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2,3,4,6-Tetra-*O*-acetyl-*β*-D-glucopyranosyl azide

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 10.1

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

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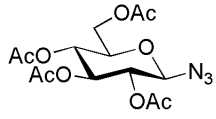
The structure of the title compound, $C_{14}H_{19}N_3O_9$, has been determined at 100 K. The six-membered ring exhibits a chair conformation, and all non-H substituents are found in equatorial positions.

Comment

1,2-*trans*-*β*-Glycosyl azides are versatile precursors for the synthesis of a wide range of glucosylamine derivatives. For example, glucosylamides can be prepared in very good yields and high anomeric equatorial stereoselectivity (Boullanger *et al.*, 2000). The title compound, 2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl azide, (I), was prepared by displacement of bromide from *α*-acetobromoglucose (Ibatullin & Shabalin, 2000). Only one isomer is formed in the reaction, which was concluded to be the *β*-anomer. In order to verify this assumption, the solid-state structure of (I) was determined by X-ray diffraction at 100 K, confirming the presence of the *β*-anomer (Fig. 1).



(I)

The title compound crystallizes in the non-centrosymmetric space group $P2_12_12_1$. The bond lengths and angles are within the expected ranges. The six-membered ring exhibits a chair conformation and all non-H substituents are found in equatorial positions. The geometry of the acetate groups is as expected; the azide functional group itself is close to linear [N1—N2—N3 = 171.4 (2)°] and the angle at C1—N1—N2 is 113.74 (14)°. The shorter terminal N2—N3 bond is 1.119 (3) Å and the bond distance N1—N2 is 1.243 (2) Å.

Experimental

2,3,4,6-Tetra-*O*-acetyl-*β*-D-glucopyranosyl azide was prepared as described previously (Ibatullin & Shabalin, 2000). Crystals suitable for single-crystal X-ray diffraction were grown from hot methanol.

Crystal data

$C_{14}H_{19}N_3O_9$
M_r = 373.32
Orthorhombic, $P2_12_12_1$
a = 7.2970 (3) Å
b = 14.7022 (7) Å
c = 15.8692 (7) Å
V = 1702.48 (13) Å^3
Z = 4
D_x = 1.457 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17 967 reflections
θ = 2.6–30.5°
μ = 0.12 mm^{-1}
T = 100 (2) K
Block, colourless
0.30 × 0.30 × 0.30 mm

Data collection

Bruker AXS SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–2002)
T_{min} = 0.803, T_{max} = 0.96
21 165 measured reflections
2935 independent reflections
2914 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 30.5°
$h = -10 \rightarrow 10$
$k = -20 \rightarrow 20$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.20
2935 reflections
292 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.3882P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

All H atoms were located in a difference density Fourier map and were refined with fixed isotropic displacement parameters of 0.035 (CH and CH_2 groups) or 0.043 Å^2 (methyl groups). In the absence of anomalous dispersion effects, Friedel pairs were merged before the refinement. The absolute configuration assignment is based on the known configuration of C atoms in the starting material. 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, 1997–2002); 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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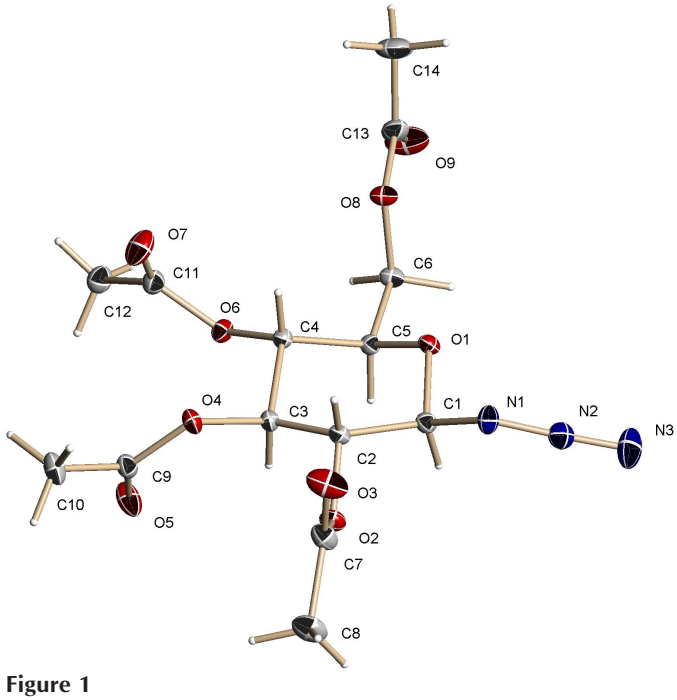

Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids.

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