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

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
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.

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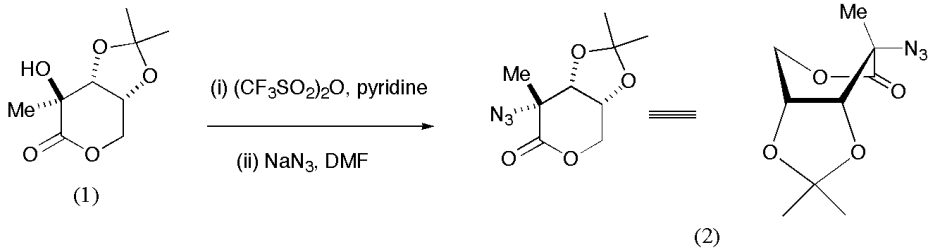
The configuration of the title azide, $C_9H_{13}N_3O_4$, prepared from an arabinonolactone, is established by the X-ray crystal structure. The methyl group on the azide-substituted C atom is in a flagpole position. There are two molecules in the asymmetric unit.

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-ribulose 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.



(1) (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

$C_9H_{13}N_3O_4$
M_r = 227.22
Monoclinic, $P2_1$
a = 6.4862 (1) Å
b = 27.9310 (5) Å
c = 6.4787 (1) Å
β = 109.8940 (7)°
V = 1103.68 (3) Å^3
Z = 4
D_x = 1.367 Mg m^{-3}
Mo Kα 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 $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 30.0°
$h = -9 \rightarrow 9$
$k = -39 \rightarrow 36$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 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 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 4.7 (8) × 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) = 1.2U_{eq}(C).

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR*92 (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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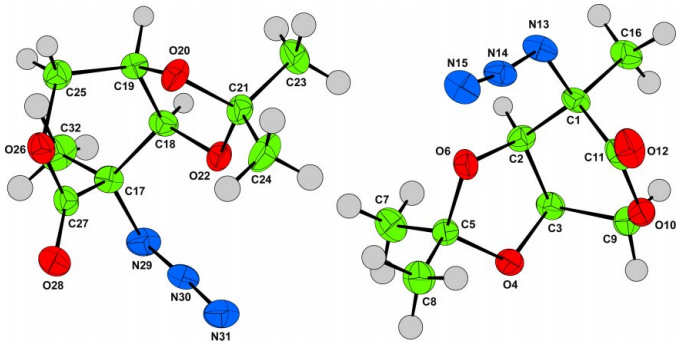

Figure 1
The asymmetric unit of (2), containing two molecules, with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

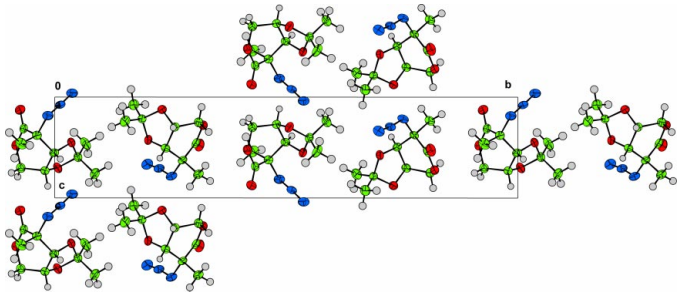

Figure 2
Packing diagram of (2), viewed down the *a* axis.

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$h = -9 \rightarrow 9$
$k = -39 \rightarrow 36$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.102$
$S = 0.99$
3200 reflections
290 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + 0.04 + 0.34P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 470 (80)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4360 (4)	0.27602 (10)	0.6243 (4)	0.0262
C2	0.2608 (4)	0.23935 (10)	0.5058 (4)	0.0270
C3	0.0828 (4)	0.25922 (10)	0.2974 (5)	0.0291

O4	0.1094 (3)	0.23142 (7)	0.1227 (3)	0.0325
C5	0.2153 (4)	0.18818 (10)	0.2154 (4)	0.0293
O6	0.3620 (3)	0.20184 (7)	0.4252 (3)	0.0328
C7	0.0525 (6)	0.15158 (12)	0.2377 (6)	0.0463
C8	0.3449 (6)	0.17048 (12)	0.0748 (5)	0.0434
C9	0.1202 (5)	0.31068 (11)	0.2503 (5)	0.0372
O10	0.3468 (4)	0.32017 (7)	0.2781 (3)	0.0379
C11	0.5067 (5)	0.30414 (10)	0.4566 (5)	0.0324
O12	0.6941 (4)	0.31213 (8)	0.4754 (4)	0.0430
N13	0.6274 (4)	0.25200 (10)	0.7844 (4)	0.0333
N14	0.7362 (4)	0.22497 (10)	0.7068 (4)	0.0314
N15	0.8515 (4)	0.19952 (10)	0.6648 (4)	0.0396
C16	0.3527 (5)	0.30998 (11)	0.7654 (5)	0.0360
C17	0.8089 (4)	0.46134 (10)	0.6214 (5)	0.0273
C18	0.6928 (4)	0.49833 (10)	0.4474 (4)	0.0294
C19	0.4854 (5)	0.47874 (10)	0.2701 (5)	0.0289
O20	0.3122 (3)	0.50613 (7)	0.2990 (3)	0.0346
C21	0.4034 (5)	0.54973 (10)	0.4053 (5)	0.0337
O22	0.6145 (3)	0.53574 (7)	0.5500 (3)	0.0371
C23	0.4252 (6)	0.58654 (12)	0.2415 (6)	0.0463
C24	0.2662 (6)	0.56632 (13)	0.5386 (6)	0.0500
C25	0.4397 (5)	0.42676 (10)	0.2975 (5)	0.0325
O26	0.4665 (3)	0.41594 (7)	0.5246 (3)	0.0322
C27	0.6411 (5)	0.43277 (10)	0.6880 (5)	0.0297
O28	0.6555 (4)	0.42482 (8)	0.8745 (4)	0.0455
N29	0.9703 (4)	0.48479 (10)	0.8150 (4)	0.0344
N30	0.8940 (4)	0.51152 (10)	0.9228 (4)	0.0300
N31	0.8510 (4)	0.53727 (10)	1.0379 (4)	0.0399
C32	0.9515 (5)	0.42758 (13)	0.5388 (6)	0.0442
H21	0.1920	0.2269	0.6055	0.0328*
H31	−0.0654	0.2558	0.3030	0.0339*
H71	0.1393	0.1232	0.2964	0.0535*
H72	−0.0166	0.1656	0.3362	0.0535*
H73	−0.0527	0.1459	0.0932	0.0535*
H91	0.0744	0.3322	0.3456	0.0446*
H92	0.0321	0.3169	0.0981	0.0446*
H161	0.4633	0.3354	0.8308	0.0466*
H162	0.2137	0.3254	0.6795	0.0466*
H163	0.3247	0.2917	0.8862	0.0466*
H181	0.7989	0.5118	0.3851	0.0354*
H191	0.4869	0.4848	0.1160	0.0359*
H231	0.4942	0.6160	0.3208	0.0526*
H232	0.2737	0.5927	0.1426	0.0526*
H233	0.5140	0.5742	0.1546	0.0526*
H251	0.2857	0.4197	0.2093	0.0398*
H252	0.5359	0.4064	0.2487	0.0398*
H321	1.0155	0.4017	0.6482	0.0571*
H322	1.0751	0.4459	0.5221	0.0571*

H323	0.8664	0.4132	0.3948	0.0571*
H81	0.4212	0.1399	0.1374	0.0518*
H82	0.4560	0.1951	0.0730	0.0518*
H83	0.2432	0.1647	−0.0783	0.0518*
H241	0.3271	0.5970	0.6149	0.0587*
H242	0.1118	0.5716	0.4393	0.0587*
H243	0.2688	0.5413	0.6500	0.0587*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0277 (12)	0.0304 (14)	0.0235 (13)	−0.0038 (11)	0.0126 (10)	−0.0058 (11)
C2	0.0260 (12)	0.0299 (14)	0.0262 (13)	−0.0026 (11)	0.0103 (10)	0.0032 (11)
C3	0.0275 (12)	0.0303 (15)	0.0289 (14)	0.0063 (11)	0.0088 (11)	−0.0031 (11)
O4	0.0349 (10)	0.0304 (11)	0.0271 (10)	0.0090 (8)	0.0042 (8)	−0.0011 (8)
C5	0.0286 (12)	0.0264 (14)	0.0266 (14)	0.0011 (11)	0.0014 (10)	0.0003 (11)
O6	0.0318 (10)	0.0228 (9)	0.0348 (11)	0.0029 (8)	−0.0002 (8)	−0.0028 (8)
C7	0.0476 (18)	0.0388 (18)	0.0447 (19)	−0.0134 (15)	0.0056 (15)	−0.0077 (15)
C8	0.0477 (18)	0.0397 (17)	0.0416 (18)	0.0159 (14)	0.0138 (14)	−0.0023 (14)
C9	0.0503 (18)	0.0293 (15)	0.0322 (15)	0.0091 (13)	0.0143 (13)	0.0011 (12)
O10	0.0610 (14)	0.0283 (11)	0.0302 (11)	−0.0060 (10)	0.0229 (11)	−0.0004 (9)
C11	0.0433 (15)	0.0241 (13)	0.0365 (16)	−0.0068 (12)	0.0225 (13)	−0.0119 (12)
O12	0.0464 (12)	0.0360 (12)	0.0594 (14)	−0.0108 (10)	0.0348 (11)	−0.0077 (10)
N13	0.0266 (11)	0.0462 (15)	0.0276 (12)	−0.0015 (10)	0.0098 (9)	−0.0085 (11)
N14	0.0249 (11)	0.0415 (15)	0.0253 (12)	−0.0080 (11)	0.0052 (9)	−0.0041 (11)
N15	0.0278 (12)	0.0488 (16)	0.0424 (15)	0.0020 (12)	0.0124 (11)	−0.0072 (13)
C16	0.0450 (16)	0.0381 (16)	0.0315 (15)	0.0014 (13)	0.0217 (13)	−0.0069 (13)
C17	0.0242 (12)	0.0318 (15)	0.0275 (14)	0.0035 (10)	0.0107 (11)	0.0004 (11)
C18	0.0306 (13)	0.0328 (15)	0.0250 (13)	−0.0038 (11)	0.0097 (11)	0.0028 (11)
C19	0.0325 (13)	0.0280 (14)	0.0258 (14)	0.0006 (11)	0.0095 (11)	−0.0008 (11)
O20	0.0300 (10)	0.0289 (11)	0.0398 (12)	−0.0005 (8)	0.0052 (9)	−0.0097 (9)
C21	0.0318 (13)	0.0253 (14)	0.0341 (16)	0.0016 (11)	−0.0015 (12)	−0.0030 (12)
O22	0.0408 (11)	0.0235 (10)	0.0333 (11)	0.0037 (9)	−0.0054 (9)	−0.0047 (8)
C23	0.0531 (19)	0.0325 (16)	0.045 (2)	0.0098 (15)	0.0057 (16)	0.0094 (14)
C24	0.0519 (19)	0.0393 (18)	0.056 (2)	0.0026 (16)	0.0146 (17)	−0.0203 (16)
C25	0.0354 (14)	0.0306 (15)	0.0336 (15)	0.0011 (12)	0.0146 (12)	−0.0016 (12)
O26	0.0347 (10)	0.0284 (10)	0.0371 (11)	−0.0004 (8)	0.0167 (9)	0.0038 (9)
C27	0.0371 (14)	0.0231 (13)	0.0336 (16)	0.0055 (11)	0.0183 (12)	0.0030 (11)
O28	0.0691 (15)	0.0387 (13)	0.0342 (12)	−0.0038 (11)	0.0246 (11)	0.0051 (10)
N29	0.0269 (11)	0.0446 (15)	0.0295 (13)	0.0083 (11)	0.0067 (10)	−0.0002 (11)
N30	0.0262 (11)	0.0381 (14)	0.0235 (12)	0.0027 (10)	0.0058 (9)	0.0061 (10)
N31	0.0418 (14)	0.0483 (16)	0.0297 (14)	0.0034 (12)	0.0125 (11)	0.0000 (12)
C32	0.0372 (15)	0.0495 (19)	0.0491 (19)	0.0117 (14)	0.0186 (14)	−0.0092 (15)

Geometric parameters (Å, º)

C1—C2	1.528 (4)	C17—C18	1.526 (4)
C1—C11	1.532 (4)	C17—C27	1.525 (4)

C1—N13	1.480 (4)	C17—N29	1.485 (4)
C1—C16	1.538 (4)	C17—C32	1.539 (4)
C2—C3	1.550 (4)	C18—C19	1.541 (4)
C2—O6	1.426 (3)	C18—O22	1.422 (4)
C2—H21	0.967	C18—H181	0.983
C3—O4	1.431 (3)	C19—O20	1.423 (3)
C3—C9	1.506 (4)	C19—C25	1.504 (4)
C3—H31	0.979	C19—H191	1.016
O4—C5	1.418 (3)	O20—C21	1.425 (3)
C5—O6	1.420 (3)	C21—O22	1.426 (3)
C5—C7	1.512 (4)	C21—C23	1.518 (5)
C5—C8	1.517 (4)	C21—C24	1.508 (5)
C7—H71	0.972	C23—H231	0.992
C7—H72	0.977	C23—H232	0.988
C7—H73	0.965	C23—H233	0.994
C8—H81	1.000	C24—H241	1.000
C8—H82	1.000	C24—H242	1.000
C8—H83	1.000	C24—H243	1.000
C9—O10	1.443 (4)	C25—O26	1.453 (4)
C9—H91	0.979	C25—H251	0.987
C9—H92	0.972	C25—H252	0.974
O10—C11	1.340 (4)	O26—C27	1.345 (4)
C11—O12	1.200 (4)	C27—O28	1.201 (4)
N13—N14	1.250 (3)	N29—N30	1.235 (4)
N14—N15	1.130 (3)	N30—N31	1.137 (4)
C16—H161	0.996	C32—H321	1.000
C16—H162	0.983	C32—H322	0.988
C16—H163	1.002	C32—H323	0.994

C2—C1—C11	109.8 (2)	C18—C17—C27	110.1 (2)
C2—C1—N13	110.6 (2)	C18—C17—N29	110.8 (2)
C11—C1—N13	110.3 (2)	C27—C17—N29	111.0 (2)
C2—C1—C16	111.2 (2)	C18—C17—C32	111.2 (2)
C11—C1—C16	110.9 (2)	C27—C17—C32	110.4 (2)
N13—C1—C16	104.1 (2)	N29—C17—C32	103.3 (2)
C1—C2—C3	113.8 (2)	C17—C18—C19	113.3 (2)
C1—C2—O6	108.7 (2)	C17—C18—O22	108.5 (2)
C3—C2—O6	104.1 (2)	C19—C18—O22	104.4 (2)
C1—C2—H21	109.8	C17—C18—H181	108.9
C3—C2—H21	109.6	C19—C18—H181	112.7
O6—C2—H21	110.7	O22—C18—H181	108.8
C2—C3—O4	103.9 (2)	C18—C19—O20	103.8 (2)
C2—C3—C9	113.2 (2)	C18—C19—C25	114.3 (2)
O4—C3—C9	106.7 (2)	O20—C19—C25	107.9 (2)
C2—C3—H31	112.4	C18—C19—H191	112.1
O4—C3—H31	111.1	O20—C19—H191	108.1
C9—C3—H31	109.3	C25—C19—H191	110.2
C3—O4—C5	107.4 (2)	C19—O20—C21	107.8 (2)

O4—C5—O6	104.3 (2)	O20—C21—O22	103.4 (2)
O4—C5—C7	111.3 (2)	O20—C21—C23	111.3 (2)
O6—C5—C7	110.6 (2)	O22—C21—C23	110.4 (3)
O4—C5—C8	108.1 (2)	O20—C21—C24	108.1 (2)
O6—C5—C8	109.5 (2)	O22—C21—C24	109.2 (2)
C7—C5—C8	112.6 (3)	C23—C21—C24	113.9 (3)
C2—O6—C5	107.8 (2)	C21—O22—C18	108.1 (2)
C5—C7—H71	105.0	C21—C23—H231	109.4
C5—C7—H72	106.4	C21—C23—H232	105.2
H71—C7—H72	113.9	H231—C23—H232	111.4
C5—C7—H73	107.7	C21—C23—H233	111.7
H71—C7—H73	111.7	H231—C23—H233	109.5
H72—C7—H73	111.6	H232—C23—H233	109.5
C5—C8—H81	109.5	C21—C24—H241	109.6
C5—C8—H82	109.3	C21—C24—H242	109.5
H81—C8—H82	109.5	H241—C24—H242	109.5
C5—C8—H83	109.6	C21—C24—H243	109.3
H81—C8—H83	109.5	H241—C24—H243	109.5
H82—C8—H83	109.5	H242—C24—H243	109.5
C3—C9—O10	112.2 (2)	C19—C25—O26	111.1 (2)
C3—C9—H91	110.8	C19—C25—H251	109.0
O10—C9—H91	108.9	O26—C25—H251	107.1
C3—C9—H92	107.3	C19—C25—H252	110.6
O10—C9—H92	108.4	O26—C25—H252	109.8
H91—C9—H92	109.2	H251—C25—H252	109.3
C9—O10—C11	120.2 (2)	C25—O26—C27	120.2 (2)
C1—C11—O10	116.9 (2)	C17—C27—O26	116.8 (2)
C1—C11—O12	124.1 (3)	C17—C27—O28	124.2 (3)
O10—C11—O12	119.0 (3)	O26—C27—O28	119.0 (3)
C1—N13—N14	116.5 (2)	C17—N29—N30	116.2 (2)
N13—N14—N15	170.9 (3)	N29—N30—N31	171.2 (3)
C1—C16—H161	110.2	C17—C32—H321	110.5
C1—C16—H162	111.9	C17—C32—H322	109.1
H161—C16—H162	108.5	H321—C32—H322	107.3
C1—C16—H163	110.1	C17—C32—H323	111.6
H161—C16—H163	109.1	H321—C32—H323	109.6
H162—C16—H163	106.9	H322—C32—H323	108.7