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6-Deoxy-α-L-talopyranose

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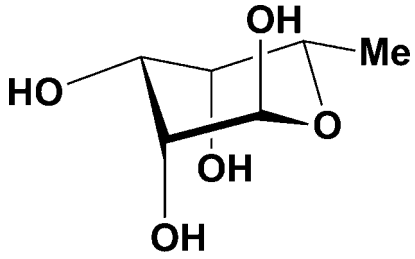
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.073; data-to-parameter ratio = 9.7.

X-ray crystallography showed that the title compound, $C_6H_{12}O_5$, crystallizes in the α-pyranose form with the six-membered ring in a chair conformation. The crystal structure exists as a three-dimensional hydrogen-bonded network of molecules with each molecule acting as a donor and aceptor for four hydrogen bonds. The absolute configuration was determined by the use of L-fucose as starting material.

Related literature

For related literature, see: Beadle *et al.* (1992); Izumori (2002, 2006); Granstrom *et al.* (2004); Yoshihara *et al.* (2008).



Experimental

Crystal data

$C_6H_{12}O_5$
M_r = 164.16
Orthorhombic, $P2_12_12_1$
a = 6.4939 (3) Å
b = 7.4874 (4) Å
c = 14.8382 (8) Å
V = 721.47 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 150 K
0.25 × 0.25 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.97, T_{max} = 1.00
(expected range = 0.967–0.997)
4390 measured reflections
968 independent reflections
863 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.072
S = 1.03
968 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H7···O1^{i}	0.81	2.04	2.818 (2)	162
O1—H8···O10	0.82	1.98	2.740 (2)	156
O10—H10···O9^{i}	0.84	1.85	2.686 (2)	177
O11—H1···O4^{ii}	0.87	1.94	2.812 (2)	177

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

Data collection: *COLLECT* (Nonius, 1997–2001).; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2652).

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supporting information

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S1. Comment

The range of rare sugars that are now readily available has increased in recent years due to both chemical (Beadle *et al.*, 1992) and biotechnological (Izumori, 2006; Izumori, 2002; Granstrom *et al.*, 2004) advances. The methodology developed by Izumori *et al.* for the interconversion of tetroses, pentoses and hexoses by enzymatic oxidation, inversion at C3 with a single epimerase, and reduction to the aldose has been seen to be generally applicable for the 1-deoxy ketohexoses (Yoshihara *et al.*, 2008) in large amounts in water.

The Izumoring method is demonstrated here with the synthesis of 6-deoxy-*L*-talose **3** from *L*-fucose **1**(Fig. 1) by a series of isomerizations. Firstly, using D-arabinose isomerase, *L*-fucose was isomerized to 6-deoxy-*L*-tagatose **2** and then using *L*-rhamnose isomerase this was further isomerized to give 6-deoxy-*L*-talose **3**.

6-Deoxy-*L*-talose crystallizes solely in the α-pyranose form (Fig. 2). The absolute configuration was determined from the starting material. The crystal exists as an hydrogen bonded network with each molecule acting as a donor and acceptor for 4 hydrogen bonds. Non-conventional hydrogen bonds have been ignored.

S2. Experimental

The title compound was recrystallized from methanol: m.p. 120–123°C; $[\alpha]_D^{20}$ -18.6 (*c*, 0.94 in H_2O).

S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned 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.

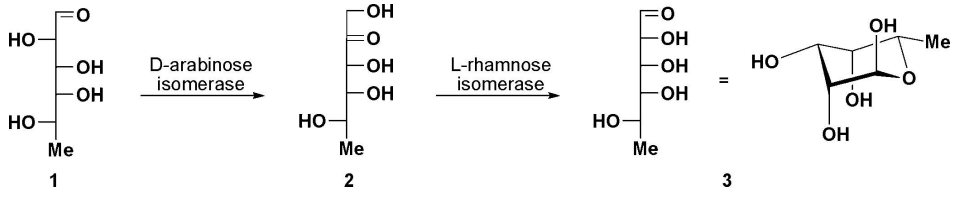

Figure 1

Synthetic scheme.

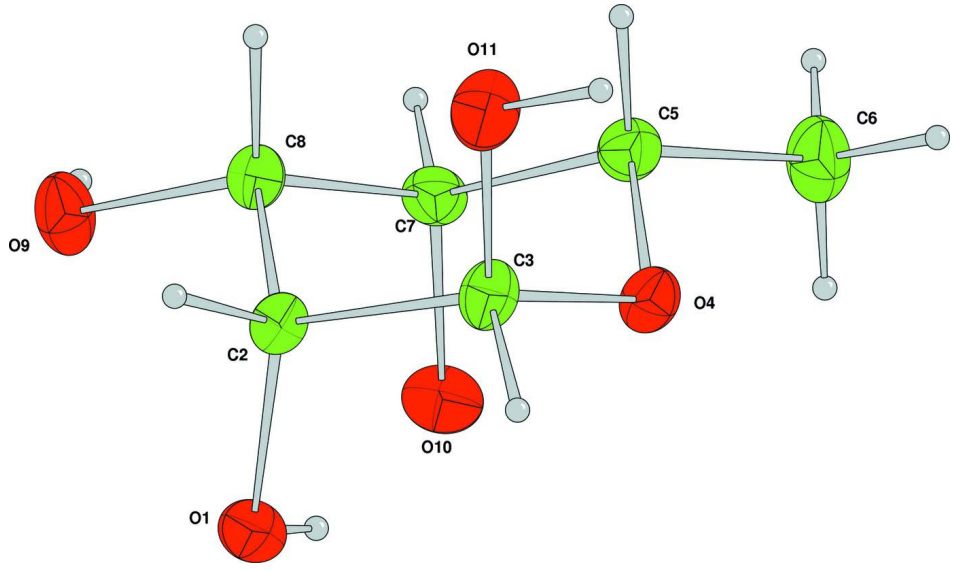

Figure 2

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

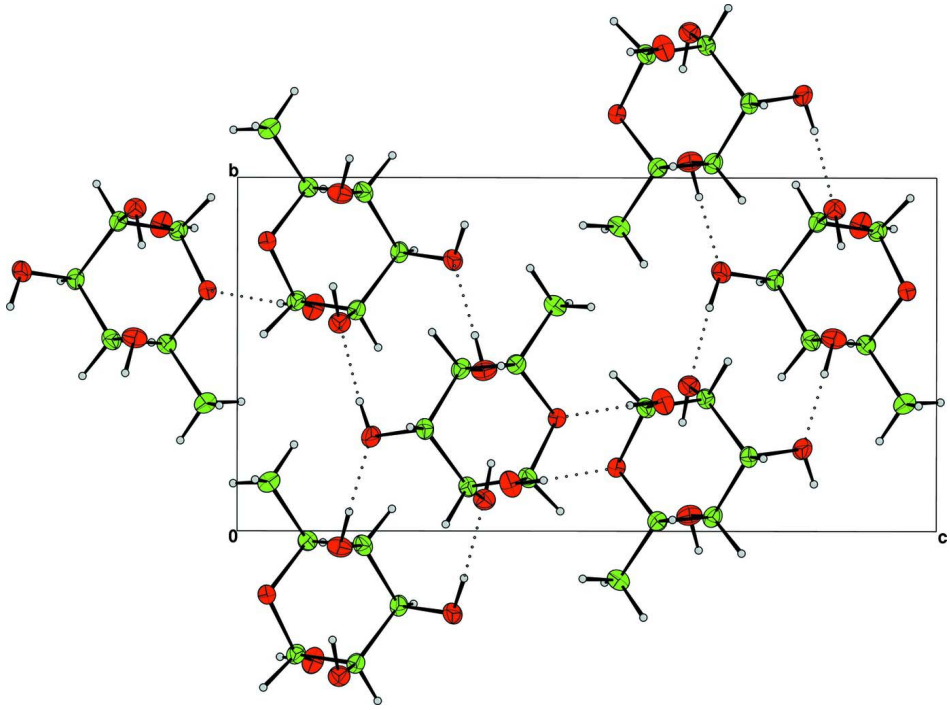

Figure 3

Packing diagram of the title compound projected along the *a*-axis.

(I)

Crystal data

$C_6H_{12}O_5$
$M_r = 164.16$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.4939\ (3)$ Å
$b = 7.4874\ (4)$ Å
$c = 14.8382\ (8)$ Å
$V = 721.47\ (6)$ Å^3
$Z = 4$
$F(000) = 352$
$D_x = 1.511$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 890 reflections
$\theta = 5$–$27°$
$\mu = 0.13$ mm^{-1}
$T = 150$ K
Plate, colourless
0.25 × 0.25 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997)
$T_{min} = 0.97$, $T_{max} = 1.00$
4390 measured reflections
968 independent reflections
863 reflections with $I > 2.0\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.072$
$S = 1.03$
968 reflections
100 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.04P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.000184$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.95002 (16)	0.08900 (18)	0.35376 (7)	0.0210
C2	0.7416 (2)	0.1250 (2)	0.32982 (10)	0.0175
C3	0.6113 (3)	0.1509 (3)	0.41551 (10)	0.0188
O4	0.65011 (17)	0.31964 (16)	0.45740 (7)	0.0183
C5	0.6150 (2)	0.4717 (2)	0.39897 (10)	0.0194
C6	0.6412 (3)	0.6382 (3)	0.45454 (12)	0.0271
C7	0.7657 (2)	0.4593 (2)	0.32003 (10)	0.0187
C8	0.7208 (2)	0.2889 (3)	0.26846 (10)	0.0186
O9	0.85143 (18)	0.26675 (18)	0.19178 (7)	0.0243
O10	0.97297 (17)	0.45445 (18)	0.35319 (8)	0.0232
O11	0.40458 (18)	0.1333 (2)	0.39115 (8)	0.0254
H21	0.6937	0.0193	0.2985	0.0197*
H31	0.6474	0.0567	0.4621	0.0197*
H51	0.4756	0.4675	0.3773	0.0236*

H61	0.6179	0.7441	0.4188	0.0406*
H62	0.5480	0.6346	0.5057	0.0398*
H63	0.7791	0.6452	0.4743	0.0401*
H71	0.7508	0.5628	0.2786	0.0214*
H81	0.5761	0.2938	0.2475	0.0221*
H7	0.8826	0.3664	0.1753	0.0374*
H8	0.9866	0.1914	0.3632	0.0333*
H10	1.0243	0.5538	0.3399	0.0350*
H1	0.3258	0.1436	0.4383	0.0408*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0198 (5)	0.0204 (7)	0.0229 (6)	0.0030 (5)	−0.0010 (5)	−0.0005 (5)
C2	0.0184 (7)	0.0188 (9)	0.0153 (7)	−0.0001 (8)	−0.0013 (6)	−0.0015 (6)
C3	0.0209 (8)	0.0186 (9)	0.0169 (7)	−0.0037 (7)	0.0005 (6)	−0.0021 (7)
O4	0.0223 (5)	0.0178 (7)	0.0150 (5)	−0.0008 (5)	−0.0006 (5)	0.0007 (5)
C5	0.0220 (8)	0.0184 (9)	0.0179 (7)	0.0019 (8)	−0.0015 (7)	0.0002 (7)
C6	0.0378 (10)	0.0202 (10)	0.0232 (8)	0.0022 (9)	0.0025 (9)	−0.0040 (7)
C7	0.0183 (7)	0.0202 (9)	0.0176 (7)	0.0031 (7)	0.0006 (7)	0.0029 (7)
C8	0.0197 (7)	0.0212 (10)	0.0149 (7)	0.0011 (7)	0.0013 (6)	−0.0005 (7)
O9	0.0335 (6)	0.0213 (7)	0.0180 (5)	0.0043 (6)	0.0087 (5)	0.0025 (5)
O10	0.0195 (5)	0.0195 (7)	0.0306 (6)	−0.0026 (5)	−0.0015 (5)	0.0023 (6)
O11	0.0199 (5)	0.0331 (8)	0.0233 (5)	−0.0074 (6)	0.0028 (5)	−0.0047 (6)

Geometric parameters (Å, º)

O1—C2	1.4250 (19)	C6—H61	0.966
O1—H8	0.815	C6—H62	0.971
C2—C3	1.540 (2)	C6—H63	0.944
C2—C8	1.534 (2)	C7—C8	1.516 (2)
C2—H21	0.969	C7—O10	1.4334 (19)
C3—O4	1.430 (2)	C7—H71	0.994
C3—O11	1.3965 (19)	C8—O9	1.4288 (18)
C3—H31	1.015	C8—H81	0.990
O4—C5	1.449 (2)	O9—H7	0.811
C5—C6	1.504 (2)	O10—H10	0.839
C5—C7	1.529 (2)	O11—H1	0.870
C5—H51	0.962		
C2—O1—H8	98.1	C5—C6—H62	109.6
O1—C2—C3	109.88 (12)	H61—C6—H62	110.8
O1—C2—C8	112.50 (13)	C5—C6—H63	108.9
C3—C2—C8	109.93 (14)	H61—C6—H63	105.9
O1—C2—H21	105.7	H62—C6—H63	110.5
C3—C2—H21	108.8	C5—C7—C8	108.34 (14)
C8—C2—H21	109.9	C5—C7—O10	109.84 (12)
C2—C3—O4	111.93 (14)	C8—C7—O10	109.43 (14)

C2—C3—O11	107.62 (12)	C5—C7—H71	111.3
O4—C3—O11	111.43 (15)	C8—C7—H71	109.0
C2—C3—H31	110.4	O10—C7—H71	108.9
O4—C3—H31	106.1	C2—C8—C7	110.89 (12)
O11—C3—H31	109.4	C2—C8—O9	109.12 (13)
C3—O4—C5	113.97 (11)	C7—C8—O9	112.67 (13)
O4—C5—C6	107.79 (12)	C2—C8—H81	107.4
O4—C5—C7	108.05 (13)	C7—C8—H81	108.0
C6—C5—C7	113.45 (14)	O9—C8—H81	108.5
O4—C5—H51	108.8	C8—O9—H7	106.4
C6—C5—H51	108.5	C7—O10—H10	105.7
C7—C5—H51	110.1	C3—O11—H1	110.4
C5—C6—H61	111.2		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H7···O1^{i}	0.81	2.04	2.818 (2)	162
O1—H8···C7	0.82	2.55	3.061 (2)	122
O1—H8···O10	0.82	1.98	2.740 (2)	156
O10—H10···O9^{i}	0.84	1.85	2.686 (2)	177
O11—H1···O4^{ii}	0.87	1.94	2.812 (2)	177

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