

2-Azido-2-deoxy-3,4-O-isopropylidene-2-C-methyl-D-ribo-1,5-lactone

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.045
wR factor = 0.102
Data-to-parameter ratio = 11.0

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

The configuration of the title azide, $\text{C}_9\text{H}_{13}\text{N}_3\text{O}_4$, prepared from an arabinonolactone, is established by the X-ray crystal structure. The methyl group on the azide-substituted C atom is in a flagpole position. There are two molecules in the asymmetric unit.

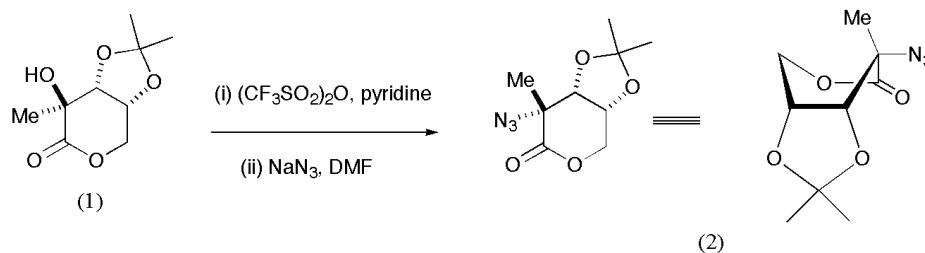
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Comment

The potential of the Kiliani ascension of ketoses to provide readily available branched scaffolds has been recognized (Hotchkiss *et al.*, 2004). A further class of branched carbohydrate building blocks may be available from the reaction of cyanide on 1-deoxyketoses, themselves prepared by addition of organometallic reagents to sugar lactones. The Kiliani ascension of a protected 1-deoxy-D-ribose gave the arabinonolactone, (1) (Punzo *et al.*, 2005). The free hydroxyl group in (1) was esterified with triflic anhydride and the resulting trifluoromethanesulfonate ester treated with sodium azide in dimethylformamide. An azide was formed in good yield by nucleophilic displacement of the trifluoromethanesulfonate, even though the C atom C1 in (2) is tertiary and highly sterically hindered. It is possible that neighbouring group participation by oxygen might be involved in the reaction, but the crystal structure shows that the reaction proceeds with clean inversion of configuration to give the ribonolactone (2) in a boat conformation with the methyl group on C1 in the flagpole position (Fig. 1). Elaboration of (2) to a novel proline derivative is in progress. There are two molecules in the asymmetric unit, related by a twofold axis of pseudosymmetry, of the form $(1.29 - z, 1.24 - y, 1.08 - x)$, lying approximately parallel to [101]. Bond lengths and angles are normal. The crystal packing is shown in Fig. 2.



Experimental

The sugar was crystallized by dissolving it in diethyl ether and allowing the slow evaporation of the solvent until clear colourless crystals formed.

Crystal data

C₉H₁₃N₃O₄
M_r = 227.22
 Monoclinic, *P*2₁
a = 6.4862 (1) Å
b = 27.9310 (5) Å
c = 6.4787 (1) Å
 β = 109.8940 (7)°
V = 1103.68 (3) Å³
Z = 4

D_x = 1.367 Mg m⁻³
 Mo K α radiation
 Cell parameters from 2156 reflections
 θ = 5–30°
 μ = 0.11 mm⁻¹
T = 120 K
 Block, colourless
 0.60 × 0.40 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
 ω scans
 Absorption correction: multi-scan
 (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
*T*_{min} = 0.96, *T*_{max} = 0.97
 5531 measured reflections

3200 independent reflections
 2511 reflections with *I* > 2 σ (*I*)
*R*_{int} = 0.016
 θ _{max} = 30.0°
h = -9 → 9
k = -39 → 36
l = -8 → 8

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.045
wR(*F*²) = 0.102
S = 0.99
 3200 reflections
 290 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F^2) + 0.04 + 0.34P]$,
 where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$
 Extinction correction: Larson
 (1970), equation 22
 Extinction coefficient: $4.7(8) \times 10^2$

In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration is known from the synthesis. H atoms were found in a difference density synthesis. Those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C–H = 0.97–1.00 Å), after which they were refined as riding, with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: COLLECT (Nonius, 2001); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: CRYSTALS (Betteridge *et al.*, 2003); molecular graphics: CAMERON (Watkin *et al.*, 1996); software used to prepare material for publication: CRYSTALS.

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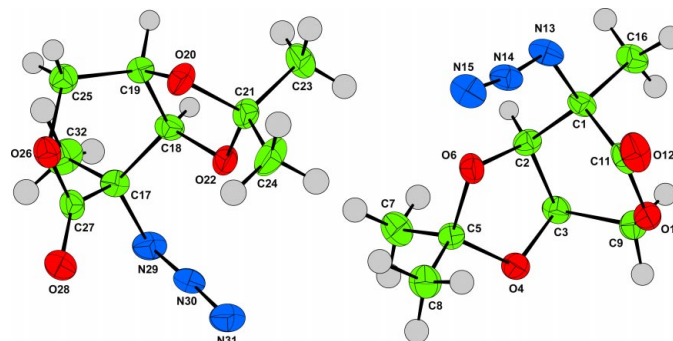


Figure 1
 The asymmetric unit of (2), containing two molecules, with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

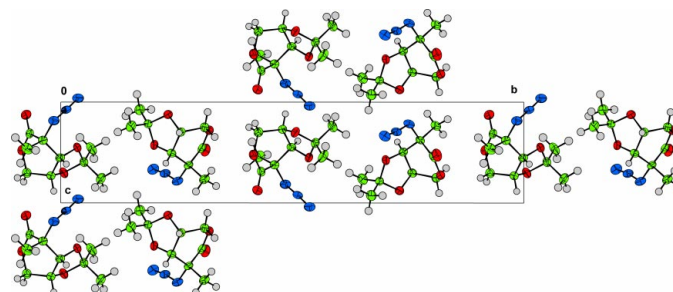


Figure 2
 Packing diagram of (2), viewed down the *a* axis.

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