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Key indicators

Single-crystal X-ray study
T = 190 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.034
wR factor = 0.078
Data-to-parameter ratio = 9.9

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

1-Amino-*N,N*-dibenzyl-1,6-dideoxy- β -L-fructofuranose

The title compound, $\text{C}_{20}\text{H}_{25}\text{NO}_4$, the product formed in the Amadori rearrangement of L-rhamnose with dibenzylamine, is shown by X-ray crystallographic analysis to be a rare example of an Amadori product crystallizing in a furanose form.

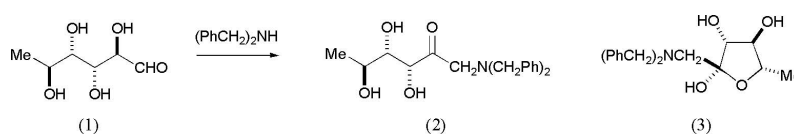
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Comment

The Amadori rearrangement, an old and complex reaction (Amadori, 1925; Hodge, 1955), is the initial step in the non-enzymatic conjugation of free amines in peptides with reducing carbohydrates to form glycation products; such materials constitute a complex and heterogeneous group of compounds which accumulate in plasma and tissues in diabetes and renal failure (Lapolla *et al.*, 2005; Smit & Lutgers, 2004). Non-enzymatic glycation has also been implicated in processes of ageing and in neurodegenerative amyloid pathologies, including Alzheimer's disease (Horvat & Jakas, 2004). Amadori ketoses are also the starting materials for the Maillard reaction (O'Brien *et al.*, 1998), the classic browning reaction of food chemistry (Martins & Van Boekel, 2005; Kwak & Lim, 2004).



L-Rhamnose, (1), on treatment with dibenzylamine in acetic acid, undergoes the Amadori rearrangement to give the

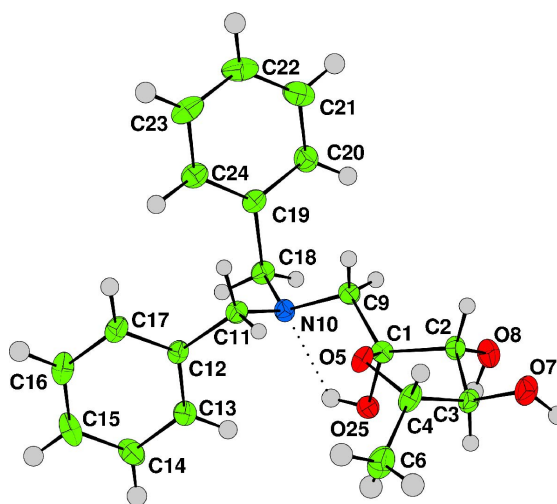


Figure 1
The molecular structure of (3), with displacement ellipsoids drawn at the 50% probability level. Also shown is an intramolecular hydrogen bond (dotted line), forming a five-membered ring with atom C9 displaced from its mean plane.

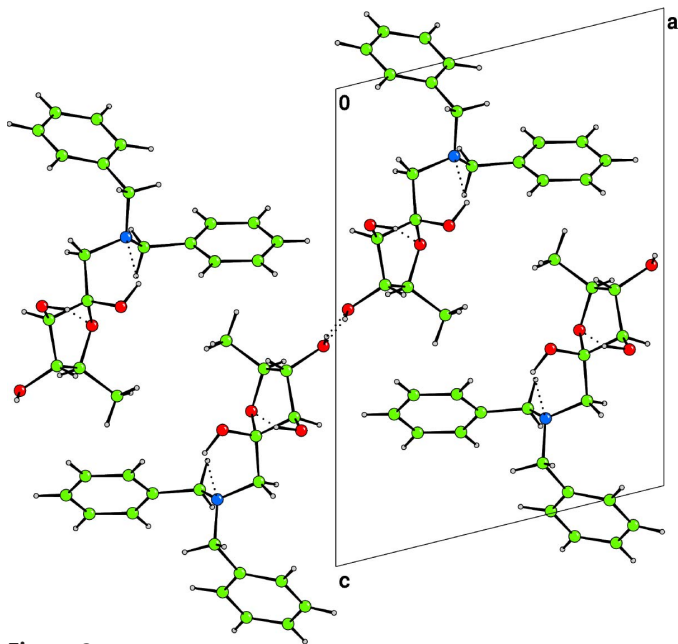


Figure 2
Packing diagram for the title compound, viewed down the *b* axis. The crystal structure is made up of columns of strongly hydrogen-bonded (dotted lines) molecules which run along the *b* axis.

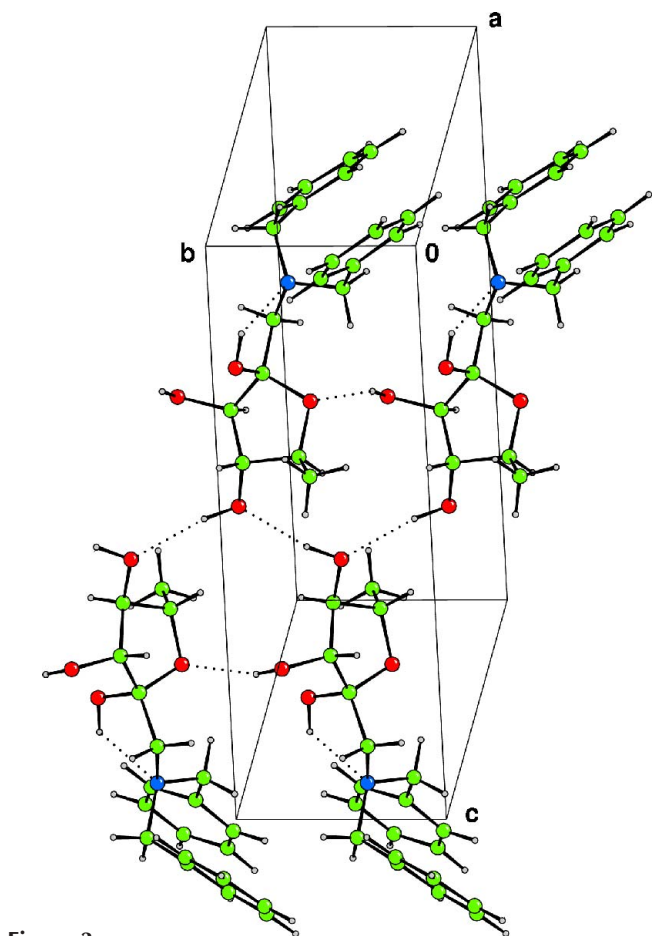


Figure 3
A view of the hydrogen-bonding (dotted lines) network in each column. Hydrogen bonds involving atom O7 form a central chain up the column, with hydrogen bonds to the furanose ring O atom from hydroxyl groups on different molecules adding support to the structure.

ketoseamine (2) (Funcke, 1978); although the solution NMR of (2) is complex and indicates a mixture of forms, the formation of crystals allowed the secure identification of the β -anomer (3). There is one other example of a furanose Amadori product (Fernández-Bolaños *et al.*, 2003).

Experimental

Crystals of the title compound were first obtained using evaporation techniques from a methanol–water mixture. They were then recrystallized from a diethyl ether/petrol solvent mixture. This yielded thin needle-like colourless crystals.

Crystal data

$C_{20}H_{25}NO_4$	$D_x = 1.283 \text{ Mg m}^{-3}$
$M_r = 343.42$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2070 reflections
$a = 10.8823 (2) \text{ \AA}$	$\theta = 1-27^\circ$
$b = 5.4690 (1) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 15.3816 (2) \text{ \AA}$	$T = 190 \text{ K}$
$\beta = 103.8824 (11)^\circ$	Block cut from needle, colourless
$V = 888.70 (3) \text{ \AA}^3$	$0.20 \times 0.20 \times 0.10 \text{ mm}$
$Z = 2$	

Data collection

Nonius KappaCCD diffractometer	2237 independent reflections
ω scans	1942 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (<i>DENZO/SCALEPACK</i> ; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.013$
$T_{\text{min}} = 0.98$, $T_{\text{max}} = 0.99$	$\theta_{\text{max}} = 27.5^\circ$
3627 measured reflections	$h = -13 \rightarrow 14$
	$k = -7 \rightarrow 6$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.09P]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = [\text{max}(F_o^2, 0) + 2F_c^2]/3$
$wR(F^2) = 0.078$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.99$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
2229 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
226 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O25–H5 \cdots N10	0.94	2.06	2.6725 (19)	121
O7–H17 \cdots O7 ⁱ	0.95	2.15	3.080 (2)	166
O8–H24 \cdots O5 ⁱⁱ	0.98	2.02	2.883 (2)	147

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

All H atoms were observed in a difference electron-density map. The hydroxyl H atoms were refined freely, whilst the others were refined with slack restraints to optimize the geometry ($C-H = 1.0 \text{ \AA}$). All were then made to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration is known from the synthesis. Several low-angle reflections were omitted from the refinement because they appeared to be obscured by the beam stop.

Data collection: *COLLECT* (Nonius, 2001); 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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