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Methyl 2,3,4-tri-*O*-acetyl-1-azido-1-deoxy-*β*-D-glucopyranuronate at 100 K

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Key indicators

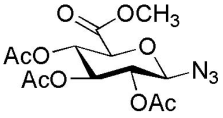
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.080
Data-to-parameter ratio = 8.3

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

A chair conformation is found for the glucopyranose ring in the title compound, $C_{13}H_{17}N_3O_9$, with the non-H substituents occupying equatorial positions.

Comment

The title compound, (I), can be prepared by a bimolecular substitution with an azide anion on the corresponding α-glucuronosyl bromide (Florio *et al.*, 2000) or by Lewis acid-promoted reaction of methyl 1,2,3,4-tetra-*O*-acetyl-*β*-D-glucopyranuronate (Wagner *et al.*, 2002) with trimethylsilyl azide $TMSN_3$ (Györgydeák & Thiem, 1995). The azide, formally a carbohydrate-derived amino acid precursor, has enjoyed wide use in the synthesis of biologically relevant compounds, for example, as mimics of sialic acids (Florio *et al.*, 2000) and as bivalent oligosaccharide ligands (Tosin *et al.*, 2005).


(I)

The solid state structure of (I) was determined by X-ray diffraction at 100 K (Fig. 1). The bond lengths and angles are within the expected ranges. The six-membered ring exhibits the chair conformation expected for a glucopyranose, and all non-H substituents are found in equatorial positions. The methyl ester and acetate groups are approximately parallel to each other and the C═O groups point alternately up and down (Fig. 1).

The *β*-configuration, 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 = 171.21 (18)°] and the C1—N1—N2 angle is 115.43 (14)°. The N—N triple- and single-bond distances N2—N3 and N1—N2 are 1.124 (2) and 1.2412 (19) Å, respectively.

The structure of the same compound, determined at room temperature, is reported independently by another research group in the following paper (Wilkinson *et al.*, 2005).

Experimental

The title compound, (I), was prepared as described in the literature (Florio *et al.*, 2000). Crystals suitable for single-crystal X-ray diffraction were obtained by slow cooling of a hot methanol solution.

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Crystal data

$C_{13}H_{17}N_3O_9$
$M_r = 359.30$
Orthorhombic, $P2_12_12_1$
$a = 7.3201$ (8) Å
$b = 13.9189$ (14) Å
$c = 15.8356$ (16) Å
$V = 1613.5$ (3) Å^3
$Z = 4$
$D_x = 1.479$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8322 reflections
$\theta = 2.6$–30.5°
$\mu = 0.13$ mm^{-1}
$T = 100$ (2) K
Block, colourless
$0.59 \times 0.54 \times 0.46$ mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min} = 0.780$, $T_{\max} = 0.940$
16714 measured reflections

2290 independent reflections
2267 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 28.3°$
$h = -9 \rightarrow 9$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.080$
$S = 1.08$
2290 reflections
277 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.4038P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

All H atoms were located in a difference density Fourier map and refined with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for methine H atoms; the range of C—H distances is 0.90 (3)–0.99 (3) Å. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before refinement. The absolute configuration was assigned on the basis of 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*.

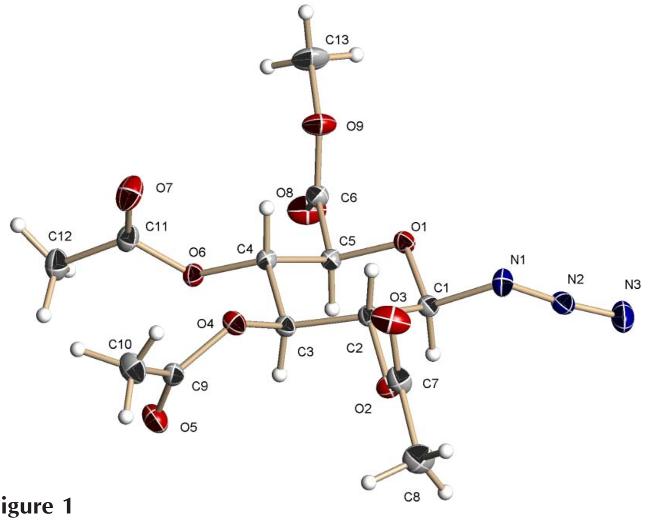

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

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