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(2S,3R,4R,5S)-3,4-*O*-Isopropylidene-2-methyl-1-oxa-6,9-diazaspiro[4.5]decane-7,10-dione

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The title spirocarbopeptoid, $C_{11}H_{13}N_2O_5$, was prepared from L-fucose in a sequence that gave this and another anomer. The crystal structure determination removes ambiguities in the synthetic sequence.

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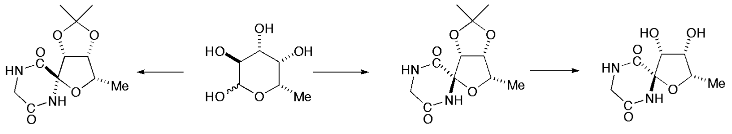
Key indicators

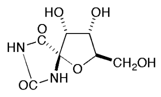
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.073
wR factor = 0.139
Data-to-parameter ratio = 7.1

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

Comment

Sugar amino acids (SAAs) provide an extensive family of peptidomimetics (Baron *et al.*, 2004; Chakraborty *et al.*, 2004), an important sub-class of which incorporate an α-amino acid at the anomeric position of a carbohydrate. Such SAAs may form spiro derivatives, some of which have been demonstrated to possess significant biological activity. Thus hydantocidin, (5), which is a potent and environmentally friendly herbicide, is a spirohydantoin of ribose (Nakajima *et al.*, 1991; Harumaya *et al.*, 1991); similar derivatives of glucose have been shown to be potent inhibitors of glycogen phosphorylase (Watson *et al.*, 1994; Bichard *et al.*, 1995).


(1) (2) (3) (4)


(5)

In an investigation of spiro carbopeptide analogues retaining the footprint of the furanose form of L-fucose, (2), the sugar (2) was transformed into a separable anomeric mixture of the acetonides (1) and (3); one of these anomers,

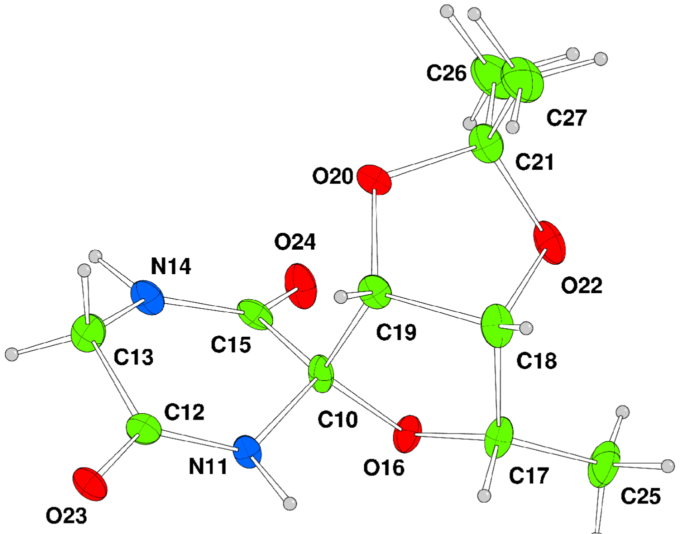


Figure 1
The ordered molecule, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

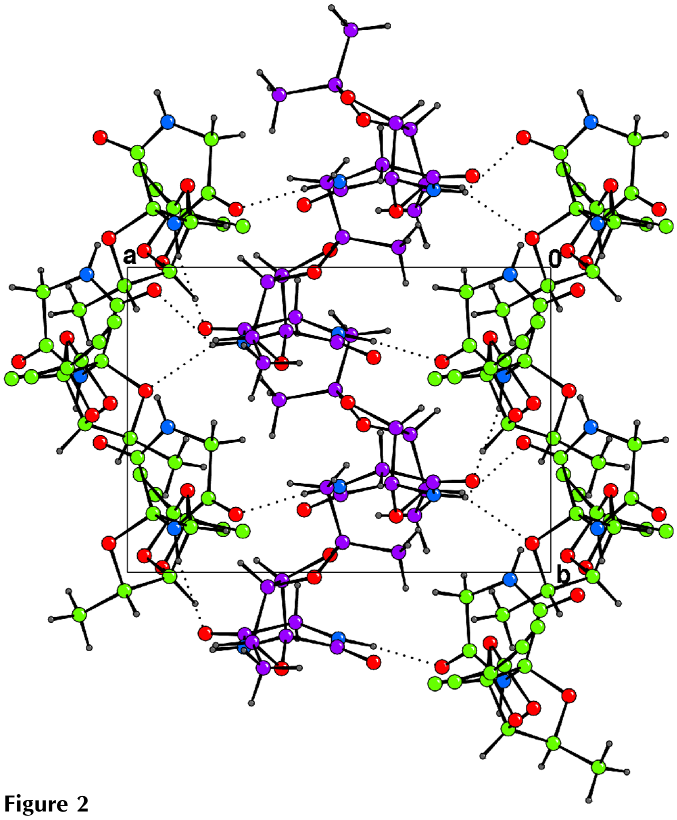


Figure 2
Packing diagram, viewed along the *c* axis. The molecules with the disordered atoms are confined to a single layer in the crystal structure. Dashed lines indicate hydrogen bonds.

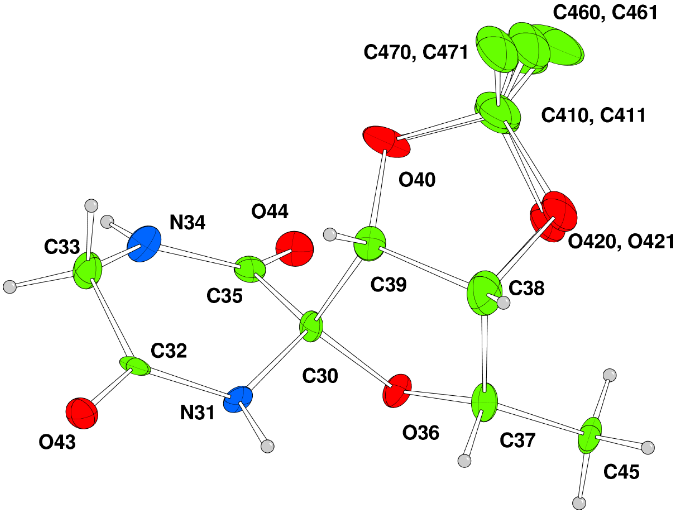


Figure 3
The disordered molecule, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

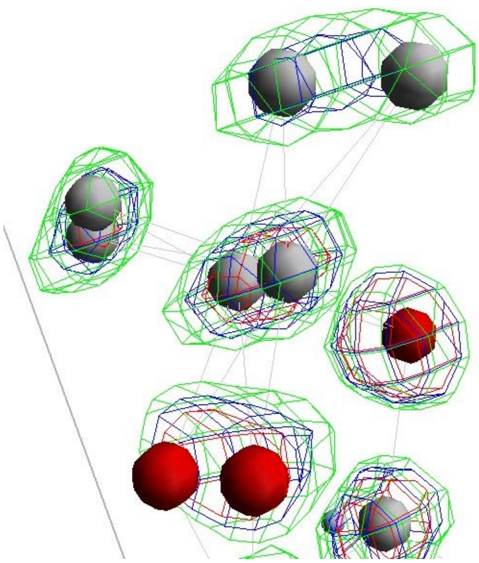


Figure 4
F_o electron density map in the region of the disordered atoms, contoured at 2 (green), 4 (blue) and 6 e Å^{-3} (red). Note the continuous electron density at each disordered atom. A model with ordered atoms and very large displacement parameters is an alternative interpretation.

(3), was highly crystalline and its structure has been shown by X-ray crystallographic analysis to have the relative stereochemistry shown in Fig. 1. The acetonide (3) was transformed into the deprotected analogue, (4), for biological evaluation. Fig. 2 is a packing diagram viewed down the *c* axis. The molecules containing the disordered atoms are, as is not uncommon for $Z' = 2$ structures, confined to a distinct layer through the crystal structure. The overall geometry is essentially the same in the ordered and the disordered molecules and is unexceptional.

Experimental

The title material (Blèriot *et al.*, 2004) was crystallized by vapour diffusion of hexane into a solution in ethyl acetate to give small chunky crystals.

Crystal data

$C_{11}H_{13}N_2O_5$
$M_r = 253.24$
Monoclinic, $P2_1$
$a = 11.100$ (2) Å
$b = 7.994$ (2) Å
$c = 13.895$ (2) Å
$\beta = 93.00$ (2)°
$V = 1231.3$ (4) Å^3
$Z = 4$

$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5932 reflections
$\theta = 2.0$–$26.6°$
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Nonius DIP2000 diffractometer
ω scans
Absorption correction: none
5932 measured reflections
2417 independent reflections
2404 reflections with $I > -3\sigma(I)$

$R_{\rm int} = 0.035$
$\theta_{\rm max} = 26.6°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma F^2)] = 0.073$
$wR(F^2) = 0.139$
$S = 1.07$
2404 reflections
338 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.034P^2 + 0.093P]$,
where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.39$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.37$ e Å^{-3}

The data were collected on a single-axis image plate diffractometer, leading to missing reflections in the cusp volume. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assigned on the basis of the known configuration of the starting material. Part of one molecule was modelled with disordered atoms (Fig. 3). The occupancies of the

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two disorder components were refined to 0.566 (13) and 0.434 (13). Anisotropic displacement parameters were constrained to be equal for corresponding atoms, and restraints were applied to the geometry of the disordered portions to keep them similar to the ordered molecules. Fig. 4 is a part of the F_o electron density map in the region of the disordered atoms. Attempts to model the 'split' atoms with electron density distributed along a line (Schröder *et al.*, 2004) led to slightly higher *R* values, possibly because the disorder trajectory is actually curved. It appears that both the split-atom and a very large displacement model would fit equally well. All H atoms were found in difference density syntheses. They were initially refined with soft geometry restraints to regularize their geometry (bond lengths to accepted values, angles either set by symmetry or to accepted values), after which they were refined with riding constraints only. C—H distances are all close to 0.98 Å, N—H distances are 0.83–1.09 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier atom).

Data collection: *XPRESS* (MacScience, 1989); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *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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