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(1*R*,5*S*,8*R*)-1,8-Dihydroxy-6-oxa-3-azabicyclo-[3.2.1]octan-2-one

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Key indicators

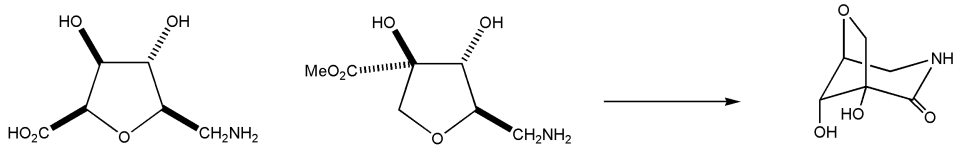
Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.069
Data-to-parameter ratio = 9.7

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

The crystal structure of the title bicyclic lactam, $C_6H_9NO_4$, has firmly established the stereochemistry of the branched δ-sugar amino acid scaffold.

Comment

Sugar amino acids (SAA) have been extensively investigated as peptidomimetics (Chakraborty *et al.*, 2004). δ-Tetrahydrofuran (THF) SAA have been shown to be dipeptide isosteres (Grotenberg *et al.*, 2004; van Well *et al.*, 2003); in particular, those THF SAA which have the carboxylic acid and amino methyl components *cis* to each other, as in (1) (see scheme), almost invariably induce β-turn-like structures in their homooligomers (Smith *et al.*, 1998, 2003).



Most such THF SAA have been derived from carbohydrates and all examples previously have contained a linear

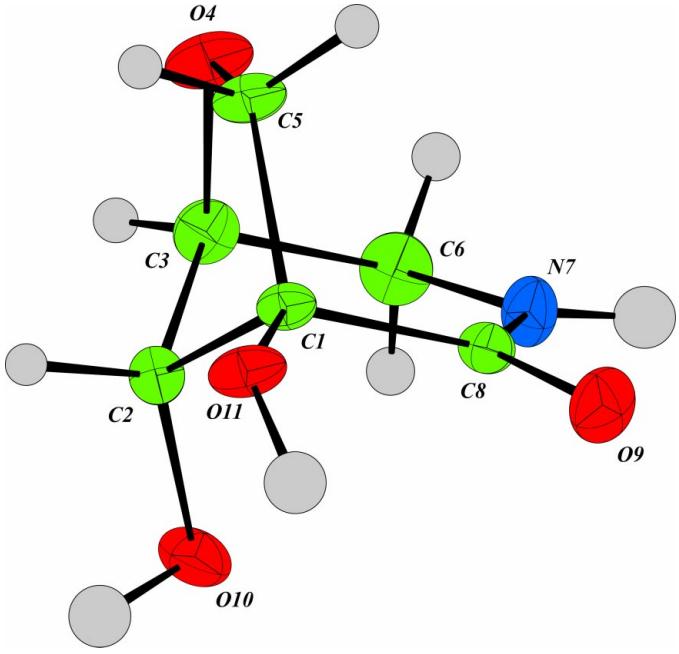


Figure 1
The molecular structure of (3), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

carbon chain. The branched THF SAA scaffold (2), prepared from a branched sugar lactone (Hotchkiss *et al.*, 2004), spontaneously underwent an intramolecular cyclization to form the crystalline bicyclic lactam (3) (Figs. 1 and 2, and Table 1). A number of stereochemical and structural uncertainties in the synthesis of (2) are removed by the X-ray crystallographic analysis of (3).

Experimental

The bicyclic compound was dissolved in methanol in a flask and then crystallized as the solvent slowly evaporated to give colourless plate-like crystals. A suitable piece was cut from a larger crystal.

Crystal data

$C_6H_9NO_4$
M_r = 159.14
Orthorhombic, $P2_12_12_1$
a = 5.9624 (1) Å
b = 10.5889 (2) Å
c = 10.7089 (2) Å
V = 676.11 (2) Å^3
Z = 4
D_x = 1.563 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1160 reflections
θ = 5–30°
μ = 0.13 mm^{-1}
T = 190 K
Block cut from plate, colourless
0.50 × 0.30 × 0.20 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
1981 measured reflections
1158 independent reflections
1158 reflections with no $I/\sigma(I)$ cutoff
R_{int} = 0.007
θ_{max} = 30.0°
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.069
S = 1.03
1158 reflections
119 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F^*) + 0.035p^2 + 0.136p]$ where $p = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 160 (40)

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.5387 (17)	C3—O4	1.4383 (17)
C1—C5	1.5292 (17)	C3—C6	1.5125 (19)
C1—C8	1.5330 (16)	O4—C5	1.4374 (16)
C1—O11	1.4028 (14)	C6—N7	1.4651 (17)
C2—C3	1.5263 (18)	N7—C8	1.3363 (16)
C2—O10	1.4080 (15)	C8—O9	1.2348 (15)
C2—C1—C5	100.65 (10)	C2—C3—C6	110.98 (10)
C2—C1—C8	107.94 (9)	O4—C3—C6	109.68 (11)
C5—C1—C8	110.19 (10)	C3—O4—C5	109.17 (10)
C2—C1—O11	115.93 (10)	C1—C5—O4	105.12 (10)
C5—C1—O11	109.32 (9)	C3—C6—N7	110.52 (10)
C8—C1—O11	112.17 (10)	C6—N7—C8	125.51 (10)
C1—C2—C3	98.03 (9)	C1—C8—N7	116.23 (10)
C1—C2—O10	114.20 (10)	C1—C8—O9	121.38 (11)
C3—C2—O10	111.84 (10)	N7—C8—O9	122.35 (11)
C2—C3—O4	103.70 (10)		

As the data were collected with molybdenum radiation, there were no measurable anomalous differences, as a consequence of which it was admissible to merge Friedel pairs of reflections. The H atoms were all seen in a difference map but those attached to carbon were placed geometrically. Their positions and U_{iso} were regularized using slack restraints. The refinement was completed using riding constraints for the H atoms bonded to carbon, and retaining the slack restraints for the other H atoms.

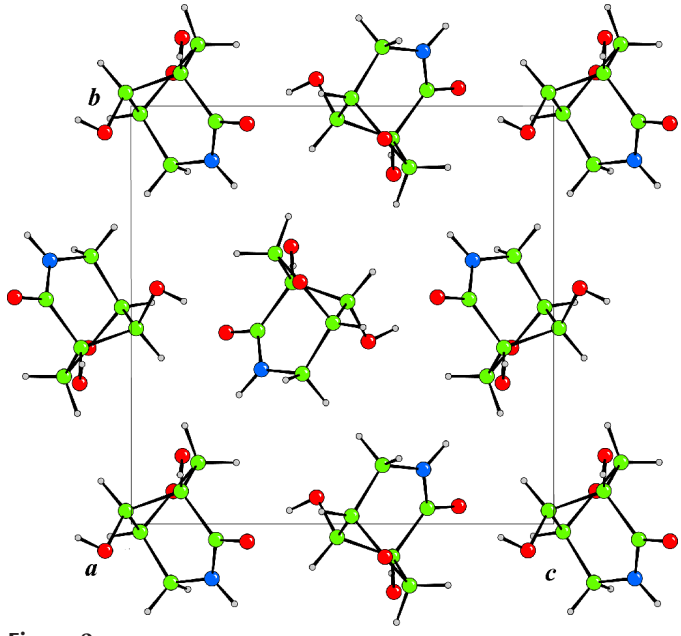


Figure 2
Packing diagram of (3), viewed down the *a* axis.

Data collection: *COLLECT* (Nonius, 1997); 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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