organic compounds

Methyl β-D-galactopyranosyl-(1→4)-β-D-allopyranoside tetrahydrate

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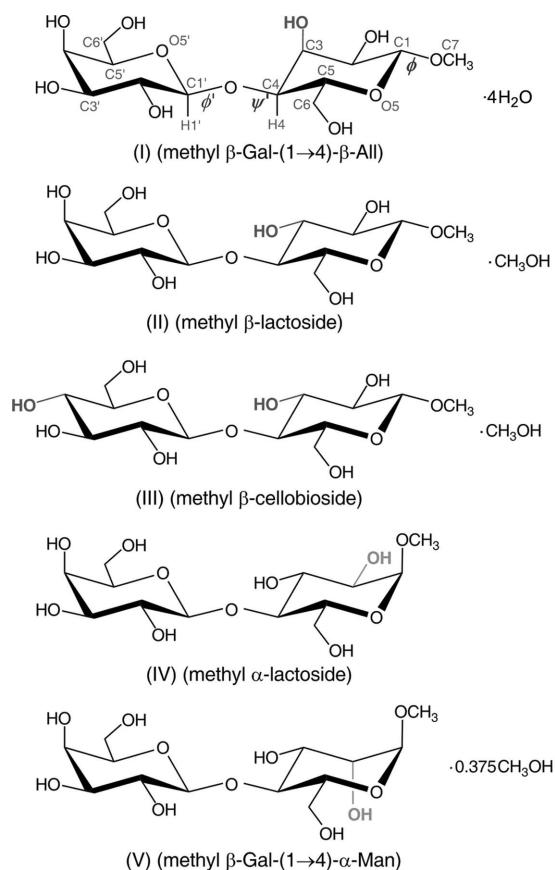Correspondence e-mail: aseriann@nd.edu

The title compound, $C_{13}H_{24}O_{11}\cdot 4H_2O$, (I), crystallized from water, has an internal glycosidic linkage conformation having φ' (O5$_{Gal}$—C1$_{Gal}$—O1$_{Gal}$—C4$_{All}$) = $-96.40\,(12)°$ and ψ' (C1$_{Gal}$—O1$_{Gal}$—C4$_{All}$—C5$_{All}$) = $-160.93\,(10)°$, where ring-atom numbering conforms to the convention in which C1 denotes the anomeric C atom, C5 the ring atom bearing the exocyclic hydroxymethyl group, and C6 the exocyclic hydroxymethyl (CH$_2$OH) C atom in the βGal$p$ and βAll$p$ residues. Internal linkage conformations in the crystal structures of the structurally related disaccharides methyl β-lactoside [methyl β-D-galactopyranosyl-(1→4)-β-D-glucopyranoside] methanol solvate [Stenutz, Shang & Serianni (1999). *Acta Cryst.* C**55**, 1719–1721], (II), and methyl β-cellobioside [methyl β-D-glucopyranosyl-(1→4)-β-D-glucopyranoside] methanol solvate [Ham & Williams (1970). *Acta Cryst.* B**26**, 1373–1383], (III), are characterized by $\varphi' = -88.4\,(2)°$ and $\psi' = -161.3\,(2)°$, and $\varphi' = -91.1°$ and $\psi' = -160.7°$, respectively. Inter-residue hydrogen bonding is observed between O3$_{Glc}$ and O5$_{Gal/Glc}$ in the crystal structures of (II) and (III), suggesting a role in determining their preferred linkage conformations. An analogous inter-residue hydrogen bond does not exist in (I) due to the axial orientation of O3$_{All}$, yet its internal linkage conformation is very similar to those of (II) and (III).

Comment

As a component of experimental and theoretical studies of the effects of primary structure and solvation on the conformations and dynamics of biologically important oligosaccharides, the title disaccharide, (I), was prepared with single sites of ^{13}C-enrichment at either C1′ or C2′ to permit measurements of its constituent trans-glycoside J_{CH} and J_{CC} values (Bose *et al.*, 1998; Cloran *et al.*,1999; Zhao *et al.*, 2008). Disaccharide (I) is structurally related to methyl β-lactoside [methyl β-D-galactopyranosyl-(1→4)-β-D-glucopyranoside] methanol monosolvate, (II) (Stenutz *et al.*, 1999), and methyl β-cellobioside [methyl β-D-glucopyranosyl-(1→4)-β-D-glucopyranoside] methanol monosolvate, (III) (Ham & Williams, 1970); these disaccharides differ only in the configuration at C3 and/or C4′. The structural differences at C3 occur near the internal glycosidic linkage, and thus might be expected to affect linkage conformation in solution and in the solid state. This situation contrasts with that involving structural changes at C2 discussed in a recent comparison of the crystal structures of methyl α-lactoside [methyl β-D-galactopyranosyl-(1→4)-α-D-glucopyranoside], (IV) (Pan *et al.*, 2005), and methyl β-D-galactopyranosyl-(1→4)-α-D-mannopyranoside methanol 0.375-solvate, (V) (Hu *et al.*, 2010). While the latter change is more remote from the internal linkage, X-ray crystal structures of (IV) and (V) show significant differences in linkage conformation. In solution, however, (IV) and (V) appear to assume virtually identical internal linkage conformations, based on analyses of trans-glycoside J-couplings (Klepach & Serianni, private communication). As shown herein, in the crystalline state, the internal linkage conformations in (I) and (II) are very similar, whereas in solution the conformations differ (Klepach & Serianni, private communication). Overall results from (I)–(V) show that the structural characteristics of related disaccharides in the solid state cannot be expected to mimic those found in solution, due to the inherent flexibility of the glycosidic linkage and the effects of solvation and/or crystal packing forces in the solution and solid states, respectively.

(I) (methyl β-Gal-(1→4)-β-All)

(II) (methyl β-lactoside)

(III) (methyl β-cellobioside)

(IV) (methyl α-lactoside)

(V) (methyl β-Gal-(1→4)-α-Man)

An analysis of endocyclic C—C bond lengths in (I)–(III) shows that $r_{C1,C2}$ [1.523 (7) Å] [in the following discussion, averages were calculated using the appropriate molecular

doi:10.1107/S0108270110029471
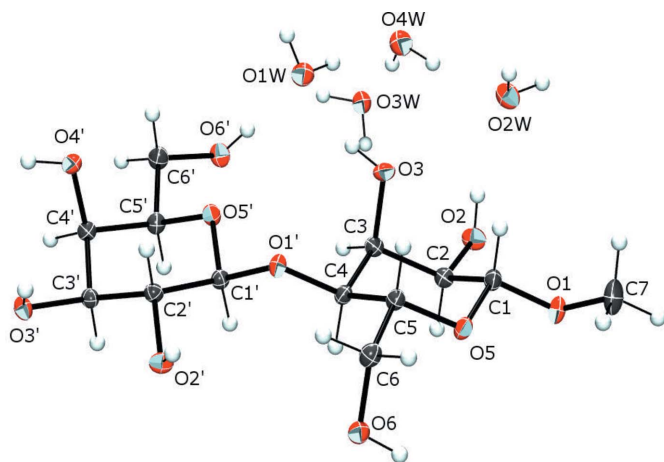
Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are depicted at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Figure 2
A packing diagram for (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

parameter in both residues of (I)–(III); *e.g.* for $r_{C1,C2}$, both $r_{C1,C2}$ and $r_{C1',C2'}$ (a total of six values) were used to obtain 1.523 (7) Å] is very similar to the remaining C—C bond lengths in the aldopyranosyl ring constituents [1.529 (5) Å], whereas $r_{C5,C6}$ appears shorter [1.513 (6) Å] than all the other endocyclic C—C bonds. These results compare very favorably with trends reported recently from structural comparisons of (IV), (V) and several methyl aldopyranosides (Hu *et al.*, 2010), where values of 1.524 (6), 1.528 (7) and 1.518 (3) Å, respectively, were observed.

The endocyclic C—O bonds in (I)–(III) (*i.e.* $r_{C1,O5}$ and $r_{C5,O5}$) are 1.429 (7) Å, in good agreement with the value of 1.428 (9) Å observed in (IV), (V) and related monosaccharides (Hu *et al.*, 2010). The exocyclic C—O bonds not involving the anomeric C atoms and other C atoms in a glycosidic linkage are 1.424 (9) and 1.426 (2) Å for equatorial and axial bonds, respectively; as reported previously (Hu *et al.*, 2010), non-anomeric bond orientation exerts essentially no discernible effect on C—O bond length. The anomeric C1—O1 bonds (all equatorial) are 1.387 (6) Å for the βGal*p* and βGlc*p* residues of (I)–(III). In contrast, $r_{C1',O1'}$ in (I) (βGal*p* residue) is 1.4014 (15) Å, which is lengthened relative to the remaining C—O bonds involving anomeric C atoms. The remaining C—O bonds, $r_{C4,O1'}$, at 1.434 (5) Å, are slightly longer than the other endocyclic equatorial C—O bonds in (I)–(III), although the elongation appears considerably reduced compared with observations made in (IV) and (V) (Hu *et al.*, 2010).

The internal glycosidic C1'—O1'—C4 bond angles in (I)–(III) [115.6 (7)°] are larger than the related C1—O1—C7 bond angles [113.0 (7)°], presumably due to the greater steric demands of the internal linkage.

Inter-residue (intramolecular) hydrogen bonding is not observed in (I). In (II) and (III), the interatomic distance between atoms O3 and O5' of 2.763 (1) Å is consistent with the presence of a hydrogen bond between atoms O3H (donor) and O5' (acceptor). In (I), the corresponding distance is 3.4475 (14) Å.
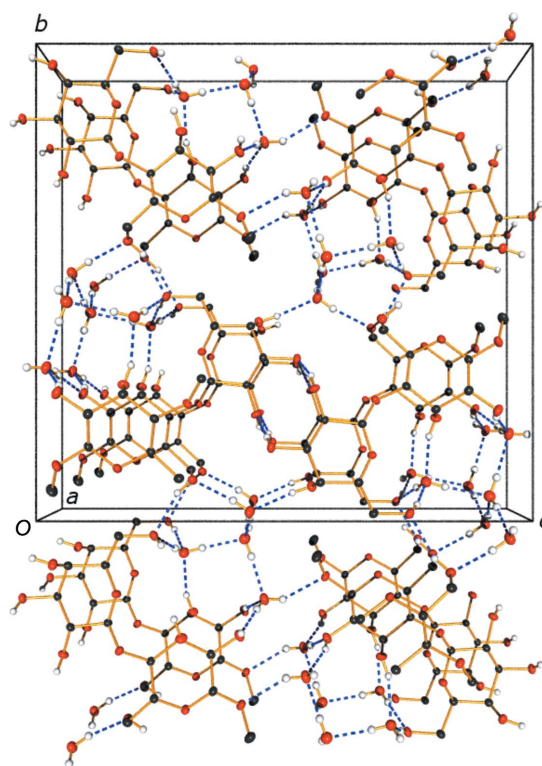
The endocyclic torsion angles in the pyranosyl rings of (I)–(III) differ considerably from the idealized 60° expected for perfect $^4C_1$ chair forms; for example, torsion angles involving a terminal C1/C1' vary from 44 to 70° (absolute values) (Table 1). These deviations suggest the existence of $^4C_1$ chair forms that deviate from ideal conformations. Calculation of Cremer–Pople puckering parameters (Cremer & Pople, 1975) for the aldopyranosyl rings of (I)–(III) are given in Table 3. The extent of the distortion, embodied in θ, is smaller for the βGal*p* residues of (I) and (II) and the βAll*p* residue of (I) (θ values ranging from 2.4–4.7°) than for the βGlc*p* residues of (II) and (III) (θ values ranging from 10.0–12.7°). The direction of the distortion, embodied in φ, also varies widely. The βGal*p*, βGlc*p* and βGlc*p*OMe residues of (I), (II) and (III), respectively, have similar φ values [336 (5)°], suggesting a distortion towards $^0S_2$ forms. The βAll*p* residue of (I), with φ near 70°, is distorted towards $B_{1,4}$, while the βGal*p* and βGlc*p* residues of (II) and (III) [$\varphi = 24$ (6)°] are distorted towards $^3S_1$. Overall, less pyranosyl ring distortion is observed in (I) than in (II) and (III), despite the presence of an axial atom O3 in the former.

The internal glycosidic torsion angles are very similar in (I)–(III): −92 (4)° for φ' (O5'—C1'—O1'—C4) and −161.0 (3)° for ψ' (C1'—O1'—C4—C5). The variability in φ' is considerably larger than that in ψ', which might be unexpected since φ' is controlled mainly by stereoelectronic and steric effects, whereas ψ' is controlled largely by sterics. The absence of an inter-residue hydrogen bond between atoms O3H and O5' in

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(I) does not significantly alter the linkage conformation relative to (II) and (III), in which this hydrogen bond is observed. In comparison, the internal glycosidic torsion angles in (IV) and (V) are −93.6 and −68.2 (3)°, respectively, for φ', and −144.8 and −123.9 (2)°, respectively, for ψ', despite internal glycosidic linkages identical to those found in (I)–(III) (*i.e.* β-Gal-(1→4) linkages to Glc*p*, Man*p* or All*p* residues). It is noteworthy that φ' in (V) deviates significantly from the related torsion angles in (I)–(IV), whereas the ψ' values in (IV) and especially in (V) deviate considerably from the corresponding values observed in (I)–(III). Thus, within (I)–(V), the φ' values range from −68 to −96°, with four values clustered near −90°, whereas the ψ' values range from −124 to −161°, with three values clustered near −160°. While the structural difference at C2 in (IV) and (V) is more remote from the internal glycosidic linkage than that at C3 in (I) and (II), the effect on linkage conformation appears greater in the former.

The exocyclic hydroxymethyl conformation in (I)–(III) is similar, with ω averaging −57 (4)° and ω' averaging 57 (4)°. These values correspond to a *gg* conformation (H5 *anti* to O6) for ω and a *gt* conformation for ω' (C4′ *anti* to O6′).

Methyl β-lactoside, (II), crystallizes as a methanol solvate, whereas (I) crystallizes as a tetrahydrate. This difference exerts a major effect on the hydrogen-bonding networks displayed by both molecules in the crystalline state. Five of the 11 O atoms in (I), namely O3, O5, O1′, O4′ and O5′, do not act as hydrogen-bond acceptors. Of the remaining six O atoms, only two serve as mono-acceptors to other adjacent molecules of (I), namely O2′ and O3′. The remaining four O atoms are hydrogen bonded to water, with three (O1, O2 and O6) serving as mono-acceptors, and O6′ serving as a double hydrogen-bond acceptor to water and an adjacent molecule of (I). All H atoms bonded to O atoms in (I) participate in hydrogen bonding, with H atoms on O6, O2′ and O3′ bonded to adjacent molecules of (I), and H atoms on O2, O3, O4′ and O6′ hydrogen bonded to water. The four water molecules are fully hydrogen bonded (*i.e.*, each serves as a dual acceptor and donor) to other water molecules or to molecules of (I). The water molecules are all located within a channel bounded by molecules of (I) within the lattice. This channel runs through the lattice parallel to the *a* axis (Fig. 2). Thus, the solvent water molecules in (I) play a dominant role in the crystal packing arrangement by hydrogen bonding extensively with themselves and with multiple molecules of (I). The overall hydrogen-bonded connectivity results in a three-dimensional network.

In (II), by comparison with (I), all hydroxy H atoms bonded to O atoms are involved in intermolecular hydrogen bonding as donors, except for atom O3 which participates in intramolecular hydrogen bonding to atom O5′. All O atoms in (II), including atom O3, serve as mono-acceptors, except for O1, O5, O1′ and O4′, which are not hydrogen bonded in the crystal structure. The methanol hydroxy H atom is hydrogen bonded to atom O6 of one molecule of (II), while the methanol O atom serves as a mono-acceptor to the H atom on O4′ of an adjacent molecule of (II).

Experimental

The crystal structure of (I) was determined using a ^{13}C-labeled form of the molecule, which was prepared according to a nine-step synthesis described in supplementary Fig. 3; full details are available in the archived CIF. The final purified product, obtained initially as a syrup after Dowex 50 × 8 (200–400 mesh) (Ca^{2+}) chromatography (Angyal *et al.*, 1979), was dissolved in a small amount of water, and the solution was concentrated by evaporation at room temperature. A small crystal of (I) was harvested for use in the structure determination.

Crystal data

$C_{13}H_{24}O_{11} \cdot 4H_2O$	$Z = 4$
$M_r = 428.39$	Synchrotron radiation
Orthorhombic, $P2_12_12_1$	$\lambda = 0.7749$ Å
$a = 4.7071$ (5) Å	$\mu = 0.12$ mm^{-1}
$b = 20.125$ (2) Å	$T = 150$ K
$c = 20.903$ (2) Å	$0.10 \times 0.04 \times 0.04$ mm
$V = 1980.1$ (4) Å^3	

Data collection

Bruker APEXII diffractometer	28515 measured reflections
Absorption correction: empirical (using intensity measurements) (*SADABS*; Sheldrick, 2008)	5985 independent reflections
	5510 reflections with $I > 2\sigma(I)$
$T_{min} = 0.988$, $T_{max} = 0.995$	$R_{int} = 0.078$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.102$	
$S = 1.06$	
5985 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
278 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
8 restraints	Absolute structure: Flack (1983), with 2548 Friedel pairs
	Flack parameter: −0.1 (5)

Due to the small size and light-atom nature of the sample, synchrotron radiation was employed to perform the diffraction study. Despite several recrystallization attempts, it was not possible to obtain larger crystals. The instrumentation is outlined in the tables. The radiation wavelength was tuned using a channel-cut Si(111) crystal monochromator. The instrumental set-up is identical to a laboratory source, differing only in the orientation of the goniometer (vertical *cf* horizontal), due to the highly polarized X-ray source of the Advanced Light Source at Lawrence Berkeley National Laboratory. Data collection, reduction and structure solution and refinement (with appropriate neutral-atom scattering factors) are otherwise as would normally be undertaken at a standard X-ray facility.

Due to the use of intense synchrotron radiation, two reflections overloaded the detector, even when employing high-speed retakes or attenuation of the beam. The software assigns a zero intensity value for these reflections and it becomes immediately obvious in the F_o^2 versus F_c^2 analysis that they are misassigned; they were not included in the refinement.

H atoms bonded to C atoms were included in geometrically calculated positions, with C—H = 0.98–1.00 Å, and U_{iso}(H) = $1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(C) for all others. Hydroxy H atoms were initially included in their observed positions and subsequently constrained, allowing for re-orientation to optimize any potential hydrogen-bond interactions. H atoms on water molecules were all located in a difference Fourier map and initially included in

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O2—H2⋯O2W	0.84	1.94	2.7489 (16)	160
O3—H3⋯O1W	0.84	1.99	2.7992 (15)	163
O6—H6⋯O6′i	0.84	2.01	2.8057 (13)	157
O2′—H2′⋯O3′ii	0.84	1.90	2.7149 (14)	162
O3′—H3′⋯O2′ii	0.84	1.97	2.8117 (14)	179
O4′—H4′⋯O3Wiii	0.84	1.98	2.8152 (14)	173
O6′—H6′⋯O1W	0.84	1.90	2.7307 (16)	171
O1W—H1WA⋯O4W	0.83 (1)	1.92 (1)	2.7418 (16)	176 (2)
O1W—H1WB⋯O6′iv	0.85 (1)	1.98 (1)	2.8198 (16)	178 (2)
O2W—H2WA⋯O1v	0.82 (1)	2.15 (1)	2.8972 (15)	152 (2)
O2W—H2WB⋯O2iv	0.84 (1)	1.91 (1)	2.7561 (16)	177 (2)
O3W—H3WA⋯O4Wvi	0.84 (1)	1.97 (1)	2.8046 (16)	174 (2)
O3W—H3WB⋯O6vii	0.84 (1)	1.95 (1)	2.7652 (14)	166 (2)
O4W—H4WA⋯O3W	0.84 (1)	1.94 (1)	2.7748 (17)	172 (2)
O4W—H4WB⋯O2W	0.84 (1)	1.95 (1)	2.7800 (16)	170 (2)

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

Table 2
Comparison of structural parameters in (I)–(III) (Å, °).

Parameter	(I)	(II)	(III)
Bond lengths			
C1—C2	1.5264 (18)	1.516 (3)	1.513 (6)
C2—C3	1.5272 (18)	1.519 (3)	1.528 (6)
C3—C4	1.5365 (17)	1.531 (3)	1.533 (6)
C4—C5	1.5321 (17)	1.530 (3)	1.528 (6)
C5—C6	1.5163 (18)	1.508 (3)	1.505 (6)
C1′—C2′	1.5286 (17)	1.527 (3)	1.526 (6)
C2′—C3′	1.5234 (17)	1.531 (3)	1.534 (6)
C3′—C4′	1.5309 (17)	1.521 (3)	1.529 (6)
C4′—C5′	1.5327 (17)	1.521 (3)	1.531 (6)
C5′—C6′	1.5205 (17)	1.511 (3)	1.515 (6)
C1—O1	1.3944 (15)	1.384 (3)	1.379 (6)
C1—O5	1.4267 (15)	1.413 (3)	1.434 (5)
C2—O2	1.4274 (15)	1.418 (3)	1.439 (5)
C3—O3	1.4274 (17)	1.421 (3)	1.430 (5)
C5—O5	1.4412 (15)	1.428 (3)	1.432 (6)
C6—O6	1.4357 (18)	1.424 (3)	1.440 (6)
C1′—O1′	1.4014 (15)	1.387 (3)	1.390 (5)
C1′—O5′	1.4271 (15)	1.425 (3)	1.432 (5)
C2′—O2′	1.4286 (14)	1.414 (3)	1.416 (5)
C3′—O3′	1.4369 (14)	1.422 (3)	1.431 (5)
C4′—O4′	1.4265 (17)	1.423 (3)	1.410 (5)
C5′—O5′	1.4339 (15)	1.432 (3)	1.429 (6)
C6′—O6′	1.4418 (16)	1.426 (3)	1.434 (5)
C4—O1′	1.4292 (15)	1.437 (3)	1.437 (5)
O3⋯O5′	3.448 (1)	2.764 (2)	2.762†
Bond angles			
C1′—O1′—C4	114.77 (10)	116.2 (2)	115.8 (3)
C1—O1—CH₃	112.30 (11)	113.7 (2)	113.1 (3)
Torsion angles			
C1′—C2′—C3′—C4′	−53.48 (14)	−54.8 (2)	−51.0†
C1—C2—C3—C4	−56.77 (14)	−44.2 (3)	−45.0
C1—O5′—C5′—C4′	63.16 (13)	65.0 (2)	67.4
C1—O5—C5—C4	61.78 (13)	67.6 (2)	70.1
C2′—C1′—O1′—C4 (φ')	144.74 (10)	153.8 (2)	152.0
C2—C1—O1—CH₃ (φ)	165.10 (11)	164.2 (2)	166.8
C1′—O5′—C4—C3 (ψ')	77.55 (13)	78.4 (2)	80.3
C1′—O1′—C4—C5 (ψ')	−160.93 (10)	−161.3 (2)	−160.7
O5′—C1′—O1′—C4 (φ')	−96.40 (12)	−88.4 (2)	−91.1
O5—C1—O1—CH₃ (φ)	−76.36 (14)	−77.4 (3)	−76.1
H1′—C1′—O1′—C4 (φ')	23.9	31.9	24.3
C1′—O1′—C4—H4 (ψ')	−43.6	−43.7	−47.7
O5′—C5′—C6′—O6′ (ω')	60.84 (14) (gt)‡	57.4 (2) (gt)	52.4 (gt)
O5—C5—C6—O6 (ω)	−61.92 (14) (gg)	−54.6 (2) (gg)	−55.1 (gg)

† s.u. values on intramolecular hydrogen-bond lengths and torsion angles in (III) were not reported. ‡ gg is gauche–gauche and gt is gauche–trans.

Table 3
Cremer–Pople puckering parameters in (I)–(III).

Compound	θ (°)	φ (°)	Q (Å)	q_2 (Å)	q_3 (Å)
(I), βGalp	2.8	335.3	0.5807	0.0283	0.5800
(I), βAllp	2.4	69.6	0.6018	0.0254	0.6012
(II), βGalp	4.7	28.2	0.5948	0.0485	0.5928
(II), βGlcp	12.0	341.5	0.5579	0.1159	0.5457
(III), βGlcOMe	12.7	330.9	0.5766	0.1269	0.5625
(III), βGlcp	10.0	19.5	0.5909	0.1026	0.5819

those positions. They were subsequently refined with mild O—H bond-distance restraints [O—H = 0.84 (1) Å]. All H atoms bonded to O atoms were treated isotropically, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

Data collection: *APEX2* (Bruker Nonius, 2009); cell refinement: *SAINT* (Bruker Nonius, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008), *POV-RAY* (Cason, 2003) and *DIAMOND* (Brandenburg, 2009); software used to prepare material for publication: *XCIF* (Sheldrick, 2008), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2010).

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