3	107.55 (19)
C2—O2—C7	118.0 (2)	C5—C4—H4A	108.5
C3—O3—H3	109.5	O5—C5—C4	110.50 (18)
C4—O4—H4	109.5	O5—C5—H5A	109.4
C5—O5—H5	109.5	O5—C5—C6	109.2 (2)
C2—O6—C6	115.07 (18)	C4—C5—H5A	109.4
O1—C1—H1A	109.2	C6—C5—C4	109.10 (19)
O1—C1—H1B	109.2	C6—C5—H5A	109.4
O1—C1—C2	112.0 (2)	O6—C6—C5	111.47 (19)
H1A—C1—H1B	107.9	O6—C6—H6A	109.3
C2—C1—H1A	109.2	O6—C6—H6B	109.3
C2—C1—H1B	109.2	C5—C6—H6A	109.3
O2—C2—O6	112.37 (18)	C5—C6—H6B	109.3
O2—C2—C1	111.97 (18)	H6A—C6—H6B	108.0
O2—C2—C3	105.08 (18)	O2—C7—H7A	110.0

O6—C2—C1	104.65 (19)	O2—C7—H7B	110.0
O6—C2—C3	109.62 (19)	O2—C7—C8	108.3 (3)
C1—C2—C3	113.31 (19)	H7A—C7—H7B	108.4
O3—C3—C2	111.58 (18)	C8—C7—H7A	110.0
O3—C3—H3A	108.4	C8—C7—H7B	110.0
O3—C3—C4	108.88 (18)	C7—C8—H8A	109.5
C2—C3—H3A	108.4	C7—C8—H8B	109.5
C4—C3—C2	111.12 (18)	C7—C8—H8C	109.5
C4—C3—H3A	108.4	H8A—C8—H8B	109.5
O4—C4—C3	110.43 (18)	H8A—C8—H8C	109.5
O4—C4—H4A	108.5	H8B—C8—H8C	109.5
O4—C4—C5	113.33 (19)	H7C—O7—H7D	104.5
C3—C4—H4A	108.5		

O1—C1—C2—O2	−175.77 (18)	C2—O2—C7—C8	−173.8 (2)
O1—C1—C2—O6	62.2 (2)	C2—O6—C6—C5	57.3 (3)
O1—C1—C2—C3	−57.1 (3)	C2—C3—C4—O4	177.90 (18)
O2—C2—C3—O3	56.1 (2)	C2—C3—C4—C5	−58.0 (2)
O2—C2—C3—C4	−65.6 (2)	C3—C4—C5—O5	178.40 (18)
O3—C3—C4—O4	54.6 (2)	C3—C4—C5—C6	58.4 (2)
O3—C3—C4—C5	178.73 (18)	C4—C5—C6—O6	−57.9 (3)
O4—C4—C5—O5	−59.3 (3)	C6—O6—C2—O2	61.8 (2)
O4—C4—C5—C6	−179.3 (2)	C6—O6—C2—C1	−176.52 (19)
O5—C5—C6—O6	−178.70 (19)	C6—O6—C2—C3	−54.7 (2)
O6—C2—C3—O3	177.04 (18)	C7—O2—C2—O6	56.5 (3)
O6—C2—C3—C4	55.3 (2)	C7—O2—C2—C1	−61.0 (3)
C1—C2—C3—O3	−66.5 (2)	C7—O2—C2—C3	175.6 (2)
C1—C2—C3—C4	171.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.82	2.36	3.018 (2)	137
O1—H1···O4^{i}	0.82	2.25	2.975 (3)	148
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O5—H5···O3^{ii}	0.82	2.08	2.842 (3)	154
O7—H7*C*···O5^{iii}	0.85	1.90	2.738 (3)	168
O7—H7*D*···O1^{iv}	0.85	2.09	2.909 (3)	163

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