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2,3,4-Tri-*O*-acetyl-*β*-L-fucopyranosyl azide

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The title compound, $C_{12}H_{17}N_3O_7$, crystallizes with two crystallographically independent molecules in the asymmetric unit. Both virtually identical molecules exhibit the expected chair conformation.

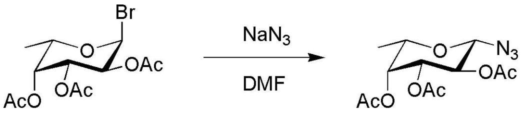
Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.110
Data-to-parameter ratio = 10.3

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

Comment

2,3,4-Tri-*O*-acetyl-*β*-L-fucopyranosyl azide, (2), is formed as a result of an S_N2 displacement of bromide by the azide anion from the corresponding *α*-fucopyranosyl bromide, (1) (Cao & Roy, 1996), or by the Lewis acid-promoted reaction of 1,2,3,4-tetra-*O*-acetyl-L-fucose with $TMSN_3$ (Györgydeák & Szilágyi, 1985). Azide (2) has been employed in Strecker and Ugi chemistry (Kunz *et al.*, 1991) and as a precursor to peptidosaccharide derivatives (Subramaniam, 1997).



(1) (2)

The solid-state structure of (2) was determined by X-ray diffraction at 100 K (Fig. 1). It crystallizes in the non-centrosymmetric space group $P2_1$ with Z = 4 and two crystallographically independent molecules in the asymmetic unit. Only marginal differences are found between the two molecules, the weighted r.m.s. deviation being 0.1016 Å. The derived bond lengths and angles are within the expected ranges, and the six-membered rings exhibit the chair conformation expected for a fucopyranose. The *O*-acetyl groups at

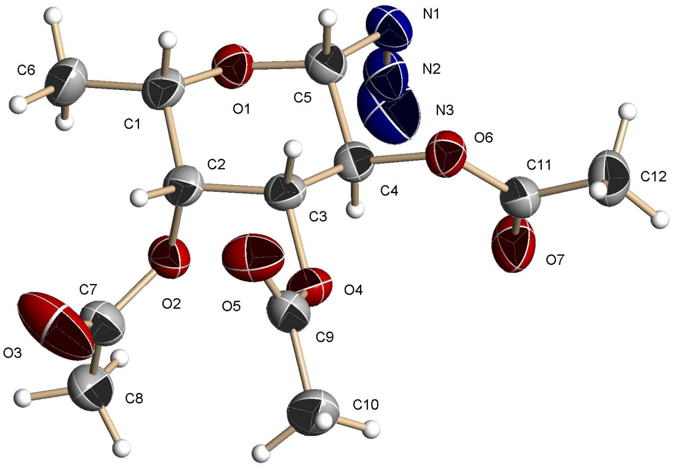


Figure 1
One of the independent molecules of (2), showing 50% probability displacement ellipsoids.

atom C2, and C22 for the second molecule, are located in axial positions, and all other non-H substituents are found in equatorial positions. The acetate groups, as well as the azide group of each molecule, are approximately parallel to each other, as may be seen from Fig. 1.

The β-configuration of (2) with the azide group occupying the equatorial position at the anomeric C atom is clearly confirmed. The azide functional group is close to linear [N1—N2—N3 = 172.2 (3)° and N4—N5—N6 = 171.7 (3)°] and the C5—N1—N2 and C25—N4—N5 angles are 114.1 (2) and 113.3 (2)°, respectively. The N≡N triple-bond distances N2≡N3 and N5≡N6 are 1.120 (3) and 1.112 (3) Å, respectively, and the N—N single bond lengths N1—N2 and N4—N5 are 1.230 (3) and 1.246 (3) Å, respectively.

Experimental

The sample of (2) was prepared according to the method of Cao & Roy (1996). Crystals suitable for single-crystal X-ray diffraction were obtained by slow cooling of a hot ethanol solution.

Crystal data

$C_{12}H_{17}N_3O_7$
$M_r = 315.29$
Monoclinic, $P2_1$
$a = 10.8447\ (6)$ Å
$b = 8.3527\ (5)$ Å
$c = 17.443\ (1)$ Å
$\beta = 96.257\ (1)°$
$V = 1570.62\ (16)$ Å^3
$Z = 4$
$D_x = 1.333$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6342 reflections
$\theta = 2.7–30.6°$
$\mu = 0.11$ mm^{-1}
$T = 200\ (2)$ K
Block, colourless
0.46 × 0.42 × 0.40 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min} = 0.868$, $T_{\max} = 0.96$
16017 measured reflections
4157 independent reflections
3917 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 28.3°$
$h = -14 \rightarrow 14$
$k = -11 \rightarrow 11$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.110$
$S = 1.05$
4157 reflections
405 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.1865P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

All H atoms were placed in calculated positions and refined with a riding model; the C—H distances were 0.98–1.00 Å, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C_{methyl}})$ and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C_{methine}})$. Friedel pairs were merged before refinement. The absolute configuration assignment is based on the known configuration of the C atoms, assuming retention of 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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