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1-Deoxy-1-fluoro-L-galactitol

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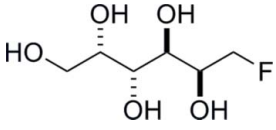
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.095; data-to-parameter ratio = 8.7.

The crystal structure unequivocally confirms the relative stereochemistry of the title compound, $C_6H_{13}FO_5$ [6-deoxy-6-fluoro-D-galactitol or (2*S*,3*R*,4*R*,5*S*)-6-fluorohexane-1,2,3,4,5-pentaol]. The absolute stereochemistry was determined from the use of D-galactose as the starting material. In the crystal, the molecules are linked by O—H···O and O—H···F hydrogen bonds, forming a three-dimensional network with each molecule acting as a donor and acceptor for five hydrogen bonds.

Related literature

For literature regarding fluorogalactitol and fluorogalactose, see: Kent & Wright (1972); Jenkinson *et al.* (2010).



Experimental

Crystal data

$C_6H_{13}FO_5$
M_r = 184.16
Monoclinic, $P2_1$
a = 4.7968 (3) Å
b = 8.5957 (5) Å
c = 9.8194 (7) Å
β = 103.233 (3)°
V = 394.12 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 150 K
0.40 × 0.10 × 0.05 mm

Data collection

Area diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.88, T_{max} = 0.99
3069 measured reflections
947 independent reflections
788 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.095
S = 0.99
947 reflections
109 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H41···O8^{i}	0.82	1.94	2.738 (4)	165
O10—H101···O12^{ii}	0.82	1.95	2.730 (4)	160
O8—H81···O10^{iii}	0.82	1.87	2.691 (4)	172
O6—H61···O4^{iv}	0.82	1.89	2.703 (4)	170
O12—H121···F1^{v}	0.84	2.08	2.895 (3)	163

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5036).

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supplementary materials

Acta Cryst. (2010). E**66**, o1330 [**doi:10.1107/S1600536810016624**]

1-Deoxy-1-fluoro-L-galactitol

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Comment

1-Deoxy-1-fluoro-L-galactitol [6-deoxy-6-fluoro-D-galactitol, (2*S*,3*R*,4*R*,5*S*)-6-fluorohexane-1,2,3,4,5-pentaol] **3** was prepared in 88% yield by reduction of 6-deoxy-6-fluoro-D-galactose **2**, itself readily available from D-galactose (Jenkinson *et al.*, 2010) with sodium borohydride in water (see fig. 1).

1-Deoxy-1-fluoro-L-galactitol **3** (Fig. 2) exists as an extensively hydrogen bonded lattice with each molecule acting as a donor and acceptor for 5 hydrogen bonds (Fig. 3 and Fig. 4). Only classical hydrogen bonding is considered.

Experimental

The title compound was recrystallised by vapour diffusion from a mixture of methanol and water: m.p. 445-447 K, $[\alpha]_D^{25}$ +4.1 (*c* 1.06, H_2O) {Lit. (Kent & Wright, 1972) m.p. 446-447 K, $[\alpha]_D^{21}$ +4.2 (*c* 0.5, H_2O)}.

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.

Figures

Fig. 1. Synthetic Scheme.

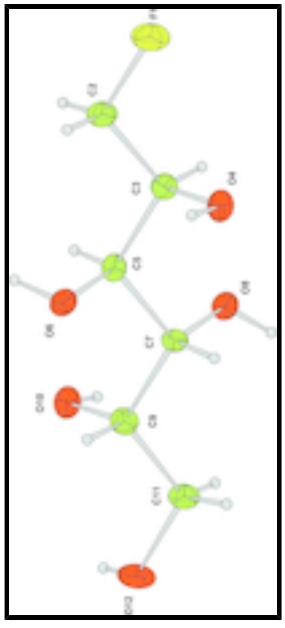


Fig. 2. The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

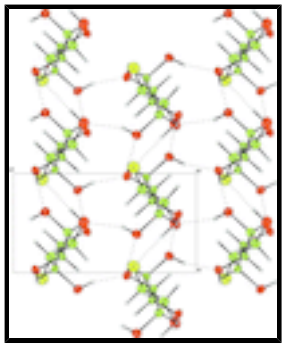


Fig. 3. Packing diagram for the title compound projected along the *c*-axis. Hydrogen bonds are indicated by dotted lines.

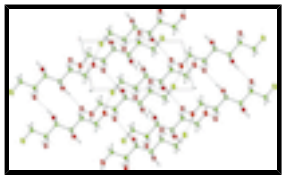


Fig. 4. Packing diagram for the title compound projected along the *b*-axis. Hydrogen bonds are indicated by dotted lines.

1-Deoxy-1-fluoro-L-galactitol

Crystal data

$C_6H_{13}FO_5$	$F(000) = 196$
$M_r = 184.16$	$D_x = 1.552$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 828 reflections
$a = 4.7968$ (3) Å	$\theta = 5$–27°
$b = 8.5957$ (5) Å	$\mu = 0.15$ mm^{-1}
$c = 9.8194$ (7) Å	$T = 150$ K
$\beta = 103.233$ (3)°	Needle, colourless
$V = 394.12$ (4) Å^3	0.40 × 0.10 × 0.05 mm

$Z = 2$

Data collection

Area diffractometer	788 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.039$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)	$h = -6 \rightarrow 6$
$T_{min} = 0.88$, $T_{max} = 0.99$	$k = -10 \rightarrow 11$
3069 measured reflections	$l = -12 \rightarrow 12$
947 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.095$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.16P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.0001$
947 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
109 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}
1 restraint	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	1.0608 (4)	0.8416 (3)	0.81046 (18)	0.0300
C2	0.8382 (7)	0.7956 (4)	0.6953 (3)	0.0222
C3	0.9617 (6)	0.7729 (3)	0.5690 (3)	0.0165
O4	1.1674 (4)	0.6479 (3)	0.5922 (2)	0.0198
C5	0.7225 (6)	0.7433 (3)	0.4383 (3)	0.0161
O6	0.5806 (5)	0.6003 (2)	0.4502 (2)	0.0195
C7	0.8349 (6)	0.7338 (3)	0.3047 (3)	0.0157
O8	0.9746 (4)	0.8784 (3)	0.2914 (2)	0.0195
C9	0.5959 (7)	0.7026 (4)	0.1752 (3)	0.0192
O10	0.3879 (4)	0.8258 (3)	0.1522 (2)	0.0207
C11	0.7088 (7)	0.6776 (4)	0.0451 (3)	0.0215
O12	0.4930 (5)	0.6156 (3)	−0.0668 (2)	0.0275
H21	0.6950	0.8781	0.6774	0.0260*
H22	0.7491	0.6999	0.7173	0.0258*
H31	1.0631	0.8691	0.5540	0.0186*

H51	0.5826	0.8297	0.4297	0.0192*
H71	0.9744	0.6473	0.3126	0.0169*
H91	0.4965	0.6083	0.1918	0.0218*
H111	0.7818	0.7757	0.0180	0.0236*
H112	0.8650	0.6017	0.0659	0.0245*
H41	1.0976	0.5692	0.6169	0.0297*
H101	0.4579	0.9011	0.1221	0.0326*
H81	1.0943	0.8692	0.2436	0.0307*
H61	0.4434	0.6205	0.4847	0.0315*
H121	0.3696	0.6866	−0.0851	0.0400*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0335 (11)	0.0350 (11)	0.0187 (9)	0.0018 (10)	0.0002 (8)	−0.0045 (8)
C2	0.0217 (15)	0.0270 (17)	0.0166 (14)	0.0023 (12)	0.0019 (12)	−0.0039 (12)
C3	0.0168 (14)	0.0140 (13)	0.0181 (13)	0.0019 (11)	0.0029 (11)	−0.0004 (11)
O4	0.0176 (11)	0.0202 (11)	0.0222 (10)	0.0029 (8)	0.0054 (8)	0.0048 (8)
C5	0.0162 (14)	0.0136 (14)	0.0194 (14)	0.0000 (11)	0.0058 (11)	0.0011 (11)
O6	0.0229 (12)	0.0169 (10)	0.0215 (10)	−0.0052 (9)	0.0109 (9)	−0.0024 (8)
C7	0.0172 (14)	0.0148 (15)	0.0149 (13)	−0.0012 (12)	0.0029 (11)	−0.0027 (11)
O8	0.0208 (12)	0.0165 (11)	0.0233 (10)	−0.0033 (9)	0.0094 (9)	−0.0024 (9)
C9	0.0200 (14)	0.0201 (15)	0.0173 (14)	−0.0015 (12)	0.0037 (12)	0.0006 (12)
O10	0.0190 (11)	0.0217 (11)	0.0223 (11)	0.0015 (9)	0.0064 (8)	0.0042 (9)
C11	0.0227 (16)	0.0257 (17)	0.0145 (14)	0.0006 (13)	0.0011 (12)	−0.0018 (13)
O12	0.0312 (14)	0.0274 (12)	0.0201 (11)	0.0025 (10)	−0.0017 (10)	−0.0078 (10)

Geometric parameters (Å, °)

F1—C2	1.422 (3)	C7—O8	1.432 (4)
C2—C3	1.504 (4)	C7—C9	1.528 (4)
C2—H21	0.975	C7—H71	0.992
C2—H22	0.974	O8—H81	0.824
C3—O4	1.441 (4)	C9—O10	1.437 (4)
C3—C5	1.534 (4)	C9—C11	1.513 (4)
C3—H31	0.987	C9—H91	0.973
O4—H41	0.816	O10—H101	0.815
C5—O6	1.422 (4)	C11—O12	1.429 (3)
C5—C7	1.530 (3)	C11—H111	0.973
C5—H51	0.992	C11—H112	0.979
O6—H61	0.825	O12—H121	0.840
F1—C2—C3	109.0 (3)	C5—C7—C9	112.2 (2)
F1—C2—H21	108.1	O8—C7—C9	110.7 (2)
C3—C2—H21	109.9	C5—C7—H71	109.7
F1—C2—H22	110.2	O8—C7—H71	109.6
C3—C2—H22	110.4	C9—C7—H71	107.3
H21—C2—H22	109.1	C7—O8—H81	111.8
C2—C3—O4	110.5 (2)	C7—C9—O10	111.3 (2)

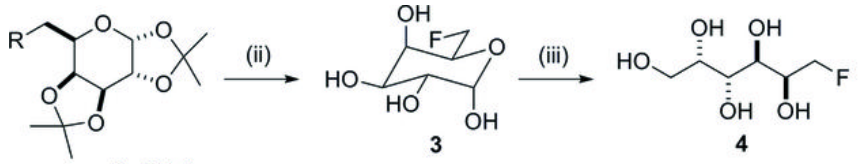
C2—C3—C5	110.6 (2)	C7—C9—C11	112.5 (2)
O4—C3—C5	111.3 (2)	O10—C9—C11	110.0 (2)
C2—C3—H31	108.3	C7—C9—H91	108.0
O4—C3—H31	107.7	O10—C9—H91	107.0
C5—C3—H31	108.3	C11—C9—H91	107.8
C3—O4—H41	110.6	C9—O10—H101	108.3
C3—C5—O6	110.8 (2)	C9—C11—O12	111.5 (3)
C3—C5—C7	112.5 (2)	C9—C11—H111	109.2
O6—C5—C7	107.1 (2)	O12—C11—H111	110.9
C3—C5—H51	108.0	C9—C11—H112	108.9
O6—C5—H51	109.1	O12—C11—H112	107.3
C7—C5—H51	109.3	H111—C11—H112	109.2
C5—O6—H61	107.1	C11—O12—H121	104.2
C5—C7—O8	107.3 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H21···O6^{i}	0.97	2.49	3.409 (4)	157
O4—H41···O8^{ii}	0.82	1.94	2.738 (4)	165
O10—H101···O12^{iii}	0.82	1.95	2.730 (4)	160
O8—H81···O10^{iv}	0.82	1.87	2.691 (4)	172
O6—H61···O4^{v}	0.82	1.89	2.703 (4)	170
O12—H121···F1^{vi}	0.84	2.08	2.895 (3)	163

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

Fig. 1



Reagents and conditions: (i) $(CF_3SO_2)_2O$, pyridine, DCM, -30°C; then TBAF, THF; (ii) Dowex (H^+), H_2O, 1,4-dioxane; (iii) $NaBH_4$, H_2O.

Fig. 2

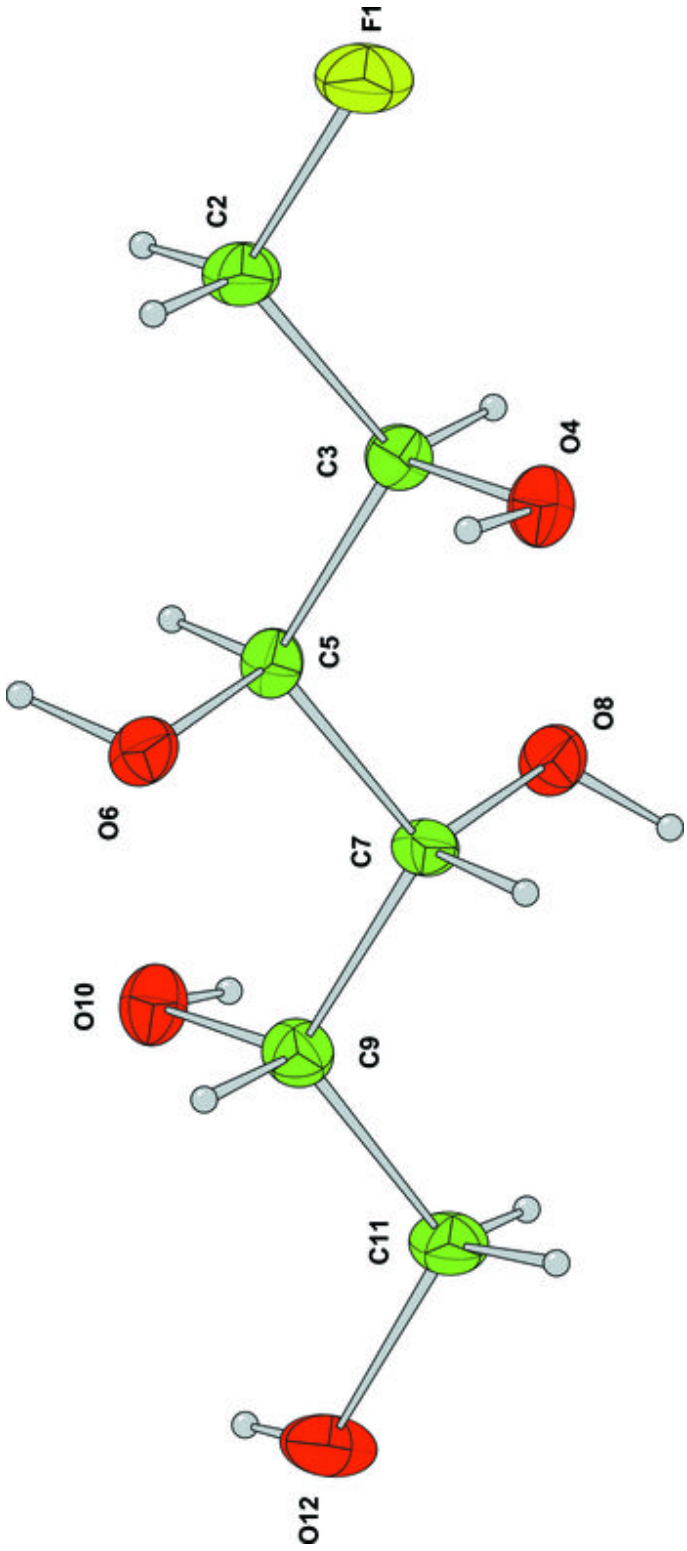


Fig. 3

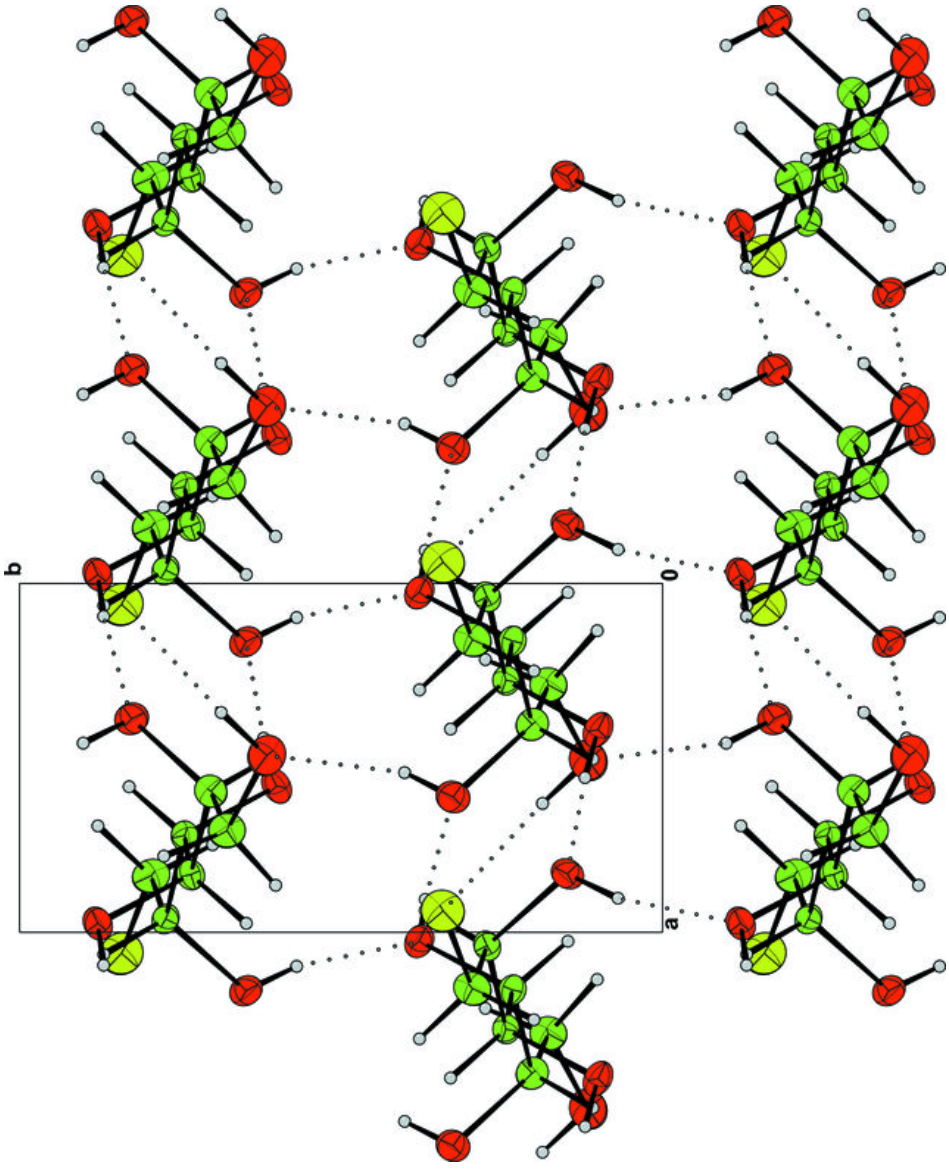


Fig. 4