

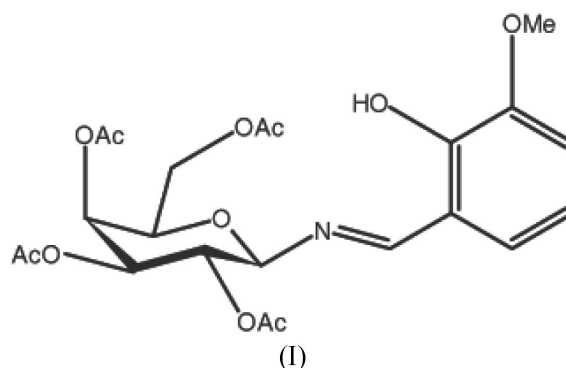
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## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.035  
 $wR$  factor = 0.094  
Data-to-parameter ratio = 9.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***N*-(2-Hydroxy-3-methoxybenzylidene)-2,3,4,6-tetra-*O*-acetyl- $\beta$ -D-galactopyranosylamine**In the title compound,  $\text{C}_{22}\text{H}_{27}\text{NO}_{11}$ , the pyranosyl ring has a chair conformation. In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bonding interactions are observed as well as intramolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bond contacts.Received 27 June 2006  
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## Comment

The asymmetric Staudinger reaction utilizing 2,3,4,6-tetra-*O*-acetyl- $\beta$ -D-galactopyranosylamine and 2,3,4,6-tetra-*O*-pivaloyl- $\beta$ -D-galactopyranosylamine as the chiral auxiliary in the synthesis of 2-azetidiones has been reported by Jarrahpour *et al.* (2004*a,b*). It has been suggested that the azomethine linkage might be responsible for the biological activities displayed by Schiff bases (Phatak *et al.*, 2000).

The molecular structure of (I) is shown in Fig. 1. The N1—C7 bond length [1.273 (3) Å] conforms to the expected value for a normal C=N bond. The methoxy group at C2 is rotated slightly around the C2—O2 bond; the torsion angle C8—O2—C2—C3 is 16.3 (4)°. The C7—C6 [1.448 (3)] and N1—C9 [1.435 (3) Å] bond lengths are consistent with those in a related structure we reported recently (Akkurt *et al.*, 2006). The pyranosyl ring adopts a chair conformation. In the crystal structure, the bond lengths and angles are in normal ranges (Akkurt *et al.*, 2006; Allen *et al.*, 1987).

A packing diagram of (I) is shown in Fig. 2. In the crystal structure, the molecular packing is stabilized by intra- and intermolecular  $\text{O}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bonding interactions (Table 1).

## Experimental

*o*-Vanillin (0.87 g, 5.71 mmol) was added to a solution of 2,3,4,6-tetra-*O*-acetyl- $\beta$ -D-galactosylamine (2.00 g, 5.76 mmol) in ethanol (35 ml). The mixture was refluxed for 5 h. The resulting yellow crystals of *N*-(2-hydroxy-3-methoxybenzylidene)-2,3,4,6-tetra-*O*-acetyl- $\beta$ -D-galac-

topyransylamine were collected in 90% yield by filtration. The title compound, (I), was recrystallized from ethanol to give prismatic pale-yellow crystals (m.p. 453–455 K). IR (KBr): 3150–3250 (OH), 1751.2 (C=O), 1635.5 (C=N)  $\text{cm}^{-1}$ .  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 62.9 MHz):  $\delta$  170.43–168.30 (4 C=O), 164.63 (C=N), 150.79–114.83 (aromatic carbons), 89.31 (C3), 72.83 (C4), 71.40 (C2), 69.77 (C6), 68.31 (C1), 61.44 (C5), 56.07 ( $\text{OCH}_3$ ), 20.69–20.56 (4  $\times$   $\text{COCH}_3$ ). MS ( $m/z$ ): 481, 331, 169, 109, 43.

Crystal data

$\text{C}_{22}\text{H}_{27}\text{NO}_{11}$   $Z = 2$   
 $M_r = 481.45$   $D_x = 1.303 \text{ Mg m}^{-3}$   
 Monoclinic,  $P2_1$  Mo  $K\alpha$  radiation  
 $a = 8.6855 (7) \text{ \AA}$   $\mu = 0.11 \text{ mm}^{-1}$   
 $b = 7.6735 (5) \text{ \AA}$   $T = 296 \text{ K}$   
 $c = 18.5577 (15) \text{ \AA}$  Prism, pale yellow  
 $\beta = 97.124 (7)^\circ$   $0.66 \times 0.55 \times 0.42 \text{ mm}$   
 $V = 1227.29 (16) \text{ \AA}^3$

Data collection

Stoe IPDS-2 diffractometer 19328 measured reflections  
 $\omega$  scans 2862 independent reflections  
 Absorption correction: integration 2154 reflections with  $I > 2\sigma(I)$   
 ( $X\text{-RED32}$ ; Stoe & Cie, 2002)  $R_{\text{int}} = 0.102$   
 $T_{\text{min}} = 0.934$ ,  $T_{\text{max}} = 0.957$   $\theta_{\text{max}} = 27.2^\circ$

Refinement

Refinement on  $F^2$  H atoms treated by a mixture of  
 $R[F^2 > 2\sigma(F^2)] = 0.035$  independent and constrained  
 $wR(F^2) = 0.094$  refinement  
 $S = 0.91$   $w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$   
 2862 reflections where  $P = (F_o^2 + 2F_c^2)/3$   
 317 parameters  $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.12 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{O1---HO1}\cdots\text{N1}$	0.92 (4)	1.80 (4)	2.641 (3)	151 (3)
$\text{C3---H3}\cdots\text{O11}^i$	0.93	2.45	3.342 (4)	160
$\text{C7---H7}\cdots\text{O3}$	0.93	2.20	2.607 (3)	105
$\text{C10---H10}\cdots\text{O5}$	0.98	2.31	2.684 (3)	102
$\text{C11---H11}\cdots\text{O7}$	0.98	2.25	2.675 (3)	105
$\text{C12---H12}\cdots\text{O5}^{ii}$	0.98	2.55	3.310 (3)	134
$\text{C12---H12}\cdots\text{O9}$	0.98	2.29	2.684 (4)	103
$\text{C13---H13}\cdots\text{O1}^{ii}$	0.98	2.60	3.557 (3)	167
$\text{C15---H15A}\cdots\text{O7}^{iii}$	0.96	2.53	3.422 (5)	155
$\text{C17---H17A}\cdots\text{O9}^{iv}$	0.96	2.27	3.209 (5)	164

Symmetry codes: (i)  $-x + 1, y - \frac{3}{2}, -z + 1$ ; (ii)  $x, y + 1, z$ ; (iii)  $x, y - 1, z$ ; (iv)  $-x + 1, y - \frac{1}{2}, -z$ .

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. The phenol H atom was located in a difference Fourier map and refined freely. H atoms bonded to C were included in calculated positions ( $\text{C---H} = 0.93\text{--}0.98 \text{ \AA}$ ) and refined using a riding-model approximation with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ . The methyl groups were allowed to rotate but not to tip.

Data collection:  $X\text{-AREA}$  (Stoe & Cie, 2002); cell refinement:  $X\text{-AREA}$ ; data reduction:  $X\text{-RED32}$  (Stoe & Cie, 2002); program(s) used to solve structure:  $\text{SHELXS97}$  (Sheldrick, 1997); program(s) used to refine structure:  $\text{SHELXL97}$  (Sheldrick, 1997); molecular graphics:  $\text{ORTEP-3 for Windows}$  (Farrugia, 1997); software used to prepare material for publication:  $\text{WinGX}$  (Farrugia, 1999).

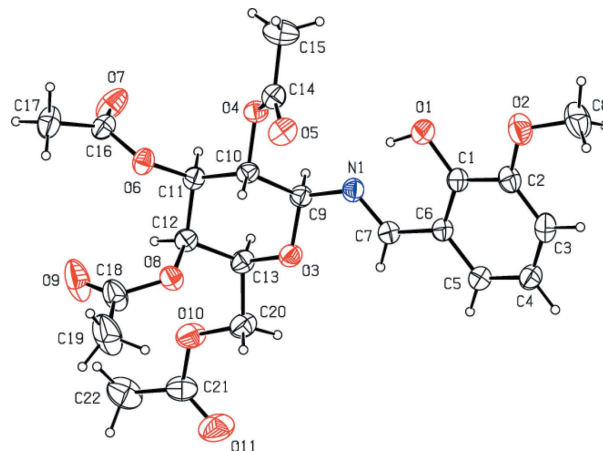


Figure 1

A view of (I), showing the atom-numbering scheme and 20% probability displacement ellipsoids.

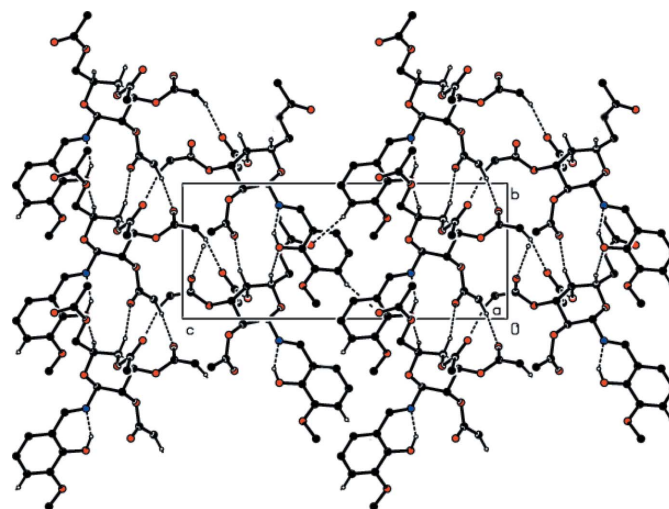


Figure 2

A packing diagram of (I), viewed along the  $a$  axis. For clarity, only those H atoms involved in hydrogen bonds (dashed lines) are shown.

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