Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Deoxy-L-mannitol (6-deoxy-L-mannitol or L-rhamnitol)

Sarah F. Jenkinson,[a] K. Victoria Booth,[a]* Pushpakiran Gullapalli,[b] Kenji Morimoto,[b] Ken Izumori,[b] George W. J. Fleet[a] and David J. Watkin[c]

[a]Department of Organic Chemistry, Chemistry Research Laboratory, University of Oxford, Mansfield Road, Oxford OX1 3TA, England, [b]Rare Sugar Research Centre, Kagawa University, 2393 Miki-cho, Kita-gun, Kagawa 761-0795, Japan, and [c]Department of Chemical Crystallography, Chemistry Research Laboratory, University of Oxford, Mansfield Road, Oxford OX1 3TA, England
Correspondence e-mail: victoria.booth@chem.ox.ac.uk

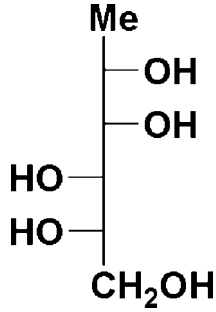
Received 25 July 2008; accepted 31 July 2008

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.072; data-to-parameter ratio = 10.3.

The crystalline form of 1-deoxy-L-mannitol, $C_6H_{14}O_5$, exists as an extensively hydrogen-bonded structure with each molecule acting as a donor and acceptor for five hydrogen bonds. There are no unusual crystal-packing features; the absolute configuration was determined from the use of 6-deoxy-L-mannose (L-rhamnose) as the starting material.

Related literature

For related literature see: Jenkinson *et al.* (2008); Gullapalli *et al.* (2007); Izumori (2002, 2006); Granstrom *et al.* (2004); Beadle *et al.* (1992); Skytte (2002); Sui *et al.* (2005); Levin (2002); Howling & Callagan (2000); Bertelsen *et al.* (1999); Takata *et al.* (2005); Menavuvu *et al.* (2006); Hossain *et al.* (2006); Donner *et al.* (1999).



Experimental

Crystal data

$C_6H_{14}O_5$
M_r = 166.17
Orthorhombic, $P2_12_12_1$
a = 7.3650 (3) Å
b = 7.6272 (3) Å
c = 13.7676 (5) Å
V = 773.39 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 150 K
0.40 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.89, T_{max} = 0.99
5170 measured reflections
1033 independent reflections
974 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.072
S = 0.97
1033 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O10—H1···O1^{i}	0.85	1.98	2.782 (2)	158
O4—H2···O6^{ii}	0.87	1.92	2.779 (2)	168
O8—H3···O4^{ii}	0.84	1.97	2.742 (2)	152
O6—H4···O10^{iii}	0.87	1.92	2.772 (2)	165
O1—H5···O8^{i}	0.87	1.84	2.704 (2)	173

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

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

This work was supported in part by the Program for Promotion of Basic Research Activities for Innovative Biosciences (PROBRAIN). We also thank the Oxford University Chemical Crystallography service for use of the instruments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2670).

References

Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.
Beadle, J. R., Saunders, J. P. & Wajda, T. J. (1992). US Patent No. 5 078 796.
Bertelsen, H., Jensen, B. B. & Buemann, B. (1999). *World Rev. Nutr. Diet.* **85**, 98–109.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Donner, T. W., Wilber, J. F. & Ostrowski, D. (1999). *Diab. Obes. Metab.* **1**, 285–291.
Granstrom, T. B., Takata, G., Tokuda, M. & Izumori, K. (2004). *J. Biosci. Bioeng.* **97**, 89–94.
Gullapalli, P., Shiji, T., Rao, D., Yoshihara, A., Morimoto, K., Takata, G., Fleet, G. W. J. & Izumori, K. (2007). *Tetrahedron Asymmetry*, **18**, 1995–2000.
Hossain, M. A., Wakabayashi, H., Izuishi, K., Okano, K., Yachida, S., Tokuda, M., Izumori, K. & Maeta, H. (2006). *J. Biosci. Bioeng.* **101**, 369–371.
Howling, D. & Callagan, J. L. (2000). PCT Int. App. WO 2 000 042 865.
Izumori, K. (2002). *Naturwissenschaften*, **89**, 120–124.
Izumori, K. (2006). *J. Biotech.* **124**, 717–722.

Jenkinson, S. F., Booth, K. V., Yoshihara, A., Morimoto, K., Fleet, G. W. J., Izumori, K. & Watkin, D. J. (2008). *Acta Cryst.* E**64**, o1429.
Levin, G. V. (2002). *J. Med. Food*, **5**, 23–36.
Menavuvu, B. T., Poonperm, W., Leang, K., Noguchi, N., Okada, H., Morimoto, K., Granstrom, T. B., Takada, G. & Izumori, K. (2006). *J. Biosci. Bioeng.* **101**, 340–345.
Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Skytte, U. P. (2002). *Cereal Foods World*, **47**, 224–224.
Sui, L., Dong, Y. Y., Watanabe, Y., Yamaguchi, F., Hatano, N., Tsukamoto, I., Izumori, K. & Tokuda, M. (2005). *Intl. J. Ocol.* **27**, 907–912.
Takata, M. K., Yamaguchi, F., Nakanose, Y., Watanabe, Y., Hatano, N., Tsukamoto, I., Nagata, M., Izumori, K. & Tokuda, M. (2005). *J. Biosci. Bioeng.* **100**, 511–516.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

supporting information

Acta Cryst. (2008). E**64**, o1705–o1706 [doi:10.1107/S1600536808024586]

1-Deoxy-L-mannitol (6-deoxy-L-mannitol or L-rhamnitol)

Sarah F. Jenkinson, K. Victoria Booth, Pushpakiran Gullapalli, Kenji Morimoto, Ken Izumori, George W. J. Fleet and David J. Watkin

S1. Comment

The properties of 1-deoxy ketohexose sugars have been little studied. The crystal structure of 6-deoxy-L-galactitol has recently been published (Jenkinson *et al.*, 2008) and herein we report the crystal structure of a similar deoxy polyol, 1-deoxy-L-mannitol an intermediate in the synthesis of 1-deoxy-L-fructose, **3** (Fig. 1) (Gullapalli *et al.*, 2007).

The demand for the large scale production of rare sugars by biotechnological (Izumori, 2006; Izumori, 2002; Granstrom *et al.*, 2004) and chemical (Beadle *et al.*, 1992) methods is driven by the demand for alternative foodstuffs (Skytte, 2002) and D-tagatose itself is used as a low calorie sweetener (Levin, 2002; Howling & Callagan, 2000; Bertelsen *et al.* 1999). Rare monosaccharides themselves, however, have been found to demonstrate interesting pharmaceutical properties, for example, D-psicose (Takata *et al.*, 2005; Menavuvu *et al.*, 2006) and D-allose (Sui *et al.*, 2005; Hossain *et al.*, 2006) have significant chemotherapeutic properties and D-tagatose has been found to be an anti-hyperglycemic agent (Donner *et al.*, 1999) and therefore potentially useful in the treatment of diabetes.

1-Deoxy-L-mannitol **2** (Fig. 2) was prepared from the reduction by catalytic hydrogenation of 6-deoxy-L-mannose **1** (L-rhamnose). The X-ray structure shows that the crystal exists as an extensively hydrogen bonded lattice with each molecule acting as a donor and an acceptor for 5 hydrogen bonds (Fig.3).

S2. Experimental

1-Deoxy-L-mannitol was recrystallized from methanol: m.p. 390K, $[\alpha]_D^{20}$ +1.4 (*c*, 1.4 in H_2O).

S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was determined from the starting material.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

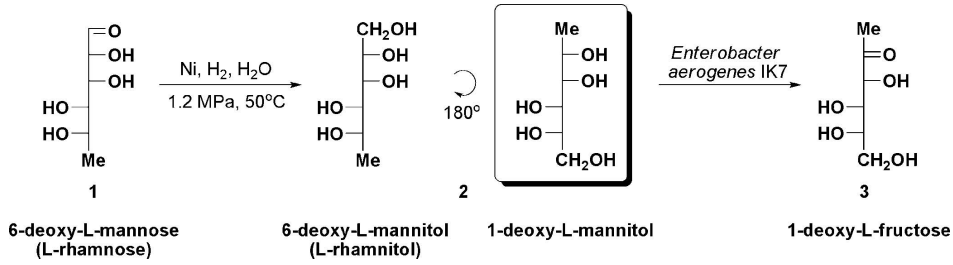


Figure 1

Synthetic scheme.

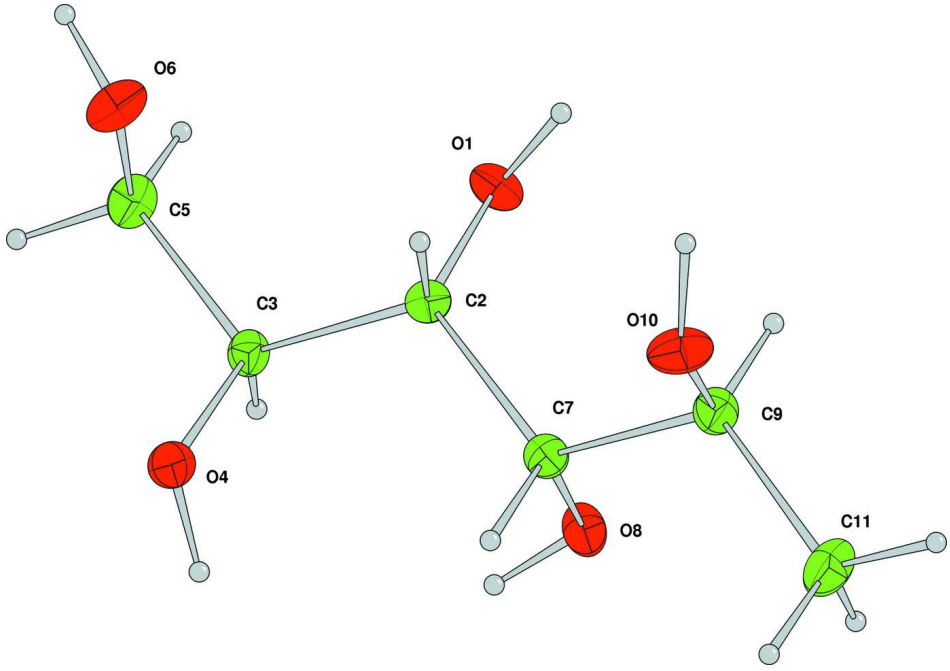


Figure 2

The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

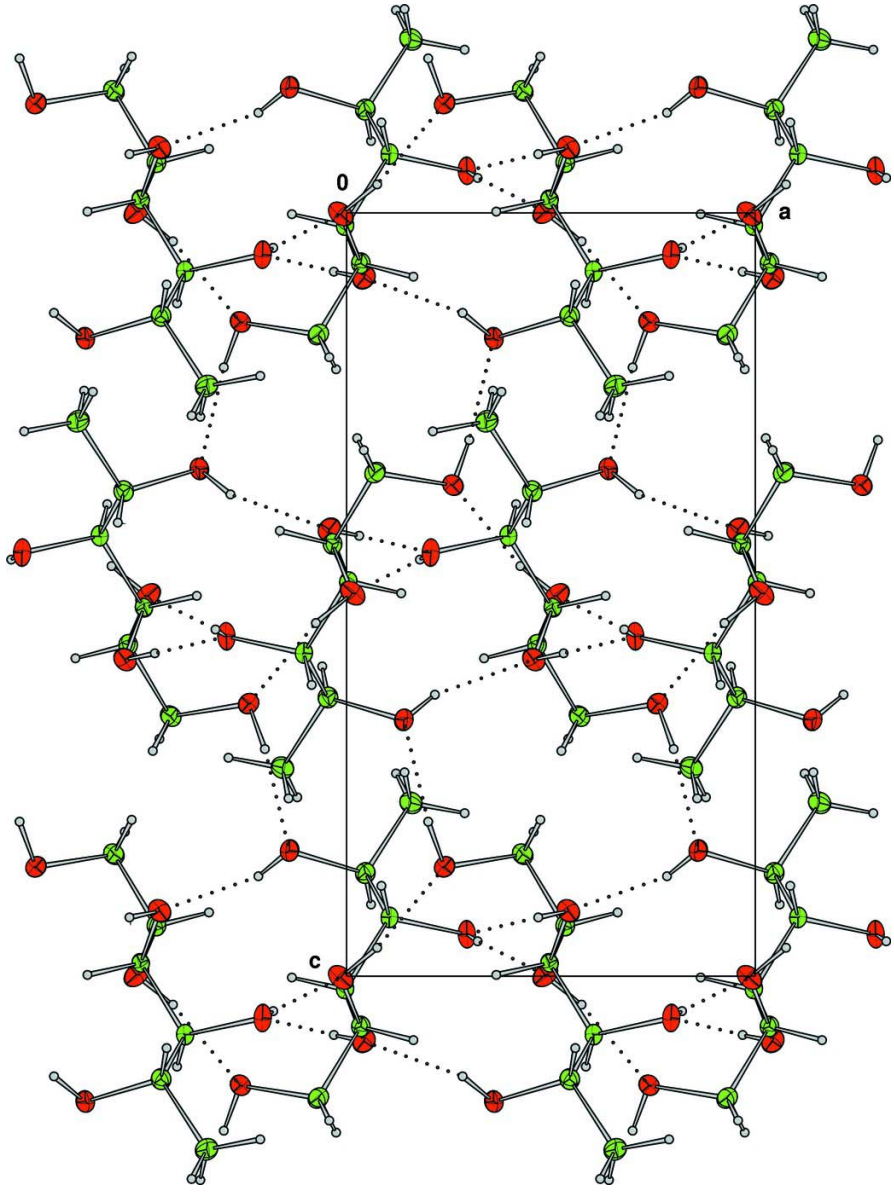


Figure 3

Packing diagram for the title compound projected along the *b* axis. Hydrogen bonds are shown as dotted lines.

1-Deoxy-L-mannitol

Crystal data

$C_6H_{14}O_5$
M_r = 166.17
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 7.3650 (3) Å
b = 7.6272 (3) Å
c = 13.7676 (5) Å
V = 773.39 (5) Å^3
Z = 4
$F(000)$ = 360
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1002 reflections
θ = 5–27°
μ = 0.12 mm^{-1}
T = 150 K
Plate, colourless
0.40 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.89, T_{max} = 0.99
5170 measured reflections
1033 independent reflections
974 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 5.2°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.072
S = 0.97
1033 reflections
100 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.19P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.000327
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.45760 (15)	0.66827 (14)	0.58528 (7)	0.0158
C2	0.5038 (2)	0.53406 (18)	0.51734 (10)	0.0121
C3	0.4654 (2)	0.35710 (19)	0.56608 (11)	0.0129
O4	0.51432 (16)	0.21669 (13)	0.50177 (8)	0.0180
C5	0.5694 (2)	0.3334 (2)	0.65961 (11)	0.0160
O6	0.76010 (15)	0.34756 (16)	0.64310 (8)	0.0190
C7	0.3954 (2)	0.55797 (19)	0.42326 (11)	0.0125
O8	0.20579 (15)	0.57629 (14)	0.44513 (8)	0.0163
C9	0.4543 (2)	0.7196 (2)	0.36498 (10)	0.0140
O10	0.63971 (16)	0.69611 (16)	0.33563 (8)	0.0188
C11	0.3428 (3)	0.7388 (2)	0.27300 (11)	0.0195
H21	0.6338	0.5422	0.5017	0.0146*
H31	0.3366	0.3507	0.5836	0.0149*
H51	0.5258	0.4239	0.7048	0.0180*
H52	0.5424	0.2171	0.6890	0.0191*
H71	0.4147	0.4569	0.3816	0.0137*
H91	0.4402	0.8236	0.4059	0.0171*
H111	0.3791	0.8390	0.2343	0.0290*
H112	0.2112	0.7500	0.2863	0.0299*
H113	0.3580	0.6334	0.2330	0.0284*
H1	0.7159	0.7532	0.3689	0.0319*
H2	0.4249	0.1898	0.4627	0.0307*
H3	0.1795	0.4708	0.4542	0.0290*
H4	0.8002	0.3523	0.7025	0.0312*
H5	0.5310	0.7560	0.5771	0.0285*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0168 (6)	0.0130 (5)	0.0175 (5)	−0.0022 (4)	0.0027 (5)	−0.0036 (4)
C2	0.0103 (7)	0.0137 (6)	0.0122 (6)	0.0004 (6)	0.0007 (6)	−0.0009 (5)
C3	0.0118 (7)	0.0125 (6)	0.0145 (7)	0.0011 (6)	0.0000 (6)	0.0014 (5)
O4	0.0207 (6)	0.0137 (5)	0.0198 (5)	0.0040 (5)	−0.0064 (5)	−0.0030 (4)
C5	0.0146 (8)	0.0191 (7)	0.0144 (7)	0.0011 (6)	0.0020 (6)	0.0022 (6)
O6	0.0144 (6)	0.0283 (6)	0.0142 (5)	0.0026 (5)	−0.0015 (4)	−0.0002 (4)
C7	0.0103 (7)	0.0127 (7)	0.0146 (7)	0.0004 (5)	0.0003 (6)	−0.0003 (6)
O8	0.0102 (5)	0.0128 (5)	0.0259 (6)	0.0000 (4)	−0.0005 (4)	0.0033 (4)
C9	0.0130 (7)	0.0143 (6)	0.0148 (7)	−0.0010 (6)	0.0003 (6)	0.0017 (6)
O10	0.0120 (6)	0.0284 (6)	0.0160 (5)	−0.0049 (5)	0.0009 (4)	−0.0020 (5)
C11	0.0173 (8)	0.0250 (8)	0.0163 (7)	0.0002 (7)	−0.0017 (6)	0.0065 (6)

Geometric parameters (Å, º)

O1—C2	1.4277 (17)	O6—H4	0.870
O1—H5	0.868	C7—O8	1.4354 (18)
C2—C3	1.5335 (19)	C7—C9	1.533 (2)
C2—C7	1.5323 (19)	C7—H71	0.971
C2—H21	0.983	O8—H3	0.837
C3—O4	1.4354 (18)	C9—O10	1.4352 (19)
C3—C5	1.509 (2)	C9—C11	1.516 (2)
C3—H31	0.980	C9—H91	0.979
O4—H2	0.875	O10—H1	0.845
C5—O6	1.4269 (18)	C11—H111	0.969
C5—H51	0.983	C11—H112	0.991
C5—H52	0.995	C11—H113	0.981
C2—O1—H5	108.6	C2—C7—O8	109.94 (12)
O1—C2—C3	107.49 (11)	C2—C7—C9	112.99 (12)
O1—C2—C7	110.15 (12)	O8—C7—C9	107.87 (12)
C3—C2—C7	112.26 (12)	C2—C7—H71	109.2
O1—C2—H21	109.3	O8—C7—H71	110.1
C3—C2—H21	109.3	C9—C7—H71	106.7
C7—C2—H21	108.4	C7—O8—H3	99.4
C2—C3—O4	109.91 (11)	C7—C9—O10	108.44 (13)
C2—C3—C5	112.67 (12)	C7—C9—C11	111.20 (12)
O4—C3—C5	108.04 (12)	O10—C9—C11	106.98 (12)
C2—C3—H31	109.3	C7—C9—H91	108.7
O4—C3—H31	111.0	O10—C9—H91	111.4
C5—C3—H31	105.9	C11—C9—H91	110.2
C3—O4—H2	111.5	C9—O10—H1	114.5
C3—C5—O6	110.76 (12)	C9—C11—H111	112.7
C3—C5—H51	106.9	C9—C11—H112	112.6
O6—C5—H51	111.7	H111—C11—H112	107.7
C3—C5—H52	110.6	C9—C11—H113	109.2

O6—C5—H52	109.2	H111—C11—H113	107.8
H51—C5—H52	107.6	H112—C11—H113	106.6
C5—O6—H4	100.8		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H1···O1^{i}	0.85	1.98	2.782 (2)	158
O4—H2···O6^{ii}	0.87	1.92	2.779 (2)	168
O8—H3···O4^{ii}	0.84	1.97	2.742 (2)	152
O6—H4···O10^{iii}	0.87	1.92	2.772 (2)	165
O1—H5···O8^{i}	0.87	1.84	2.704 (2)	173

Symmetry codes: (i) $x+1/2, -y+3/2, -z+1$; (ii) $x-1/2, -y+1/2, -z+1$; (iii) $-x+3/2, -y+1, z+1/2$.