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2-*C*-Hydroxymethyl-2,3-*O*-isopropylidene-D-ribono-1,5-lactam. Corrigendum

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The name of one of the authors in the paper by Newton *et al.* [*Acta Cryst.* (2004), E**60**, o909–o910] is corrected.

In the paper by Newton *et al.* (2004), the second author is incorrectly given as 'Iezzi Simone Michela'. The correct name should be 'Michela Iezzi Simone', as given above.

References

Newton, C. R., Michela, I. S., Fleet, G. W. J., Blériot, Y. & Watkin, D. J. (2004). *Acta Cryst.* E**60**, o909–o910.

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.087
Data-to-parameter ratio = 9.7

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

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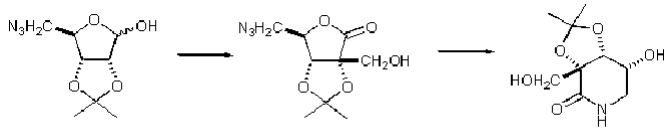
Accepted 5 April 2004
Online 30 April 2004

The title compound, $C_9H_{14}NO_5$, was formed by catalytic hydrogenation of an azidolactone using Pd-black in 1,4-dioxane.

Comment

The replacement of the ring O atom of a carbohydrate by nitrogen gives a range of sugar mimics (Winchester & Fleet, 1992), many of which are natural products widely spread in plants (Asano *et al.*, 2000). Because of the multitude of potential biological activities, interest in understanding the structures in the search for transition-state analogues continues (Heck *et al.*, 2004). Almost all of the natural products and their synthetic analogues contain straight carbon chains; however, there are some very promising indications that carbohydrate mimics with hydroxymethyl branches (Ichikawa & Igarashi, 1995; Ichikawa *et al.*, 1998), as well as their deoxygenated equivalents (Lillelund *et al.*, 2003; Ostrowski *et al.*, 2003), will show significant inhibition of sugar-metabolizing enzymes. However, the chemistry of simple branched sugars as starting materials is little explored. The title compound, (3), is a powerful intermediate in which a stereochemical ambiguity arises from an aldol reaction; additionally, information about the conformation of both protected and unprotected lactams may help to understand the basis of their biological activity.



(1) (2) (3)

The azidolactol (1) was prepared from D-ribose and submitted to the key aldol branching step. Subsequent oxidation of the aldol product with bromine water gave the branched azidolactone (2). Hydrogenation of (2) resulted in initial reduction of the azide to the corresponding amine which underwent subsequent isomerization to the title lactam (3). The X-ray crystal structure of (3) removes any ambiguity about the course of the aldol condensation.

Experimental

2-*C*-Hydroxymethyl-2,3-*O*-isopropylidene-D-ribono-1,5-lactam was obtained on reduction of 5-azido-2,3-*O*-isopropylidene-D-hamamelono-1,4-lactone, (2), using Pd-black and hydrogen gas in 1,4-dioxane at low reaction concentration (2.5 mg ml^{-1}). A quantitative yield of the title compound was obtained. The title material was then

recrystallized using solvent evaporation (methanol), appearing as colourless block crystals.

Crystal data

$C_9H_{14}NO_5$
M_r = 216.21
Orthorhombic, $P2_12_12_1$
a = 7.3137 (1) Å
b = 10.6657 (2) Å
c = 12.6476 (3) Å
V = 986.59 (3) Å^3
Z = 4
D_x = 1.456 Mg m^{-3}
Mo Kα radiation
Cell parameters from 1329 reflections
θ = 5–27°
μ = 0.12 mm^{-1}
T = 150 K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.976, T_{max} = 0.988
2272 measured reflections
1315 independent reflections
1195 reflections with $I > 2\sigma(I)$
R_{int} = 0.01
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 0.98
1315 reflections
136 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^*) + (0.0403p)^2 + 0.549p]$ where $p = 0.333\max(F_o^2,0) + 0.667F_c^2$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C14	1.528 (3)	C4—N5	1.473 (3)
C1—O11	1.428 (2)	N5—C6	1.331 (3)
C1—C6	1.531 (3)	C6—O7	1.259 (3)
C1—C2	1.521 (3)	O9—C10	1.438 (2)
C2—O9	1.432 (2)	C10—C13	1.513 (3)
C2—C3	1.513 (3)	C10—C12	1.512 (3)
C3—O8	1.431 (2)	C10—O11	1.444 (2)
C3—C4	1.517 (3)	C14—O15	1.419 (3)
C14—C1—O11	107.00 (17)	C6—N5—C4	125.82 (18)
C14—C1—C6	110.38 (17)	O7—C6—N5	122.03 (19)
O11—C1—C6	107.11 (16)	O7—C6—C1	118.13 (18)
C14—C1—C2	114.23 (17)	N5—C6—C1	119.79 (18)
O11—C1—C2	103.03 (16)	C10—O9—C2	108.04 (15)
C6—C1—C2	114.30 (16)	C13—C10—C12	114.02 (19)
O9—C2—C3	110.70 (16)	C13—C10—O11	107.24 (16)
O9—C2—C1	102.89 (15)	C12—C10—O11	110.31 (17)
C3—C2—C1	111.33 (17)	C13—C10—O9	111.49 (18)
O8—C3—C4	109.83 (16)	C12—C10—O9	107.55 (17)
O8—C3—C2	111.11 (16)	O11—C10—O9	105.94 (16)
C4—C3—C2	109.97 (16)	C1—O11—C10	109.05 (14)
N5—C4—C3	110.11 (16)	O15—C14—C1	109.28 (17)

H atoms were placed geometrically after each cycle, at a distance of 1.0 Å; U_{iso} values were set to 1.2 times the U_{eq} value of the parent atom. The absolute configuration was assumed to be the same as that of the sugar and the Friedel pairs were merged in the final refinement.

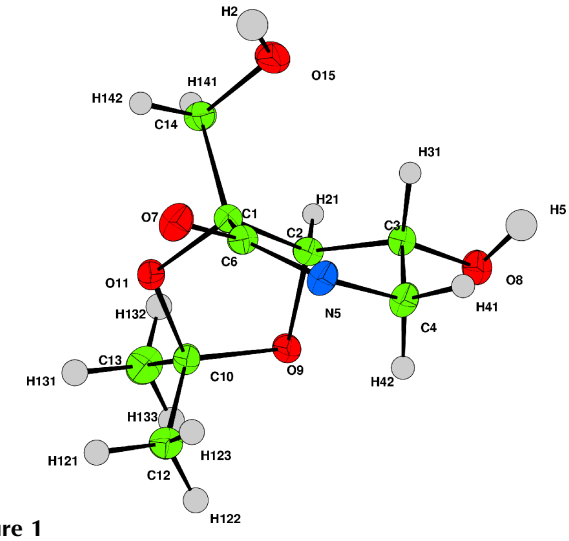


Figure 1
The molecular structure of (3), with 50% probability displacement ellipsoids.

Data collection: *COLLECT* (Nonius, 1997–2001); 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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supporting information

Acta Cryst. (2004). E**60**, o909–o910 [https://doi.org/10.1107/S1600536804008141]

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$C_9H_{14}NO_5$
$M_r = 216.21$
Orthorhombic, $P2_12_12_1$
$a = 7.3137$ (1) Å
$b = 10.6657$ (2) Å
$c = 12.6476$ (3) Å
$V = 986.59$ (3) Å^3
$Z = 4$
$F(000) = 460$
$D_x = 1.456$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1329 reflections
$\theta = 5$–27°
$\mu = 0.12$ mm^{-1}
$T = 150$ K
Plate, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1996)
$T_{min} = 0.976$, $T_{max} = 0.988$
2272 measured reflections
1315 independent reflections
1195 reflections with $I > 2\sigma(I)$
$R_{int} = 0.01$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.087$
$S = 0.98$
1315 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^*) + (0.0403p)^2 + 0.549p]$ where $p = 0.333\max(F_o^2,0) + 0.667F_c^2$
$(\Delta/\sigma)_{max} = 0.000238$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7866 (3)	0.0035 (2)	0.02895 (15)	0.0188
C2	0.7198 (3)	0.11166 (19)	−0.03931 (16)	0.0193
C3	0.6725 (3)	0.0679 (2)	−0.14980 (15)	0.0201

C4	0.5206 (3)	−0.0288 (2)	−0.14517 (16)	0.0229
N5	0.5697 (2)	−0.12992 (17)	−0.07111 (15)	0.0231
C6	0.6915 (3)	−0.1217 (2)	0.00682 (17)	0.0206
O7	0.7278 (2)	−0.21316 (13)	0.06611 (13)	0.0263
O8	0.6178 (2)	0.17082 (15)	−0.21510 (11)	0.0246
O9	0.5593 (2)	0.15309 (15)	0.01535 (11)	0.0233
C10	0.5842 (3)	0.1282 (2)	0.12615 (16)	0.0202
O11	0.7351 (2)	0.04121 (14)	0.13309 (11)	0.0213
C12	0.4107 (3)	0.0679 (2)	0.16656 (17)	0.0270
C13	0.6394 (3)	0.2453 (2)	0.18566 (19)	0.0292
C14	0.9941 (3)	−0.0134 (2)	0.02935 (16)	0.0218
O15	1.0513 (2)	−0.05872 (15)	−0.07082 (12)	0.0291
H21	0.8129	0.1802	−0.0492	0.0233*
H31	0.7838	0.0248	−0.1813	0.0262*
H41	0.4980	−0.0634	−0.2173	0.0283*
H42	0.4059	0.0146	−0.1199	0.0283*
H121	0.4229	0.0489	0.2435	0.0338*
H122	0.3890	−0.0127	0.1269	0.0338*
H123	0.3048	0.1253	0.1548	0.0338*
H131	0.6555	0.2262	0.2625	0.0368*
H132	0.7581	0.2780	0.1564	0.0368*
H133	0.5432	0.3116	0.1772	0.0368*
H141	1.0564	0.0687	0.0449	0.0264*
H142	1.0305	−0.0751	0.0866	0.0264*
H2	1.1000	−0.1353	−0.0668	0.0500*
H5	0.6767	0.1578	−0.2858	0.0500*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0176 (10)	0.0214 (9)	0.0176 (9)	−0.0002 (8)	0.0004 (8)	0.0006 (8)
C2	0.0184 (9)	0.0199 (9)	0.0194 (9)	0.0000 (8)	0.0014 (8)	−0.0007 (8)
C3	0.0200 (9)	0.0221 (10)	0.0182 (9)	0.0017 (8)	−0.0010 (8)	0.0019 (8)
C4	0.0229 (10)	0.0246 (11)	0.0213 (10)	−0.0012 (9)	−0.0052 (9)	0.0020 (8)
N5	0.0229 (8)	0.0226 (8)	0.0237 (8)	−0.0016 (8)	−0.0054 (8)	0.0017 (8)
C6	0.0189 (9)	0.0219 (9)	0.0209 (10)	0.0006 (8)	0.0004 (8)	0.0004 (8)
O7	0.0277 (8)	0.0214 (7)	0.0298 (8)	−0.0024 (7)	−0.0048 (7)	0.0043 (6)
O8	0.0267 (8)	0.0261 (8)	0.0209 (7)	0.0028 (7)	−0.0006 (6)	0.0043 (6)
O9	0.0232 (7)	0.0294 (8)	0.0172 (7)	0.0093 (7)	0.0018 (6)	0.0017 (6)
C10	0.0210 (10)	0.0226 (10)	0.0169 (9)	0.0027 (9)	−0.0016 (8)	0.0009 (8)
O11	0.0227 (7)	0.0248 (7)	0.0163 (6)	0.0055 (7)	−0.0009 (6)	−0.0005 (6)
C12	0.0222 (10)	0.0334 (12)	0.0254 (11)	0.0006 (10)	0.0011 (9)	0.0033 (10)
C13	0.0319 (12)	0.0249 (11)	0.0307 (11)	0.0028 (10)	−0.0048 (10)	−0.0046 (10)
C14	0.0180 (9)	0.0242 (10)	0.0231 (10)	0.0007 (9)	−0.0001 (9)	−0.0004 (9)
O15	0.0294 (8)	0.0325 (8)	0.0255 (8)	0.0114 (7)	0.0059 (7)	0.0040 (7)

Geometric parameters (Å, º)

C1—C14	1.528 (3)	O8—H5	1.002
C1—O11	1.428 (2)	O9—C10	1.438 (2)
C1—C6	1.531 (3)	C10—C13	1.513 (3)
C1—C2	1.521 (3)	C10—C12	1.512 (3)
C2—H21	1.007	C10—O11	1.444 (2)
C2—O9	1.432 (2)	C12—H123	0.998
C2—C3	1.513 (3)	C12—H122	1.007
C3—H31	1.016	C12—H121	0.998
C3—O8	1.431 (2)	C13—H133	1.003
C3—C4	1.517 (3)	C13—H132	1.006
C4—H42	1.010	C13—H131	0.999
C4—H41	0.998	C14—H142	1.013
C4—N5	1.473 (3)	C14—H141	1.007
N5—C6	1.331 (3)	C14—O15	1.419 (3)
C6—O7	1.259 (3)	O15—H2	0.892
C14—C1—O11	107.00 (17)	H5—O8—C3	106.752
C14—C1—C6	110.38 (17)	C10—O9—C2	108.04 (15)
O11—C1—C6	107.11 (16)	C13—C10—C12	114.02 (19)
C14—C1—C2	114.23 (17)	C13—C10—O11	107.24 (16)
O11—C1—C2	103.03 (16)	C12—C10—O11	110.31 (17)
C6—C1—C2	114.30 (16)	C13—C10—O9	111.49 (18)
H21—C2—O9	112.952	C12—C10—O9	107.55 (17)
H21—C2—C3	105.327	O11—C10—O9	105.94 (16)
O9—C2—C3	110.70 (16)	C1—O11—C10	109.05 (14)
H21—C2—C1	113.805	H123—C12—H122	109.083
O9—C2—C1	102.89 (15)	H123—C12—H121	109.798
C3—C2—C1	111.33 (17)	H122—C12—H121	109.056
H31—C3—O8	110.168	H123—C12—C10	109.863
H31—C3—C4	107.155	H122—C12—C10	109.099
O8—C3—C4	109.83 (16)	H121—C12—C10	109.920
H31—C3—C2	108.516	H133—C13—H132	108.752
O8—C3—C2	111.11 (16)	H133—C13—H131	109.264
C4—C3—C2	109.97 (16)	H132—C13—H131	109.064
H42—C4—H41	108.764	H133—C13—C10	109.966
H42—C4—N5	109.677	H132—C13—C10	109.488
H41—C4—N5	110.531	H131—C13—C10	110.279
H42—C4—C3	107.994	H142—C14—H141	107.835
H41—C4—C3	109.712	H142—C14—O15	109.835
N5—C4—C3	110.11 (16)	H141—C14—O15	109.681
C6—N5—C4	125.82 (18)	H142—C14—C1	109.874
O7—C6—N5	122.03 (19)	H141—C14—C1	110.317
O7—C6—C1	118.13 (18)	O15—C14—C1	109.28 (17)
N5—C6—C1	119.79 (18)	H2—O15—C14	112.190