Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Methyl 2-*O*-β-L-fucopyranosyl α-D-glucopyranoside monohydrate: a synchrotron study

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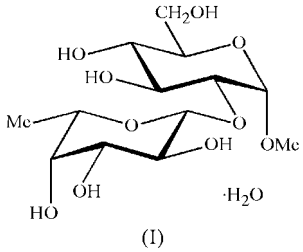
Received 11 November 2007
Accepted 15 November 2007
Online 12 January 2008

The structure of the title compound, $C_{13}H_{24}O_{10}\cdot H_2O$, is stabilized by hydrogen bonds situated adjacent to the glycoside linkage. A direct intramolecular hydrogen bond is present between the fucopyranosyl ring O atom and a glucopyranoside OH group, and a bridging water molecule mediates a hydrogen-bond-based interaction from a fucopyranosyl OH group to the methoxy O atom. The conformation of the disaccharide is described by the glycosidic torsion angles $\varphi_H = -41^\circ$ and $\psi_H = -2^\circ$.

Comment

Carbohydrates are often found as glycoconjugates in nature and they are involved in a multitude of different functions of significant importance in biomolecular systems (Carlsson *et al.*, 2007). Elucidation of the three-dimensional structure of the carbohydrate part or of substructures of a larger oligosaccharide may reveal structural features including possible epitopes essential for molecular recognition events. We have previously reported the crystal structures of methyl 4-*O*-β-L-fucopyranosyl α-D-glucopyranoside (F4G) (Eriksson *et al.*, 2000) and methyl 3-*O*-β-L-fucopyranosyl α-D-glucopyranoside (F3G) (Färnbäck *et al.*, 2003) as a hemihydrate and a trihydrate, respectively, and we report here the title compound (F2G) as a monohydrate, (I).



(I)

In F4G, the glycosidic torsion angles are $\varphi_H = -6^\circ$ (being unusual since it was close to an eclipsed conformation) and $\psi_H = 34^\circ$, whereas in F3G, the values are $\varphi_H = -38^\circ$ and $\psi_H = 18^\circ$. The hydroxymethyl group for the exocyclic torsion angle ω revealed the *gt* and *gg* conformations in F4G and F3G, respectively. Both conformations are favored by the *gauche* effect and in none of the compounds is a 1,3-*syn*-diaxial interaction to O4 present, consistent with the Hassel–Ottar effect (Jeffrey, 1990). Notably, both conformations are shifted slightly from the ideal *gauche* conformation, *i.e.* $\omega = 68^\circ$ in the former and $\omega = -66^\circ$ in the latter compound. In F3G, an intramolecular hydrogen bond was present, with O2*g* as the donor atom and O5*f* as the acceptor atom.

In the present study, the torsion angles at the glycosidic linkage are $\varphi_H \simeq -41^\circ$ and $\psi_H \simeq -2^\circ$. The exocyclic torsion angle ω is $\sim -64^\circ$, *i.e.* the *gg* conformation (Table 1). The calculated Cremer & Pople (1975) parameters show that the fucose ring is close to the expected chair conformation, *i.e.* $^1C_4$, and that the glucose ring has the anticipated $^4C_1$ conformation. The parameters for the fucose ring are $Q = 0.592\ (3)$ Å, $\theta = 173.1\ (3)^\circ$ and $\varphi = 256\ (3)^\circ$, and those for the glucose ring $Q = 0.544\ (3)$ Å, $\theta = 12.1\ (3)^\circ$ and $\varphi = 338\ (2)^\circ$. An internal hydrogen bond is present, with atom O5*f* as the acceptor and atom O3*g* as the donor (*cf.* F3G where O5*f* is the acceptor and O2*g* the donor), adjacent to the glycosidic linkage (Fig. 1). On the other side, the water molecule present in the crystal structure confers additional structure to F2G. The water molecule is positioned in such a way that atom O2*f* acts as a donor, with its hydroxy H atom pointing towards the water O atom, and both water H atoms act as donors, to atoms O1*g* and O3*g* in a neighbouring molecule at $(x-1, y, z)$. Further details about the hydrogen bonding are shown in Fig. 1 and given in

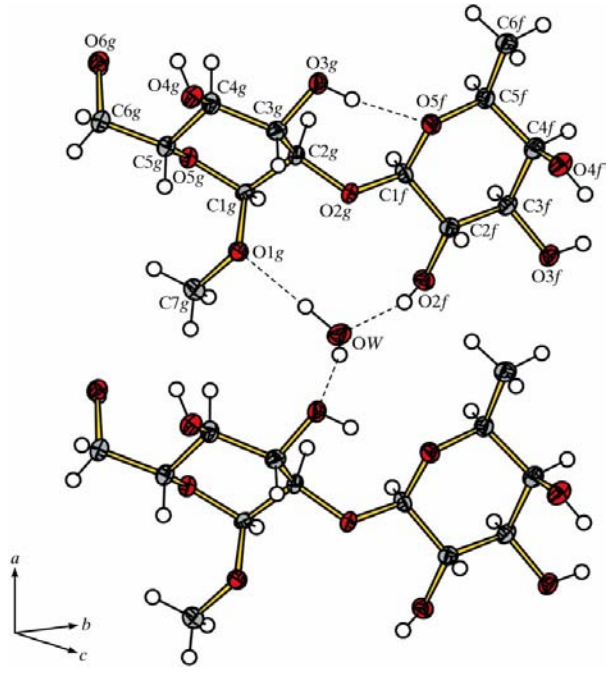


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radii and hydrogen bonds are shown as dashed lines. The water molecule mediates a hydrogen bond between two molecules.

 DOI: 10.1107/S0108270107059756

Table 2. Thus, the water molecule is present as a bridge between atoms O2*f* and O1*g*. Bridging water molecules of structural importance have been reported to be present in disaccharides using molecular dynamics simulations (Naidoo & Brady, 1999) and NMR spectroscopy (Sheng & van Halbeek, 1995). As a result of the intramolecular hydrogen-bonding pattern in the crystal structure of F2G, it is surmised that the disaccharide may be exceptionally well structured in aqueous solution, a matter that remains to be investigated.

Experimental

The synthesis of F2G was described by Baumann *et al.* (1991). Suitable crystals was grown using the sitting-drop technique, in which the solvent was allowed to evaporate. F2G was dissolved in water to a concentration of 375 mg ml^{-1} and mixed with an equal amount of 20% PEG 400 in water at ambient temperature. Crystals formed after a few days and were mounted in capillaries. Data were collected on beamline I711 at the Swedish synchrotron radiation facility, MAXLAB, Lund.

Crystal data

$C_{13}H_{24}O_{10}\cdot H_2O$
M_r = 358.34
Monoclinic, $P2_1$
a = 7.166 (2) Å
b = 6.3924 (16) Å
c = 18.518 (4) Å
β = 96.876 (12)°
V = 842.1 (4) Å^3
Z = 2
Synchrotron radiation
λ = 0.872 Å
μ = 0.20 mm^{-1}
T = 100 (2) K
0.12 × 0.06 × 0.03 mm

Data collection

Smart 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.97, T_{max} = 0.99
4040 measured reflections
1751 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.120
S = 1.07
1751 reflections
229 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

H1*f*···H2*g*	2.19		
O5*g*—C1*g*—O1*g*—C7*g*	62.5 (3)	O5*g*—C5*g*—C6*g*—O6*g*	−63.6 (3)
C2*g*—C1*g*—O1*g*—C7*g*	−176.0 (2)	C1*f*—O2*g*—C2*g*—H2*g*	−1.8
O5*f*—C1*f*—O2*g*—C2*g*	79.1 (3)	H1*f*—C1*f*—O2*g*—C2*g*	−41.5
C3*g*—C2*g*—O2*g*—C1*f*	−119.0 (2)	C7*g*—O1*g*—C1*g*—H1*g*	−57.5
C1*g*—C2*g*—O2*g*—C1*f*	116.5 (2)		

H atoms of the water molecule were located in a difference density map and the O—H distances were restrained to retain the previously known geometry of the water molecule; U_{iso}(H) values were set at 1.5U_{eq}(O). All other H atoms were positioned geometrically and constrained to ride on the parent atom. The C—H bond distances are 0.98 Å for CH_3, 0.99 Å for CH_2 and 1.00 Å for CH, and the O—H bond distance is 0.84 Å for OH groups. The U_{iso}(H) values were set at 1.5U_{eq}(C,O) for the CH_3 and OH groups and 1.2U_{eq}(C) for all other H atoms. The value of the Flack (1983) parameter was not meaningful owing to the absence of significant anomalous scatterers; thus, the data were merged using MERG 3 in *SHELXL97* (Sheldrick, 1997). The absolute configuration of each sugar residue is known from the starting compounds used in the synthesis.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*f*—H2*fA*···O*W*	0.84	1.90	2.712 (3)	162
O3*f*—H3*fA*···O2*f*^{i}	0.84	1.86	2.657 (3)	158
O4*f*—H4*fA*···O3*f*^{i}	0.84	2.07	2.888 (3)	163
O3*g*—H3*gA*···O5*g*^{ii}	0.84	2.15	2.835 (3)	139
O3*g*—H3*gA*···O5*f*	0.84	2.51	3.006 (3)	118
O4*g*—H4*gA*···O6*g*^{iii}	0.84	1.89	2.728 (3)	178
O6*g*—H6*g*···O3*g*^{iv}	0.84	1.99	2.795 (3)	160
O*W*—H*W*1···O3*g*^{v}	0.95 (5)	2.00 (5)	2.915 (3)	161 (3)
O*W*—H*W*2···O1*g*	0.95 (5)	1.95 (5)	2.866 (3)	161 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

This work was supported by a grant from the Swedish Research Council (VR).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF3069). Services for accessing these data are described at the back of the journal.

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