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Methyl (2′*R*,3′*R*)-2-azido-2,6-dideoxy-3,4-*O*-(2′,3′-dimethoxybutane-2′,3′-diyl)-α-L-glucopyranoside

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.032
wR factor = 0.058
Data-to-parameter ratio = 7.0

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

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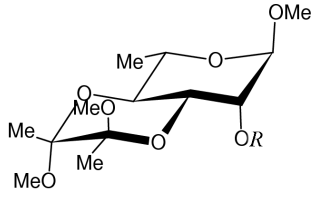
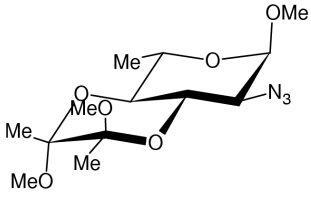
Unlike other α-mannopyranoside-2-sulphonates, the 2-*O*-trifluoromethylsulfonyl (trifluoromethanesulfonate) derivative, (II), underwent an S_N2 displacement with an azide ion to give methyl (2′*R*,3′*R*)-2-azido-2,6-dideoxy-3,4-*O*-(2′,3′-dimethoxybutane-2′,3′-diyl)-α-L-glucopyranoside, $C_{13}H_{23}N_3O_6$, (IV), in preference to the E2 reaction.

Comment

There are two, essentially identical, independent molecules in the asymmetric unit of the title compound, (IV). The structure of one of these molecules is shown in Fig. 1. There are no unusual bond lengths or angles. Comparison with the structure of (III) (Barnes *et al.*, 2002) shows that the original configuration is preserved at all centres except for inversion of the configuration at the reaction centre C2. In the absence of a heavy atom, the absolute configuration of (IV) could not be determined but the alternate assumption that the entire configuration had reversed, apart from C2, is untenable.



(I) R = H
(II) R = SO_2CF_3
(III) R = $SO_2C_6H_4Me$-*p*



(IV)

Experimental

(II) and (IV) were prepared as described for the D-enantiomers (Dix *et al.*, 2001) Analysis of (IV), found: C 49.34, H 7.59, N 13.18%; calculated: C 49.20, H 7.31, N 13.24%. $[\alpha]_D$ −291° (*c* = 1, $CHCl_3$). Crystals from ethyl acetate/hexane.

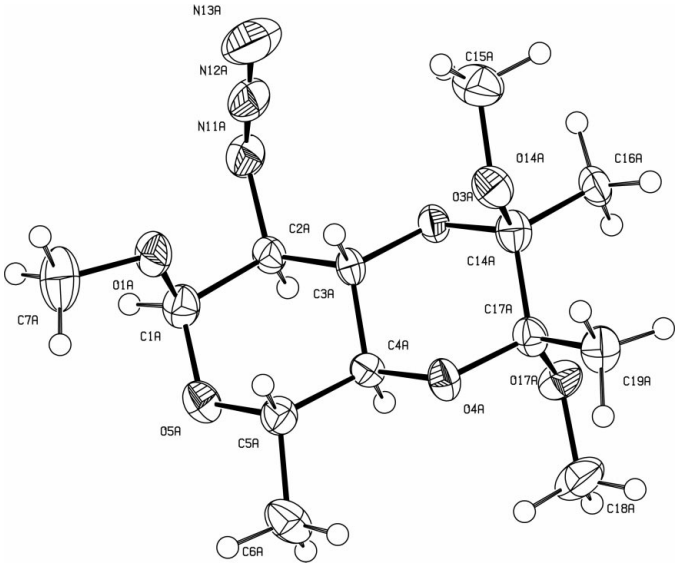


Figure 1
The structure of one of the independent molecules of (IV), showing 50% probability displacement ellipsoids.

Crystal data

$C_{13}H_{23}N_3O_6$
M_r = 317.34
Orthorhombic, $P2_12_12_1$
a = 10.065 (4) Å
b = 10.512 (16) Å
c = 30.696 (10) Å
V = 3248 (5) Å^3
Z = 8
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 250 reflections
θ = 2.1–25.0°
μ = 0.10 mm^{-1}
T = 150 (2) K
Block, colourless
0.32 × 0.18 × 0.10 mm

Data collection

Enraf–Nonius FAST system diffractometer
Absorption correction: none
11775 measured reflections
2861 independent reflections
1724 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 11$
$l = -34 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.058
S = 0.72
2861 reflections
409 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0051P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.027
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Details of the data collection are given in Darr *et al.* (1993). Owing to a lack of any significant anomalous dispersion effects, the absolute configuration cannot be determined from the diffraction experiment. Friedel pairs have been merged prior to refinement. All H atoms were introduced at calculated positions as riding atoms (C—H = 0.97–0.98 Å, with a displacement parameter equal to 1.2 (CH) or 1.5 (CH_3) times that of the parent atom.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1990); cell refinement: *MADNES*; data reduction: *MADNES*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLUTON*92 (Spek, 1992) and *PLATON*92 (Spek, 1992); software used to prepare material for publication: *SHELXL*97.

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