Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isopropyl 6-amino-2,5-anhydro-3,6-dideoxy-6-*N*-(2,5-anhydro-6-azido-3,6-dideoxy-L-*arabino*-hexonyl)-L-*arabino*-hexonate

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.005 Å
R factor = 0.054
wR factor = 0.106
Data-to-parameter ratio = 9.0

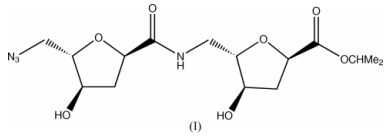
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 September 2003
Accepted 7 October 2003
Online 15 October 2003

The title compound, $C_{15}H_{24}N_4O_7$, crystallizes with great difficulty as fine hair-like crystals. It is a significant material, because it displays a weak internal hydrogen bond which may help with the interpretation of the secondary structure preferences of this class of compounds.

Comment

Sugar amino acids (SAAs) are sugars that contain amine and acid moieties and are extensively employed in the search for analogues of active peptides, predominantly as dipeptide isosteres (Schweizer, 2002). SAAs have also been utilized as chiral scaffolds that contain several orthogonal points of diversification (Sofia, 1998). Carbopeptoids are formed when SAAs are linked together *via* amide bonds and are of use as oligosaccharide mimics (Suhara *et al.*, 1996) and as foldamers (Gellman, 1998) (polymers with a strong tendency to adopt a specific compact conformation; Hill *et al.*, 2001). We have been able to identify several hydrogen-bonded conformations, analogous to known peptidic secondary structures, from carbopeptoids derived from furanose SAAs (Smith *et al.*, 2003).



(I)

The title dipeptide SAA (AE54; Fig. 1), (I), was synthesized as part of ongoing research to identify compact conformations in carbopeptoids. Its crystal structure is of particular interest to this work because of the weak hydrogen bond between the ring O atom of one furanose and the amide H atom of the other furanose ring [O6···H91 = 2.28 (4) Å and O6···H91—N9 = 110 (3)°]. Although this hydrogen bond is thought to be responsible for the conformation of the molecule, there has been no previous experimental evidence for this interaction. The crystal packing consists of helical rods with a hydrogen-bonded core and a hydrophobic exterior (Figs. 2 and 3).

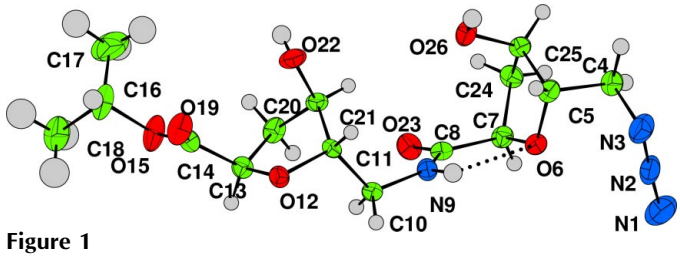


Figure 1
The molecular structure of (I), with displacement ellipsoids at the 50% probability level.

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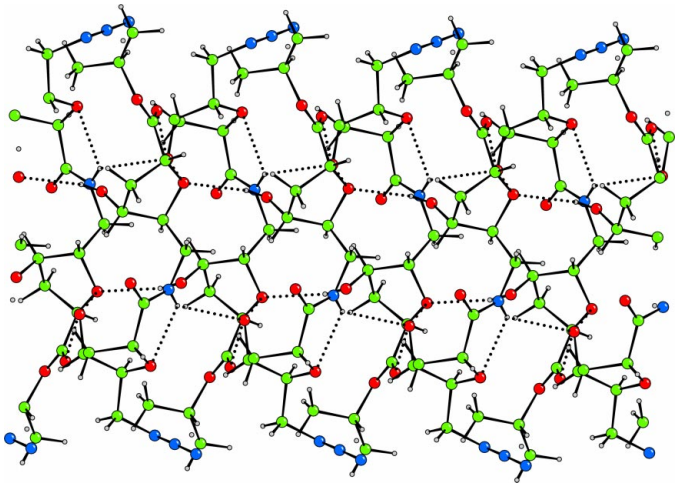

Figure 2
The hydrogen-bonded helix in (I), viewed along the *a* axis. The *b* axis (the helix direction) is horizontal.

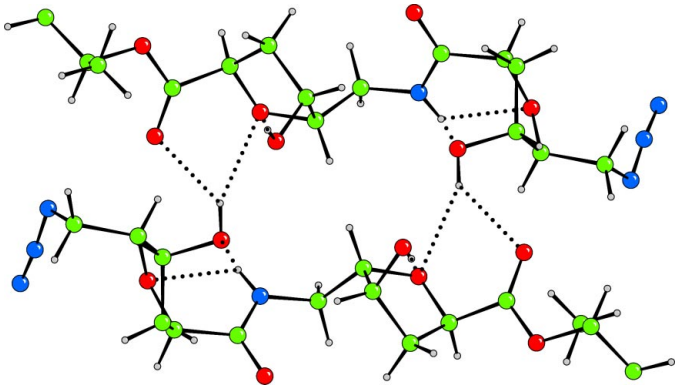

Figure 3
The hydrogen-bonded helix, viewed along its axis, the cell *b* axis.

Experimental

The title material was one of only two compounds to crystallize out of trials with four related substances. For each material, crystallization was attempted by solvent evaporation (EtOAc/cyclohexane, EtOAc/diethyl ether, EtOAc/40–60 petroleum ether) and solvent diffusion (EtOAc/diethyl ether, EtOAc/40–60 petroleum ether, dichloromethane/diethyl ether, dichloromethane/40–60 petroleum ether). This material crystallized by solvent diffusion from dichloromethane/40–60 petroleum ether as a cotton-wool-like mass, but containing two long fine needles. On cutting, one of these broke into a fibrous mass, so the second needle was used as found. The data were corrected simultaneously for absorption and irradiated volume effects by the multi-scan method (Otwinowski & Minor, 1997), giving correction factors in the range 1–1.99.

Crystal data

$C_{15}H_{24}N_4O_7$
M_r = 372.38
Monoclinic, $P2_1$
a = 9.7238 (3) Å
b = 5.6312 (2) Å
c = 16.2836 (5) Å
β = 92.0137 (14)°
V = 891.08 (5) Å^3
Z = 2
D_x = 1.388 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1985 reflections
θ = 5–27°
μ = 0.11 mm^{-1}
T = 190 K
Needle, colourless
12.30 × 0.02 × 0.01 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.5, T_{max} = 0.99
3754 measured reflections
2204 independent reflections
2204 reflections with $I > 10\sigma(I)$
R_{int} = 0.02
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 10\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.106
S = 0.95
2204 reflections
244 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F^2) + (0.0472P)^2 + 0.453P]$, where $P = [2\max(F_o^2,0) + F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.131 (4)	C11—O12	1.456 (3)
N2—N3	1.218 (4)	O12—C13	1.437 (3)
N3—C4	1.483 (5)	C13—C20	1.533 (4)
C4—C5	1.518 (4)	C13—C14	1.508 (4)
C5—C25	1.524 (4)	C14—O19	1.197 (3)
C5—O6	1.436 (4)	C14—O15	1.328 (3)
O6—C7	1.440 (3)	O15—C16	1.475 (4)
C7—C24	1.527 (5)	C16—C18	1.497 (6)
C7—C8	1.517 (4)	C16—C17	1.424 (7)
C8—O23	1.232 (3)	C20—C21	1.515 (4)
C8—N9	1.337 (4)	C21—O22	1.433 (4)
N9—C10	1.456 (3)	C24—C25	1.526 (4)
C10—C11	1.528 (4)	C25—O26	1.431 (3)
C11—C21	1.506 (4)		
N3—N2—N1	172.6 (4)	C20—C13—C14	112.8 (3)
C4—N3—N2	114.5 (3)	C20—C13—O12	106.3 (2)
C5—C4—N3	112.3 (3)	C14—C13—O12	108.9 (2)
C25—C5—O6	107.3 (2)	O19—C14—O15	123.9 (3)
C25—C5—C4	111.3 (3)	O19—C14—C13	125.8 (3)
O6—C5—C4	109.4 (2)	O15—C14—C13	110.3 (2)
C7—O6—C5	110.1 (2)	C16—O15—C14	116.6 (2)
C24—C7—C8	112.9 (2)	C18—C16—C17	114.6 (5)
C24—C7—O6	105.2 (2)	C18—C16—O15	105.2 (3)
C8—C7—O6	113.1 (2)	C17—C16—O15	108.0 (4)
O23—C8—N9	124.3 (2)	C21—C20—C13	102.6 (2)
O23—C8—C7	118.7 (2)	O22—C21—C11	106.9 (2)
N9—C8—C7	117.0 (2)	O22—C21—C20	111.7 (2)
C10—N9—C8	122.6 (2)	C11—C21—C20	102.0 (2)
C11—C10—N9	113.8 (2)	C25—C24—C7	102.8 (2)
C21—C11—O12	104.2 (2)	O26—C25—C5	112.3 (2)
C21—C11—C10	114.6 (2)	O26—C25—C24	107.3 (2)
O12—C11—C10	107.5 (2)	C5—C25—C24	102.7 (2)
C13—O12—C11	109.2 (2)		

The small volume of the sample led to a data set which contained many weak reflections [20% with $I < 2\sigma(I)$]. The data were, however, adequate to characterize the material unambiguously. H atoms on carbon were found in a difference map, but were then positioned geometrically and refined with riding constraints. Other H atoms were located in the map and their positions were refined. The terminal isopropyl group was found to librate about the O15—C16 bond, but not sufficiently to justify a disorder model.

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* (Watkin *et al.*, 2001); molecular graphics: *CAMERON*

(Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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