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Methyl 2,4-anhydro-5-azido-5,6-dideoxy-L-altronate

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Key indicators

Single-crystal X-ray study
T = 185 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.095
Data-to-parameter ratio = 10.8

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

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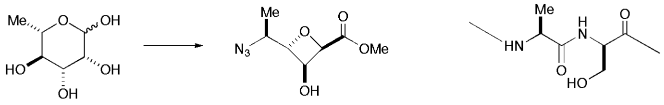
The title compound, $C_7H_{11}N_3O_4$, was prepared from L-rhamnose as a conformationally restricted dipeptide isostere containing an oxetane ring. Its crystal structure was determined to confirm the synthetic product.

Comment

Sugar amino acids (SAA) are an important class of peptidomimetics (Schweizer, 2002; Gruner *et al.*, 2002). In particular, D-amino acid scaffolds derived from pyranoses (Kriek *et al.*, 2003; El Oualid *et al.*, 2002) and furanoses (van Well *et al.*, 2003; Chakraborty *et al.*, 2002) provide a well established series of conformationally fixed dipeptide isosteres. The azido ester described here, (I), prepared from L-rhamnose, is among the first examples of building blocks for dipeptide isosteres which contain an oxetane ring; it may be viewed as a conformationally restricted dipeptide isostere of L-ala-D-ser, (II).



L-rhamnose (I) (II)

Fig. 1 shows the asymmetric unit (I). Its absolute structure (C4 R conformation, and C6 and C9 S conformation) was assumed based on the known absolute structure of the starting material.

The crystal packing for (I) consists of slightly pleated ribbons of molecules linked by weak hydrogen bonds, with the sheets stacked in van der Waals contact (Fig. 2).

Experimental

Compound (I) (Johnson *et al.*, 2004) was recrystallized from chloroform by solvent diffusion with hexane to give colourless plate-shaped crystals.

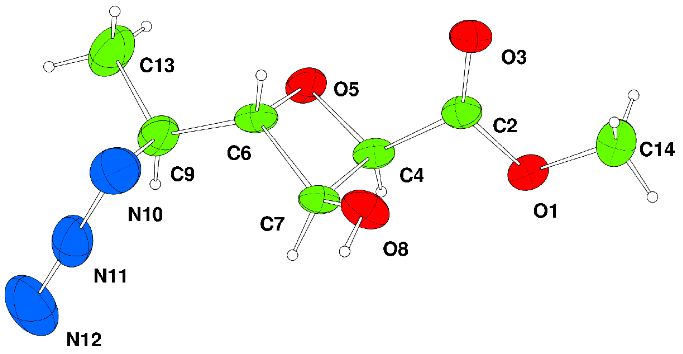


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

Crystal data

$C_7H_{11}N_3O_4$
$M_r = 201.18$
Monoclinic, $P2_1$
$a = 4.6318\ (2)$ Å
$b = 9.8575\ (5)$ Å
$c = 10.6310\ (6)$ Å
$\beta = 92.084\ (2)°$
$V = 485.07\ (4)$ Å^3
$Z = 2$
$D_x = 1.377$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1439 reflections
$\theta = 5$–$32°$
$\mu = 0.11$ mm^{-1}
$T = 185$ K
Plate, colourless
0.50 × 0.40 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
$T_{\min} = 0.96$, $T_{\max} = 0.98$
4689 measured reflections
1733 independent reflections
1733 reflections with $I > -3\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 32.0°$
$h = -6 \rightarrow 6$
$k = -14 \rightarrow 8$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.095$
$S = 1.01$
1733 reflections
160 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + (0.034P)^2 + 0.093P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.331 (2)	C6—C7	1.533 (2)
O1—C14	1.445 (3)	C6—C9	1.521 (3)
C2—O3	1.204 (2)	C7—O8	1.405 (2)
C2—C4	1.513 (3)	C9—N10	1.486 (3)
C4—O5	1.439 (2)	C9—C13	1.515 (3)
C4—C7	1.540 (2)	N10—N11	1.234 (3)
O5—C6	1.451 (2)	N11—N12	1.132 (4)
C2—O1—C14	116.48 (17)	C7—C6—C9	117.75 (15)
O1—C2—O3	124.83 (18)	C4—C7—C6	84.73 (13)
O1—C2—C4	110.25 (15)	C4—C7—O8	114.53 (15)
O3—C2—C4	124.92 (17)	C6—C7—O8	117.18 (15)
C2—C4—O5	111.04 (14)	C6—C9—N10	105.37 (17)
C2—C4—C7	114.58 (14)	C6—C9—C13	111.88 (19)
O5—C4—C7	91.58 (13)	N10—C9—C13	110.54 (19)
C4—O5—C6	91.52 (12)	C9—N10—N11	113.4 (2)
O5—C6—C7	91.38 (13)	N10—N11—N12	174.7 (3)
O5—C6—C9	110.23 (15)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O8—H5$\cdots$O3$^{\rm i}$	0.82 (4)	2.25 (3)	2.990 (2)	150 (3)
O8—H5$\cdots$O5$^{\rm i}$	0.82 (4)	2.32 (3)	2.962 (2)	135 (3)

Symmetry code: (i) $-x, \frac{1}{2}+y, 1-z$.

Because the intensity data were collected with molybdenum radiation, there were no measurable anomalous differences, as a consequence of which it was admissible to merge Freidel pairs of reflections. The absolute structure of (I) was assumed to correlate with the known absolute structure of the L-rhamnose starting material. All H atoms were found in difference-density syntheses. They were initially refined with soft restraints on the bonds to regularize their geometry (bond lengths to accepted values, angles either set by symmetry or to accepted values, and $U_{\rm iso}$ dependent upon the adjacent bonded atom), after which they were refined with riding constraints only.

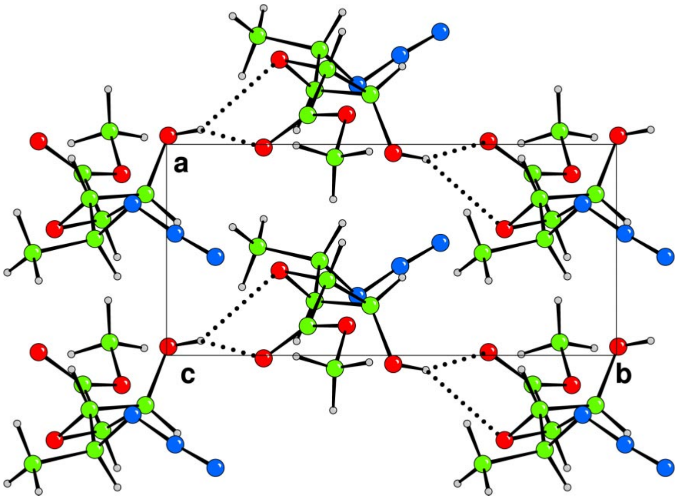


Figure 2
Packing diagram of (I), viewed down the *c* axis. The weakly hydrogen-bonded pleated ribons in the *bc* plane are simply stacked along the *a* axis. Hydrogen bonds are shown as dashed lines.

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO/SCALEPACK*; data reduction: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); 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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