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2-Hydroxy-*N*-(2,3,4,6-tetra-*O*-pivaloyl-β-D-glucopyranosyl)benzylideneamine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.110
Data-to-parameter ratio = 9.2

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

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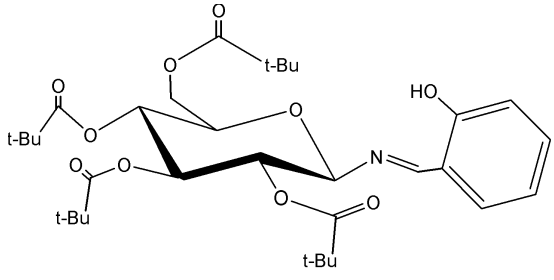
The title compound $C_{33}H_{49}NO_{10}$, which is an important chiral template for the synthesis of chiral amino compounds, has been successfully synthesized. The glucopyranose ring adopts a chair conformation. There is an intramolecular O—H···N hydrogen bond.

Comment

Imine chemistry has achieved a dramatic development in recent decades. Due to their importance in organic synthesis, many nitrogen-substituted imines have been developed and applied to the synthesis of α-amino acids, β-lactams, heterocyclic alkaloids, aziridines and amines (Enders & Reinhold, 1997). Kunz's group has reported the use of *O*-pivaloylated D-galactosylimines as chiral auxiliaries for stereoselective synthesis of amino acids (Kunz *et al.*, 1991) and other nitrogen compounds (Follmann *et al.*, 2001). Compared to *O*-pivaloylated D-galactosylimine, all functional groups necessary for the stereodifferentiation are the same in *O*-pivaloylated D-glucosylimine. Additionally, *O*-pivaloylated D-glucosylimine is more stable and less expensive. With the aim of gaining further insight into the structural aspects responsible for the stereoselective synthesis, the crystallographic analysis of the title compound, (I), has been carried out.



(I)

The molecular structure of (I), with the atom-numbering scheme, is shown in Fig. 1. The glucopyranose ring assumes a chair conformation in the solid state and is in the β anomeric configuration (Table 1). A strong intramolecular hydrogen bond occurs between the hydroxy O atom and the azomethine N atom (Table 2).

In the crystal structure, there are only ordinary, weak, van der Waals interactions.

Experimental

To a solution of 2,3,4,6-tetra–*O*-pivaloyl-β-D-glucopyranosylamine (5.15 g, 10 mmol) in 2-propanol (25 ml) were added salicylaldehyde (1.82 g, 15 mmol) and 15 drops of acetic acid. After 1 h, 2-hydroxyl-*N*-(2,3,4,6-tetra-*O*-pivaloyl-β-D-glucopyranosyl)benzyl-

ideneamine, (I), precipitated from the 2-propanol solution and was collected by filtration and rapidly washed with ice-cooled 2-propanol (m.p. 469–490 K). ^{1}H NMR: δ 8.55 (1H), 7.34 (1H), 7.28 (1H), 6.95 (1H), 6.88 (1H), 5.48 (1H), 5.24 (1H), 5.06 (1H), 4.97 (1H), 4.32 (1H), 4.18 (1H), 3.92 (1H), 1.07–1.29 (36H). Single crystals suitable for X-ray structure analysis were obtained by slow evaporation from methanol at room temperature.

Crystal data

$C_{33}H_{49}NO_{10}$
M_r = 619.73
Orthorhombic, $P2_12_12_1$
a = 11.1774 (8) Å
b = 11.2461 (8) Å
c = 29.072 (2) Å
V = 3654.4 (4) Å^3
Z = 4
D_x = 1.126 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2270 reflections
θ = 4.6–37.1°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, yellow
0.37 × 0.27 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.907, T_{max} = 0.980
20250 measured reflections
4140 independent reflections
3502 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 26.0°
$h = -13 \rightarrow 13$
$k = -12 \rightarrow 13$
$l = -35 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.110
S = 0.87
4035 reflections
440 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Table 1
Selected torsion angles (°).

C1—C2—C3—C4	55.5 (4)	C4—C5—O1—C1	−64.3 (3)
C2—C3—C4—C5	−53.5 (4)	C5—O1—C1—C2	66.3 (3)
C3—C4—C5—O1	57.3 (4)	O1—C1—C2—C3	−61.6 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1	0.82	1.92	2.588 (4)	137

H atoms were placed in calculated positions with C—H = 0.96 Å and included in the final cycles of refinement as riding, with U_{iso}(H) = 1.2U_{eq} of the carrier atom. All C atoms belonging to *tert*-butyl groups have large displacement parameters but only one of the four *tert*-butyl groups could be modelled as disordered on six positions with occupancy factors of roughly 0.5. Friedel reflections were merged before the final refinement.

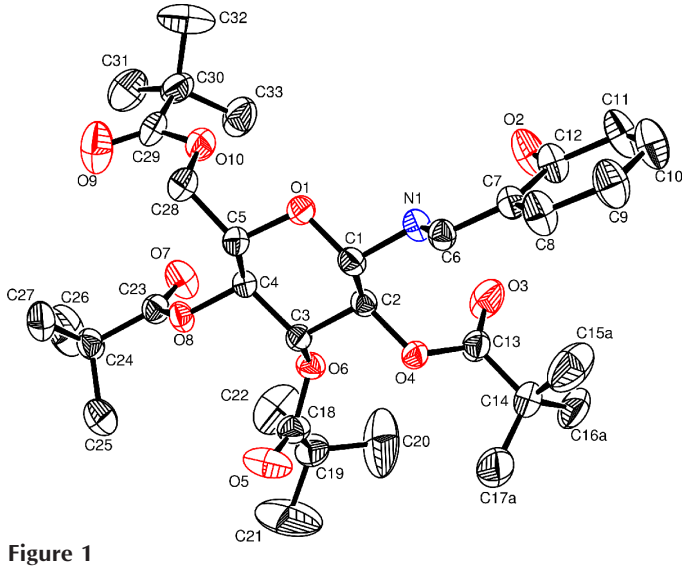


Figure 1
ORTEP-3 view (Farrugia, 1997) of the title molecule, showing the atomic numbering scheme. Ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity and only the minor component of the disordered butyl group is shown.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1999).

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