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Note

# Molecular and crystal structure of methyl 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl-(1 → 2)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-mannopyranoside ☆

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Acylated β-D-xylopyranose derivatives are known to exist as a mixture of the $^1C_4$ and $^4C_1$ conformers in rapid equilibrium in solution [3]. Several conformations have also been reported for β-D-xylopyranose derivatives in the solid state [4]. In the course of our study to investigate the functions of unique oligosaccharides expressed on the spermatozoa of a bivalve, we synthesized β-D-xylopyranose-containing oligosaccharides as intermediates. We also reported these compounds existed as mixtures of the $^1C_4$ and $^4C_1$ conformers in $CDCl_3$ solution at ambient temperature [5]. Among such compounds, we could obtain the crystals of a fully protected β-D-Xyl-(1 → 2)-α-D-Man derivative that was ideal to confirm the structure. Now the molecular structure of the disaccharide as determined by X-ray crystallography is reported herein.

☆ Part XI. Synthetic Studies on Oligosaccharides of a Glycolipid from the Spermatozoa of Bivalves. For Parts IX and X, see refs [1,2].

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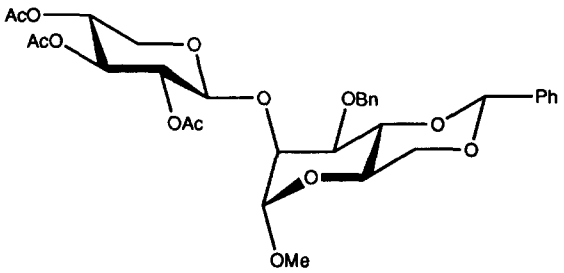
## 1. Experimental

Methyl 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl-(1 → 2)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-mannopyranoside (**1**) crystallized in the space group $P2_12_12_1$. Further crystallographic data are given in Table 1.




**1**

A colorless needle suitable for X-ray analysis was obtained by the vapor diffusion of petroleum ether into a dichloromethane solution of compound **1**. Intensity data were corrected on an Enraf–Nonius CAD4 Kappa goniometer using Mo$K_\alpha$ radiation. Unit-cell parameters were determined by least-squares refinement using 25 accurately centered

Table 1
Crystal data and X-ray diffraction analysis data for methyl 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl-(1 → 2)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-mannopyranoside (**1**)

| Crystal data | |
|---|---|
| $C_{32}H_{38}O_{13}$; mol. wt 630.7; mp 195–196°; | |
| Crystal dimensions $0.27\times0.45\times0.96/mm^3$; space group $P2_12_12_1$; | |
| $Z = 4$ | |
| Cell dimensions, $a = 8.968(1)$, $b = 37.702(3)$, $c = 9.573(1)$ Å | |
| Volume = 3236.9(4) Å$^3$ | |
| $D = 1.294$ g/cm$^3$ | |
| *Experimental and refinement data* | |
| Radiation | graphite monochromated Mo$K_\alpha$ |
| Temperature | 293 K |
| $2\theta$ range | 3–50° |
| Scan technique | $\omega-2\theta$ |
| Scan range ($\omega$/°) | $0.70+0.35\tan\theta$ |
| Reflection measured | 6170 |
| Reflections with $F_o > 2\sigma(F_o)$ | 2518 |
| Number of refined parameters | 406 |
| Final refinement values | $R = 0.064$; $R_w = 0.060$ [a] |
| Diffractometer | Enraf–Nonius CAD4 |

[a] $R_w = [\Sigma w(|F_o|-|F_o|)^2/\Sigma w(F_o)^2]^{1/2}$ where $w = 1$.

reflections ($19° < 2\theta < 28°$). Intensity data were collected for standard Lorentz and polarization effects but not for absorption. A total of 2518 reflections having $|F_o| > 2\sigma(|F_o|)$ was retained as observed and used in the solution and refinement of the structure.

The structure was solved by direct methods and refined by difference Fourier and full-matrix least-squares techniques. [1] All non-hydrogen atoms were refined with anisotropic thermal parameters, and hydrogen atoms were included in their calculated positions. Final cycles of full-matrix least-squares refinement were carried to convergence at $R = 0.064$ and $R_w = 0.060$. [2] The final difference Fourier map was judged featureless. The atomic coordinates for all non-hydrogen atoms with the isotropic equivalent thermal factors are given in Table 2. [3] The molecular structure is depicted in Fig. 1. The bond lengths, bond angles, and selected torsion angles are shown in Table 3.

## 2. Results and discussion

In a previous study [5], we reported the syntheses of xylose-containing oligosaccharides. Despite the difficulty to assign the structures as actually having β-D-configuration, we concluded that the desired linkage was formed in the coupling reaction. It was strongly supported by the neighboring participation of the C-2 acetyloxy group to form the β-isomer irrespective of the conformation of xylose. Also, none of the coupling constants for those compounds were close to those of the α anomers of xylopyranosides. Finally, it was shown by $^1$H NMR analysis that the xylosyl residue in disaccharide **1**, which was a key intermediate leading to other xylose-containing oligosaccharides, exists as a mixture of $^1C_4$ (25%) and $^4C_1$ (75%) conformers in $CDCl_3$ at ambient temperature. The molecular structure of **1** (Fig. 1) clearly shows a xylosyl residue in the $^4C_1$ conformation, with $Q = 0.53$ Å, $\varphi = 1.3°$, $\theta = 14.6°$, in the crystalline state with a β-D-glycosidic linkage. The puckering parameters [6,7] for the xylosyl residue are well described by both the ring torsional angles (52.4°, O-5′–C-1′–C-2′–C-3′; 41.9°, C-1′–C-2′–C-3′–C-4′; 40.0°, C-2′–C-3′–C-4′–C-5′; −51.6°, C-3′–C-4′–C-5′–O-5′; 65.2°, C-4′–C-5′–O-5′–C-1′; and −64.2°, C-5′–O-5′–C-1′–C-2′), and large bond angles at C-3′ (112.9°, C-2′–C-3′–C-4′).

A fused-chair conformation was found for the mannose residue with the benzylidene acetal at C-4 and C-6. The puckering parameters were $Q = 0.59$ Å, $\varphi = 250.2°$, $\theta = 2.9°$

[1] Programs used in this study included local modifications of A. Altomare et al.'s SIR92, Johnson's ORTEP II, and Scheidt and Haller's (University of Notre Dame) versions of ORFLS and ORFFE, originated by Busing, Martin, and Levy.

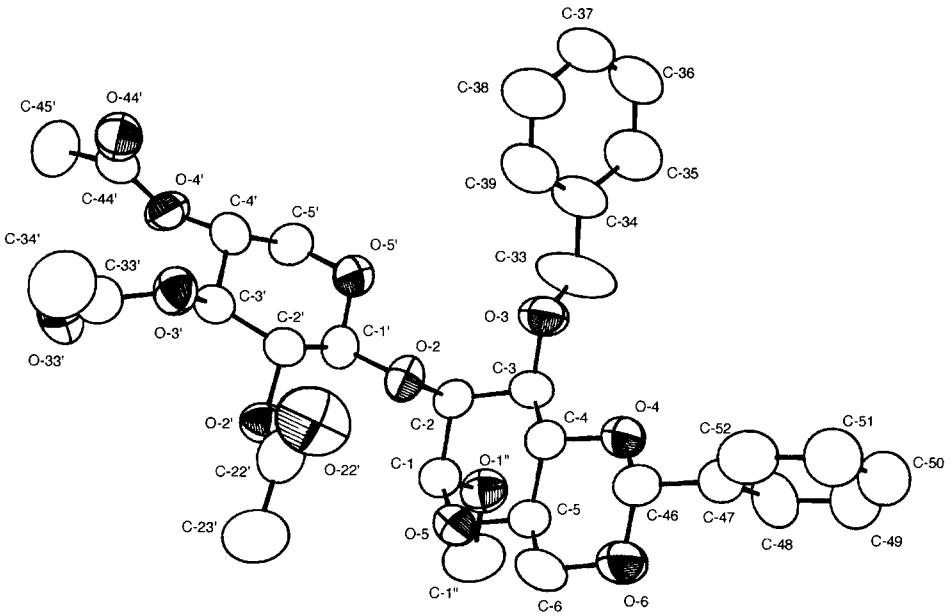
[2] The atomic scattering factors were taken from *International Tables for X-ray Crystallography*, Vol. IV, Kynoch Press, Birmingham (1974). $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w(F_o)^2]^{1/2}$ with unit weight.

[3] Tables of the anisotropic temperature factors for non-hydrogen atoms, the idealized atomic coordinates for hydrogen atoms, individual bond distance and angles, and the observed and calculated structure factors have been deposited with the Cambridge Crystallographic Data Centre and may be obtained from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

Table 2
Atomic coordinates and equivalent isotropic thermal parameters for methyl 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl-(1 → 2)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-mannopyranoside (**1**) [a]

| Atom | *x* | *y* | *z* | $B_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| C-1 | 0.1105(8) | 0.4056(2) | 0.3191(7) | 5.0(2) |
| C-2 | 0.2534(8) | 0.3838(2) | 0.3480(7) | 4.7(2) |
| C-3 | 0.3734(8) | 0.4082(2) | 0.4104(8) | 5.0(2) |
| C-4 | 0.3977(8) | 0.4391(2) | 0.3128(7) | 4.6(2) |
| C-5 | 0.2487(8) | 0.4588(2) | 0.2916(7) | 5.0(1) |
| C-6 | 0.2780(7) | 0.4881(2) | 0.1901(6) | 6.8(1) |
| C-1′ | 0.2616(8) | 0.3369(2) | 0.1835(7) | 4.3(2) |
| C-2′ | 0.2933(7) | 0.3320(2) | 0.0312(7) | 4.1(1) |
| C-3′ | 0.2729(7) | 0.2948(2) | −0.0202(7) | 4.0(1) |
| C-4′ | 0.3336(8) | 0.2674(2) | 0.0799(7) | 4.7(2) |
| C-5′ | 0.2997(7) | 0.2773(2) | 0.2306(7) | 5.7(1) |
| C-1″ | −0.0881(8) | 0.4337(2) | 0.4482(7) | 8.7(2) |
| O-2 | 0.3065(5) | 0.3708(1) | 0.2188(5) | 4.8(1) |
| O-3 | 0.5114(6) | 0.3894(1) | 0.4231(5) | 5.9(1) |
| O-4 | 0.4993(5) | 0.4638(1) | 0.3745(5) | 5.0(1) |
| O-5 | 0.1405(5) | 0.4356(1) | 0.2303(5) | 5.2(1) |
| O-6 | 0.3911(5) | 0.5117(1) | 0.2536(5) | 6.5(1) |
| O-2′ | 0.1956(5) | 0.3545(1) | −0.0475(5) | 4.6(1) |
| O-3′ | 0.3481(6) | 0.2933(1) | −0.1522(5) | 5.1(1) |
| O-4′ | 0.2618(5) | 0.2335(1) | 0.0586(5) | 5.1(1) |
| O-5′ | 0.3445(6) | 0.3119(1) | 0.2608(5) | 5.7(1) |
| O-1″ | 0.0565(6) | 0.4158(1) | 0.4507(6) | 6.0(1) |
| C-33 | 0.5432(8) | 0.3774(2) | 0.5504(8) | 11.1(2) |
| C-34 | 0.6869(9) | 0.3553(2) | 0.5458(9) | 6.3(2) |
| C-35 | 0.769(1) | 0.3529(2) | 0.6655(9) | 8.0(2) |
| C-36 | 0.897(1) | 0.3314(2) | 0.664(1) | 8.3(2) |
| C-37 | 0.939(1) | 0.3146(3) | 0.5453(9) | 8.0(3) |
| C-38 | 0.858(1) | 0.3185(3) | 0.427(1) | 10.3(3) |
| C-39 | 0.729(1) | 0.3383(3) | 0.430(1) | 8.8(3) |
| C-46 | 0.5257(9) | 0.4920(2) | 0.2781(9) | 5.6(2) |
| C-47 | 0.6318(9) | 0.5171(2) | 0.3445(8) | 5.2(2) |
| C-48 | 0.6006(9) | 0.5341(2) | 0.4683(9) | 6.6(2) |
| C-49 | 0.698(1) | 0.5598(2) | 0.5267(9) | 7.5(2) |
| C-50 | 0.830(1) | 0.5665(2) | 0.4597(9) | 7.2(2) |
| C-51 | 0.8667(9) | 0.5501(2) | 0.3370(9) | 7.6(2) |
| C-52 | 0.769(1) | 0.5244(2) | 0.276(1) | 7.2(2) |
| C-22′ | 0.2525(8) | 0.3813(2) | −0.1221(7) | 5.9(2) |
| C-23′ | 0.139(1) | 0.4022(2) | −0.1953(7) | 9.0(2) |
| C-33′ | 0.2846(9) | 0.2769(2) | −0.2597(9) | 6.5(2) |
| C-34′ | 0.374(1) | 0.2782(2) | −0.3873(9) | 10.2(2) |
| C-44′ | 0.3231(9) | 0.2102(2) | −0.0264(9) | 5.2(2) |
| C-45′ | 0.230(1) | 0.1761(2) | −0.038(1) | 7.6(2) |
| O-22′ | 0.3835(8) | 0.3861(2) | −0.1297(8) | 9.9(2) |
| O-33′ | 0.1651(8) | 0.2611(2) | −0.2494(7) | 8.4(2) |
| O-44′ | 0.4363(6) | 0.2152(2) | −0.0910(6) | 6.7(2) |

[a] Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)\times[a^2\times B(1,1)+b^2\times B(2,2)+c^2\times B(3,3)+ab(\cos\gamma)\times B(1,2)+ac(\cos\beta)\times B(1,3)+bc(\cos\alpha)\times B(2,3)]$.




for the mannose moiety and $Q = 0.63$ Å, $\varphi = 329.6°$, $\theta = 3.3°$ for the dioxolane ring, respectively. The phenyl ring of the benzylidene group is oriented approximately at a right angle to the chair ring; the dihedral angle of the two mean planes was 88.1°.

The torsion angles around the glycosidic bonds are expressed by $\phi$ and $\psi$ and are used to describe the relative orientation of each pyranose ring, which were $\phi = -76.6°$ (O-5′–C-1′–O-2–C-2) and $\phi = -94.8°$ (C-1′–O-2–C-2–C-1). The bond angle for the glycosidic linkage between xylose and mannose residues ($\tau$ = C-1′–O-2–C-2) was found to be 115.9°. The observed large difference in $\phi$ angle (ca. 60°) between compound **1** and the closely related protected β-D-Xyl-(1 → 2)-β-D-Man derivative [8] is considered due to the steric factor of the substituent groups on C-1 (the orientation of methoxy group) and C-3 (benzyl vs acetyl). The O-5–C-1–O-1″–C-1″ torsion angle for the α-methyl glycoside of the mannose residue was 64.3°, and the bridge angle C-1–O-1″–C-1″ was 114.7°.

Other notable structural parameters in the present molecule are as follows: the C-5–C-6 (1.493 Å) and C-2′–C-3′ bonds (1.49 Å) are shorter than average $sp^3$ C–C bonds. A significantly short C–O bond length (1.332 Å) is observed for O-3–C-33 other than equatorially oriented glycosidic linkage (C-1′–O-2; 1.382 Å).

Table 3
Bond lengths (Å) and bond angles (°), and selected torsion angles (°) for methyl 2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl-(1 → 2)-3-*O*-benzyl-4,6-*O*-benzylidene-α-D-mannopyranoside (**1**) [a]

| Bond lengths (Å) | | | |
|---|---|---|---|
| C-1–C-2 | 1.55(1) | O-5′–C-1′ | 1.411(8) |
| C-2–C-3 | 1.54(1) | O-5′–C-5′ | 1.395(9) |
| C-3–C-4 | 1.510(9) | O-1″–C-1 | 1.404(9) |
| C-4–C-5 | 1.542(9) | O-1″–C-1″ | 1.462(9) |
| C-5–C-6 | 1.493(10) | C-33–C-34 | 1.535(11) |
| C-1′–C-2′ | 1.50(1) | C-34–C-35 | 1.365(11) |
| C-2′–C-3′ | 1.49(1) | C-34–C-39 | 1.338(12) |
| C-3′–C-4′ | 1.512(9) | C-35–C-36 | 1.406(13) |
| C-4′–C-5′ | 1.52(1) | C-36–C-37 | 1.357(13) |
| O-2–C-1′ | 1.382(7) | C-37–C-38 | 1.353(13) |
| O-2–C-2 | 1.413(8) | C-38–C-39 | 1.375(13) |
| O-3–C-3 | 1.431(9) | C-46–C-47 | 1.48(1) |
| O-3–C-33 | 1.332(10) | C-47–C-48 | 1.376(11) |
| O-4–C-4 | 1.430(8) | C-47–C-52 | 1.418(11) |
| O-4–C-46 | 1.429(9) | C-48–C-49 | 1.418(11) |
| O-5–C-1 | 1.442(9) | C-49–C-50 | 1.374(13) |
| O-5–C-5 | 1.432(8) | C-50–C-51 | 1.368(13) |
| O-6–C-46 | 1.436(9) | C-51–C-52 | 1.428(12) |
| O-6–C-6 | 1.480(9) | C-22′–C-23′ | 1.467(13) |
| O-2′–C-2′ | 1.434(7) | C-33′–C-34′ | 1.461(13) |
| O-2′–C-22 | 1.34(1) | C-44′–C-45′ | 1.539(9) |
| O-3′–C-3′ | 1.433(7) | O-22′–C-22′ | 1.191(10) |
| O-3′–C-33 | 1.33(1) | O-33′–C-33′ | 1.231(11) |
| O-4′–C-4′ | 1.445(8) | O-44′–C-44′ | 1.202(9) |
| O-4′–C-44 | 1.32(1) | | |
| **Bond angles (°)** | | | |
| O-5–C-1–C-2 | 111.6(6) | O-2′–C-2′–C-3′ | 107.9(5) |
| O-1″–C-1–C-2 | 105.7(6) | C-2′–C-3′–C-4′ | 112.9(6) |
| O-1″–C-1–O-5 | 112.2(6) | O-3′–C-3′–C-2′ | 105.7(5) |
| O-2–C-2–C-1 | 107.9(6) | O-3′–C-3′–C-4′ | 111.2(5) |
| O-2–C-2–C-3 | 108.2(6) | C-3′–C-4′–C-5′ | 111.1(6) |
| C-3–C-2–C-1 | 109.5(5) | O-4′–C-4–C-3′ | 110.9(5) |
| C-4–C-3–C-2 | 108.8(6) | O-4′–C-4′–C-5′ | 105.2(6) |
| O-3–C-3–C-2 | 110.1(6) | O-5′–C-5′–C-4′ | 111.7(7) |
| O-3–C-3–C-4 | 108.0(6) | C-1′–O-2–C-2 | 115.9(5) |
| C-3–C-4–C-5 | 109.2(6) | C-33–O-3–C-3 | 115.5(7) |
| O-4–C-4–C-3 | 109.8(6) | C-46–O-4–C-4 | 108.9(5) |
| O-4–C-4–C-5 | 107.0(5) | C-5–O-5–C-1 | 111.4(5) |
| C-6–C-5–C-4 | 106.8(6) | C-46–O-6–C-6 | 109.5(6) |
| O-5–C-5–C-4 | 110.3(5) | C-22′–O-2′–C-2′ | 119.7(6) |
| O-5–C-5–C-6 | 107.6(6) | C-33′–O-3′–C-3′ | 120.0(6) |
| O-6–C-6–C-5 | 107.3(6) | C-44′–O-4′–C-4′ | 119.3(6) |
| O-2–C-1′–C-2′ | 107.4(5) | C-5′–O-5′–C-1′ | 111.4(6) |
| O-2–C-1′–O-5′ | 109.7(5) | C-1–O-1″–C-1″ | 114.7(7) |
| O-5′–C-1′–C-2′ | 109.1(5) | O-3–C-33–C-34 | 109.8(9) |
| C-3′–C-2′–C-1′ | 114.5(6) | C-35–C-34–C-33 | 118.(1) |
| O-2′–C-2′–C-1′ | 108.8(5) | C-39–C-34–C-33 | 121.(1) |

Table 3 (continued)

| Bond angles (°) | | | |
|---|---|---|---|
| C-39–C-34–C-35 | 120.9(8) | C-50–C-49–C-48 | 118.4(9) |
| C-34–C-35–C-36 | 118.3(9) | C-50–C-51–C-52 | 120.7(9) |
| C-37–C-36–C-35 | 120.0(9) | C-51–C-50–C-49 | 121.(1) |
| C-38–C-37–C-36 | 120.(1) | C-47–C-52–C-51 | 119.(1) |
| C-37–C-38–C-39 | 120.(1) | O-2′–C-22′–C-23′ | 113.2(9) |
| C-34–C-39–C-38 | 121.(1) | O-22′–C-22′–C-23′ | 125.1(9) |
| O-4–C-46–C-47 | 107.7(6) | O-22′–C-22′–O-2′ | 121.6(9) |
| O-4–C-46–O-6 | 110.5(6) | O-3′–C-33′–C-34′ | 113.5(9) |
| O-6–C-46–C-47 | 106.3(6) | O-33′–C-33′–C-34′ | 124.(1) |
| C-48–C-47–C-46 | 122.4(8) | O-33′–C-33′–O-3′ | 122.(1) |
| C-48–C-47–C-52 | 118.7(8) | O-4′–C-44′–C-45′ | 111.9(7) |
| C-52–C-47–C-46 | 118.9(8) | O-44′–C-44′–C-45′ | 123.6(7) |
| **Torsion angles (°)** | | | |
| O-1″–C-1–C-2–C-3 | −66.5(1) | C-6–O-6–C-46–O-4 | −62.1(1) |
| O-5–C-1–C-2–C-3 | 55.7(1) | C-46–O-6–C-6–C-5 | 60.9(1) |
| C-1–C-2–C-3–C-4 | −55.3(1) | C-5′–O-5′–C-1′–C-2′ | −64.2(1) |
| C-2–C-3–C-4–C-5 | 57.9(1) | C-1′–O-5′–C-5′–C-4′ | 65.2(1) |
| C-3–C-4–C-5–C-6 | −177.8(1) | C-1″–O-1″–C-1–C-2 | −173.9(1) |
| C-4–C-5–C-6–O-6 | −61.0(1) | C-1″–O-1″–C-1–O-5 | 64.3(1) |
| O-2–C-1′–C-2′–C-3′ | 171.2(1) | C-1′–O-2–C-2–C-3 | 146.9(1) |
| O-5′–C-1′–C-2′–C-3′ | 52.4(1) | C-4–O-4–C-46–O-6 | 64.6(1) |
| C-1′–C-2′–C-3′–C-4′ | −41.9(1) | C-46–O-4–C-4–C-5 | −64.1(1) |
| C-2′–C-3′–C-4′–C-5′ | 40.0(1) | C-1–O-5–C-5–C-4 | 61.2(1) |
| C-3′–C-4′–C-5′–O-5′ | −51.6(1) | C-5–O-5–C-1–C-2 | −59.0(1) |
| C-2–O-2–C-1′–C-2′ | 165.0(1) | C-6–O-6–C-46–O-4 | −62.1(1) |
| C-1′–O-2–C-2–C-1 | −94.8(1) | C-46–O-6–C-6–C-5 | 60.9(1) |
| C-1′–O-2–C-2–C-3 | 146.9(1) | C-5′–O-5′–C-1′–C-2′ | −64.2(1) |
| C-4–O-4–C-46–O-6 | 64.6(1) | C-1′–O-5′–C-5′–C-4′ | 65.2(1) |
| C-46–O-4–C-4–C-5 | −64.1(1) | C-1″–O-1″–C-1–C-2 | −173.9(1) |
| C-1–O-5–C-5–C-4 | 61.2(1) | C-1″–O-1″–C-1–O-5 | 64.3(1) |
| C-5–O-5–C-1–C-2 | −59.0(1) | | |

[a] Numbers in parentheses are estimated standard deviations in the least significant digits.

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