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2-Acetamido-*N*-benzyl-1,4-imino-1,2,4-trideoxy-L-ribitol

Christopher C. Harding,[a]* David J. Watkin,[a] J. S. Shane Rountree,[b,c] Terry D. Butters,[c] Mark R. Wormald,[c] Raymond A. Dwek[c] and George W. J. Fleet[b]

[a]Department of Chemical Crystallography, Chemical Research Laboratory, Oxford University, Mansfield Road, Oxford OX1 3TA, England, [b]Department of Organic Chemistry, Chemical Research Laboratory, Oxford University, Mansfield Road, Oxford OX1 3TA, England, and [c]Glycobiology Institute, Department of Biochemistry, Oxford University, South Parks Road, Oxford OX1 3QU, England

Correspondence e-mail: christopher.harding@seh.ox.ac.uk

Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.086
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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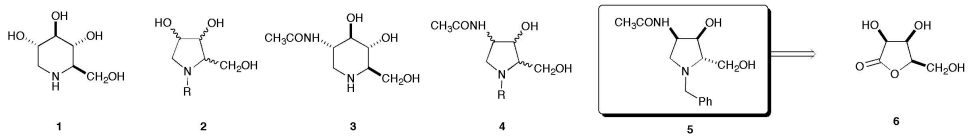
The relative configuration of the stereocentres in a potential hexosaminidase inhibitor, $C_{14}H_{20}N_2O_3$, prepared from D-lyxonolactone, has been established using X-ray crystallographic techniques.

Comment

Imino sugars, analogues of carbohydrates with the O atom of the ring replaced by an N atom, are a family of both natural products and synthetic materials which inhibit glycosidases; several such compounds have considerable therapeutic potential (Watson *et al.*, 2001; Asano *et al.*, 2000; Winchester & Fleet, 2000). For example, the natural product deoxynojirimycin, (1), is an inhibitor of a range of α-glucosidases and its derivatives have been shown to possess antiviral activity (Stütz, 1999); several related pyrrolidines, (2), are also potent inhibitors of α-glucosidases, although structure–activity relationships are not easily predictable (Asano *et al.*, 2005; Yu *et al.*, 2004; Scofield *et al.*, 1986). The synthetic *N*-acetylglucosamine analogue, (3), is a powerful hexosaminidase inhibitor (Fleet *et al.*, 1986; Boshagen *et al.*, 1987); such inhibitors have potential as anticancer agents (Woynarowska *et al.*, 1992) and for the treatment of other diseases (Liu *et al.*, 2004). By analogy with the glucosidase inhibitors, (2), a synthetic programme towards a series of diastereomeric pyrrolidines, (4), has led to the preparation of the potential hexosaminidase inhibitor, (5). While the absolute configuration of (5) is established by the use of D-lyxonolactone, (6), as the starting material, ambiguity in the relative configuration of the nitrogen substituent was removed by X-ray crystallographic analysis.



Experimental

The title compound was crystallized by cooling a warm solution in acetonitrile, forming clear block-like crystals.

Crystal data

$C_{14}H_{20}N_2O_3$
M_r = 264.32
Monoclinic, $P2_1$
a = 6.8912 (3) Å
b = 7.3504 (3) Å
c = 13.6824 (6) Å
β = 90.822 (2)°
V = 692.98 (5) Å^3
Z = 2
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1415 reflections
θ = 1–27°
μ = 0.09 mm^{-1}
T = 190 K
Block, colourless
0.20 × 0.20 × 0.10 mm

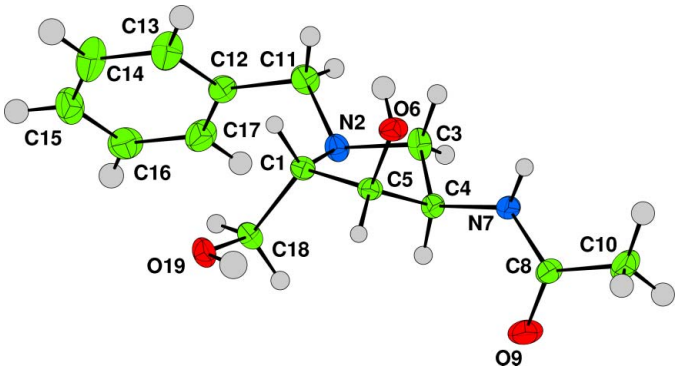


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

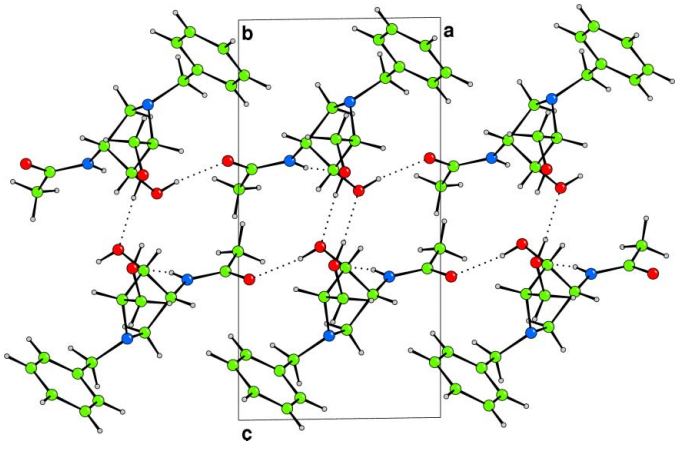


Figure 2
Packing diagram, viewed down the *b* axis. The crystal structure consists of strongly hydrogen-bonded ribbons of molecules along the *b* axis, held together by a mixture of hydrogen bonding along the *a* axis and weaker intermolecular interactions. Hydrogen bonds are represented as dotted lines.

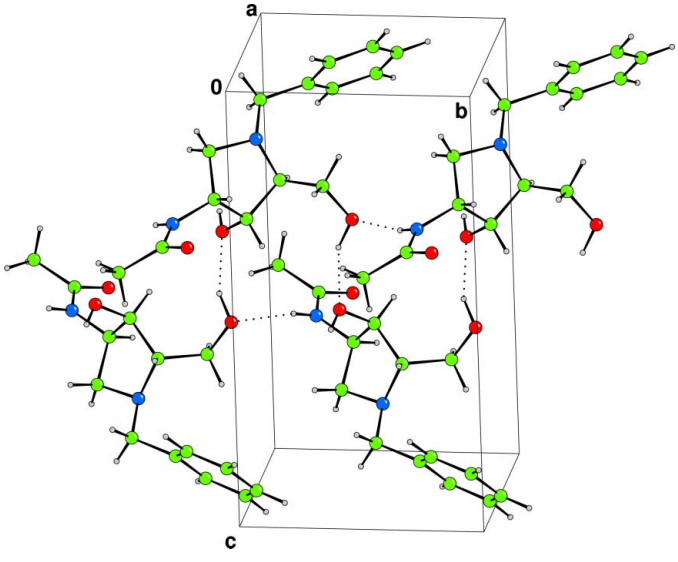


Figure 3
View of the strong hydrogen-bonding network in one of the ribbons running parallel to the *b* axis. Hydrogen bonds are represented as dotted lines.

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.98, T_{max} = 0.99
2636 measured reflections
1681 independent reflections
1499 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.086
S = 0.89
1673 reflections
172 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F_o)]^2/[33.1T_0(x) + 52.7T_1(x) + 30.8T_2(x) + 12.9T_3(x) + 3.03T_4(x)]$, where $x = F_c/F_{max}$ and $T_i(x)$ are Chebychev polynomials (Watkin, 1994; Prince, 1982)
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7—H8···O19^{i}	0.84	2.14	2.958 (2)	167
O19—H15···O6^{ii}	0.93	1.85	2.708 (2)	153
O6—H17···O9^{iii}	0.80	1.89	2.685 (2)	168

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, y+\frac{1}{2}, -z+1$; (iii) $x-1, y, z$.

All H atoms were observed in a difference electron-density map. The hydroxy and amide H atoms were refined freely, whilst the others were refined with slack restraints to optimize the geometry. They were all then made to ride on their parent atoms, with C—H distances of 0.96–1.00 Å and U_{iso}(H) = 1.2U_{eq}(parent). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration is known from the synthesis. Eight low-angle reflections were omitted from the refinement because they appeared to be obscured by the beamstop.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (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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