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Methyl 4-*O*-benzyl-6-deoxy-2,3-*O*-isopropylidene-*α*-L-talopyranoside

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.099
Data-to-parameter ratio = 9.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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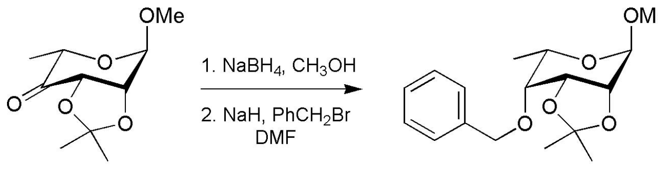
The structure of the title compound, $C_{17}H_{24}O_5$, was determined by single-crystal X-ray diffraction at 100 K.

Comment

As part of our approach to the synthesis of bacterial amino-sugars derived from L-fucosamine, the ketone, (1) (Czernecki *et al.*, 1985; Leriche *et al.*, 2003), was reduced with sodium borohydride to produce a single alcohol (Davis *et al.*, 1999), which was then protected as the corresponding benzyl ether, (2) (Haines, 1972; Gan & Kong, 1995). The essentially complete stereoselectivity observed in the reduction step is caused by the large 2,3-*O*-isopropylidene group blocking the approach of the reducing agent from below the plane of the carbonyl group. The stereochemical course of this reduction is confirmed here by the determination of the crystal structure of compound (2).



(1) (2)

The solid-state structure of (2) was determined by X-ray diffraction at 100 K (Fig. 1) and it was found to crystallize in the noncentrosymmetric space group $P2_12_12_1$. The bond lengths and angles are within the expected ranges. The chair conformation of the glucopyranose ring is slightly distorted due to the annellation of the five-membered ring. The deviation is largest for atom C3, which is dragged markedly towards the 2,3-*O*-isopropylidene protecting group (Fig. 1). The methyl group at atom C5 is found to be in an equatorial position, and the benzyl and methoxy groups are located in axial positions, confirming the expected stereochemical course of the sodium borohydride reaction (Fig. 1).

Experimental

Methyl 4-*O*-benzyl-6-deoxy-2,3-*O*-isopropylidene-*α*-L-talopyranoside, (2), was prepared as described in the literature (Haines, 1972; Gan & Kong, 1995). Crystals suitable for single-crystal X-ray diffraction were obtained from a solution in ethyl acetate and hexanes.

Crystal data

$C_{17}H_{24}O_5$
$M_r = 308.36$
Orthorhombic, $P2_12_12_1$
$a = 9.2495\ (9)$ Å
$b = 10.0013\ (10)$ Å
$c = 17.2359\ (16)$ Å
$V = 1594.4\ (3)$ Å^3
$Z = 4$
$D_x = 1.285$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 7526 reflections
$\theta = 2.4$–$30.5°$
$\mu = 0.09$ mm^{-1}
$T = 100\ (2)$ K
Block, colourless
0.50 × 0.50 × 0.38 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min} = 0.758$, $T_{\max} = 0.96$
19 051 measured reflections

2759 independent reflections
2695 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 30.5°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.099$
$S = 1.15$
2759 reflections
295 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

All H atoms were located in a difference density Fourier map and were refined isotropically. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before refinement. The absolute configuration assignment is based on the known configuration of C atoms retaining their configuration during the synthesis of the azide. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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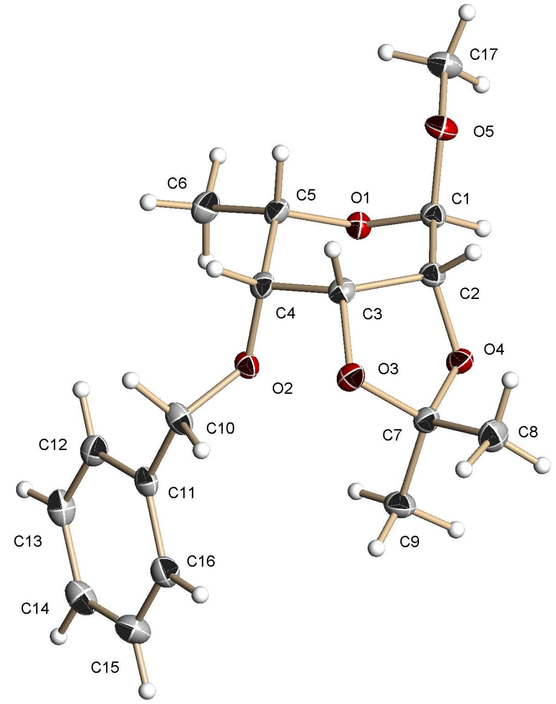

Figure 1
The molecular structure of (2), showing 50% probability displacement ellipsoids.

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