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2,3,4,6-Tetra-*O*-acetyl-*N*-(2-hydroxybenzylidene)-β-D-galactopyranosylamine

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.041
wR factor = 0.102
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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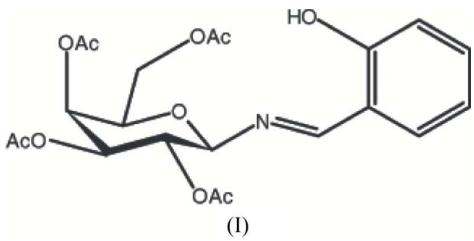
In the title compound, $C_{21}H_{25}NO_{10}$, the six-membered pyranosyl ring adopts a chair conformation. The crystal structure contains intra- and intermolecular hydrogen-bonding interactions.

Comment

Schiff bases are important intermediates for the synthesis of different bioactive compounds (Jarrahpour *et al.*, 2004*a*,*b*; Hakimelahi & Jarrahpour 1989; Hakimelahi & Jarrahpour 1990; Venturini & Gonzalez 2002; Taggi *et al.*, 2002; Halve & Goyal 1996). They are also suitable complexing agents, especially the bis Schiff bases (Jarrahpour *et al.*, 2006; Jarrahpour & Rezaei 2006). It has been proposed that when the Schiff base is derived from an optically active amine such as 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosylamine and an achiral aldehyde, a high degree of diastereoselectivity in the [2 + 2] cycloadditions is achieved (Barton *et al.*, 1990; Jarrahpour *et al.*, 2004*a*,*b*). The asymmetric Staudinger reaction utilizing 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosylamine and 2,3,4,6-tetra-*O*-pivaloyl-β-D-galactopyranosylamine as the chiral auxiliary in the synthesis of 2-azetidinones 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).

As a continuation of our research program on new chiral sugar-based Schiff base derivatives of biological interest, we have synthesized new compounds bearing an azomethine linkage. These Schiff bases (azomethines) were purposely designed to combine the sugar ring with an arylidene group bearing different substituents (Jarrahpour *et al.*, 2005*a*,*b*).

The molecular structure of the title compound, (I), with the atom-numbering scheme, is shown in Fig. 1. The C7═N1 distance of 1.259 (3) Å is consistent with a C═N double bond. All bond distances and angles in (I) are within normal ranges (Allen *et al.*, 1987). The pyranosyl ring adopts a chair conformation. The C8—N1═C7—C6 torsion angle is 179.4 (2)°. The –C8–N1═C7–C6- group is coplanar with the phenol group.



(I)

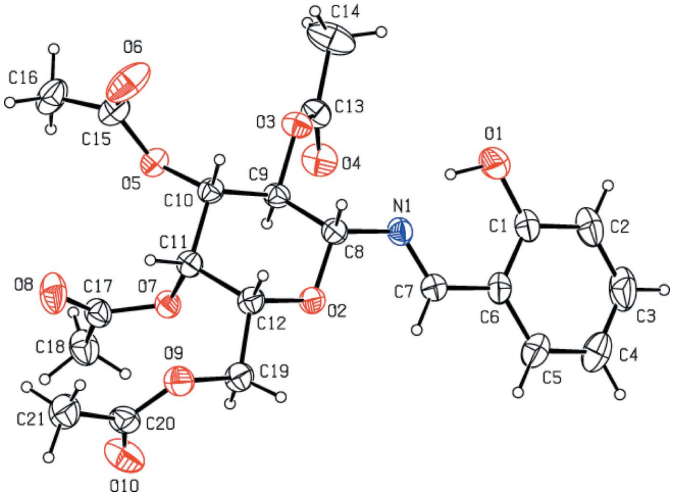


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), with the atom-numbering scheme and 20% probability displacement ellipsoids.

In the crystal structure of (I), molecules are linked along the *b* axis by C—H···O type hydrogen contacts (Table 2 and Fig. 2).

Experimental

Salicylaldehyde (0.70 g, 0.6 ml, 5.73 mmol) was added to a solution of 2,3,4,6-tetra-*O*-acetyl-β-D-galactosylamine (2.00 g, 5.76 mmol) in ethanol (35 ml). The mixture was refluxed for 5 h. The resulting pale-yellow crystals were collected (in 91% yield) by filtration. They were then recrystallized from ethanol as prismatic colorless crystals. The ^{13}C NMR spectrum showed ($COCH_3$) at 21.15–20.92 p.p.m., (Ar) at 136.05–116.32 p.p.m. and (C═O) at169.51–168.55 p.p.m. M.p. 411–413 K. IR (KBr, cm^{-1}): 3275–3150 (OH), 1743.5 (C═O), 1635.5 (C═N). ^{1}H NMR ($CDCl_3$, 250 MHz, p.p.m.): 12.24(OH, *s*, 1H), 8.50 (NCH, *s*, 1H), 7.38–6.78 (Ar—H, *m*, 4H), 5.48–4.02 (sugar H atoms, *m*, 7H), 2.16–2.02 ($COCH_3$, *s*, 3H), 2.06 ($COCH_3$, *s*, 3H), 2.01 ($COCH_3$, *s*, 3H), 1.93($COCH_3$, *s*, 3H). ^{13}C NMR ($CDCl_3$, 62.9 MHz, p.p.m.): 169.51–168.55 (4C═O), 159.94 (C═N), 136.05–116.32 (aromatic carbons), 88.64 (sugar carbon, C3), 71.87 (sugar carbon, C4), 70.34 (sugar carbon, C2), 68.83 (sugar carbon, C6), 67.06 (sugar carbon, C1), 60.77 (sugar carbon, C5), 19.78–19.68 (4$COCH_3$). MS (*m*/*z*): 451, 331, 169, 109, 43.

Crystal data

$C_{21}H_{25}NO_{10}$
M_r = 451.42
Orthorhombic, $P2_12_12_1$
a = 7.8014 (4) Å
b = 15.2381 (11) Å
c = 19.1271 (10) Å
V = 2273.8 (2) Å^3
Z = 4
D_x = 1.319 Mg m^{-3}
Mo *Kα* radiation
μ = 0.11 mm^{-1}
T = 296 K
Prism, colorless
0.57 × 0.50 × 0.45 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.942, T_{max} = 0.954
16920 measured reflections
2894 independent reflections
2002 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.102
S = 0.98
2894 reflections
298 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0146 (18)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.344 (4)	O7—C11	1.442 (3)
O2—C8	1.409 (3)	O7—C17	1.347 (3)
O2—C12	1.439 (3)	O8—C17	1.198 (4)
O3—C9	1.436 (3)	O9—C19	1.446 (3)
O3—C13	1.354 (3)	O9—C20	1.319 (4)
O4—C13	1.189 (3)	O10—C20	1.188 (4)
O5—C10	1.435 (3)	N1—C8	1.445 (3)
O5—C15	1.346 (3)	N1—C7	1.259 (3)
O6—C15	1.192 (4)		
C8—O2—C12	112.27 (18)	O7—C11—C12	109.05 (19)
C9—O3—C13	117.56 (19)	O2—C12—C11	110.85 (19)
C10—O5—C15	118.7 (2)	O2—C12—C19	104.6 (2)
C11—O7—C17	118.21 (18)	O3—C13—C14	110.8 (3)
C19—O9—C20	118.2 (2)	O4—C13—C14	126.4 (3)
C7—N1—C8	121.6 (2)	O3—C13—O4	122.8 (2)
O1—C1—C2	118.2 (3)	O5—C15—C16	112.0 (3)
O1—C1—C6	121.7 (2)	O6—C15—C16	126.1 (3)
N1—C7—C6	122.0 (2)	O5—C15—O6	121.9 (3)
O2—C8—C9	108.40 (19)	O7—C17—C18	111.6 (2)
N1—C8—C9	109.63 (19)	O8—C17—C18	124.8 (3)
O2—C8—N1	111.83 (19)	O7—C17—O8	123.6 (3)
O3—C9—C10	107.87 (18)	O9—C19—C12	107.4 (2)
O3—C9—C8	110.96 (19)	O9—C20—C21	111.0 (3)
O5—C10—C11	108.65 (19)	O10—C20—C21	125.7 (3)
O5—C10—C9	108.68 (19)	O9—C20—O10	123.3 (3)
O7—C11—C10	107.83 (19)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—HO1···N1	0.96 (4)	1.71 (4)	2.579 (3)	150 (3)
C7—H7···O2	0.93	2.26	2.643 (3)	104
C8—H8···O10^{i}	0.98	2.48	3.364 (4)	150
C9—H9···O4	0.98	2.28	2.678 (3)	103
C10—H10···O6	0.98	2.26	2.681 (4)	104
C11—H11···O4^{ii}	0.98	2.58	3.401 (3)	141
C11—H11···O8	0.98	2.30	2.707 (4)	104
C14—H14*C*···O6^{iii}	0.96	2.52	3.287 (5)	137
C16—H16*C*···O8^{iv}	0.96	2.45	3.261 (4)	143
C19—H19*A*···O10	0.97	2.25	2.671 (4)	105

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

The phenol H atom was located in a difference Fourier map and refined freely. All other H atoms were positioned geometrically and treated as riding, with C–H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

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

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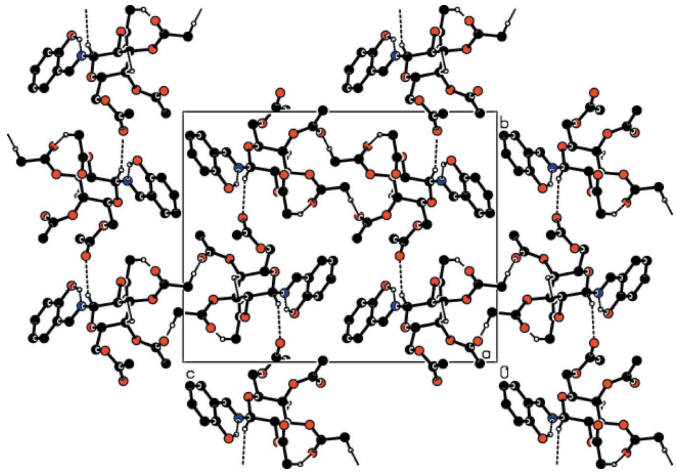

Figure 2
A view of the packing of (I), down the *a* axis. Hydrogen bonds are shown as dashed lines and H atoms not involved in these interactions have been omitted.

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