Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
R factor = 0.055
wR factor = 0.134
Data-to-parameter ratio = 10.1

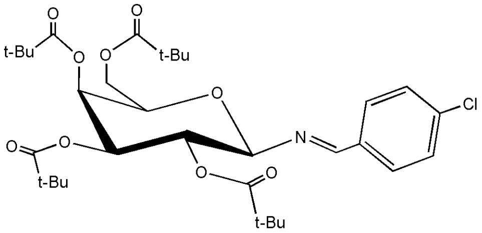
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 September 2004
Accepted 23 September 2004
Online 30 September 2004

The title compound, $C_{33}H_{48}ClNO_9$, is an important chiral template for the synthesis of α-amino acids. The galactopyranose ring adopts a chair conformation. The crystal structure contains intermolecular C—H···O interactions.

Comment

The *N*-(*O*-pivaloyl-β-D-galactopyranosyl)benzylideneamines, which are effective chiral templates, have been used for the stereoselective synthesis of α-amino acids (Kuns *et al.*, 1991). These authors have tried to rationalize the stereoselection during the Strecker reaction, by NOE NMR experiments on the aldimines. To further understand these processes, a crystallographic analysis of the title compound, (I), has been carried out and the results are presented in this paper.



(I)

The molecular structure of (I), with the atom-numbering scheme, is shown in Fig. 1. The galactopyranose ring assumes a chair conformation in the solid state and is in the β anomeric configuration. The torsion angles N1—C6—C7—C12, C6—N1—C1—O1 and C6—N1—C1—C2 are 1.40 (7), 146.95 (3) and 94.47 (4)°, respectively. The N1—C6 distance of 1.260 (6) Å is consistent with a C═N double bond. Other bond lengths and angles are unexceptional.

The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

Experimental

To a solution of 2,3,4,6-tetra-*O*-pivaloyl-β-D-galactopyranosylamine (5.15 g, 10 mmol) in 2-propanol (25 ml), 4-chlorobenzaldehyde (2.11 g, 15 mmol) and 15 drops of acetic acid were added. After 1 h, 4-chloro-*N*-(2,3,4,6-tetra-*O*-pivaloyl-β-D-galactopyranosyl)benzylideneamine precipitated from the 2-propanol solution, was collected by filtration and rapidly washed with ice-cold 2-propanol (m.p. 415–418 K, determined on an X4-Data microscopic melting point apparatus). ^{1}H NMR (recorded on a DMX 500 MHz AVANCE spectrometer in $CDCl_3$, using TMS as an internal standard; p.p.m.): 8.39 (1H), 7.68 (1H), 7.38 (1H), 5.52 (1H), 5.26 (2H), 4.68 (1H), 4.28 (1H), 4.16 (1H), 4.09 (1H), 1.04–1.29 (36H).

doi: 10.1107/S1600536804023876

Crystal data

$C_{33}H_{48}ClNO_9$
M_r = 638.20
Orthorhombic, $P2_12_12_1$
a = 10.9898 (4) Å
b = 15.7406 (7) Å
c = 20.7781 (6) Å
V = 3594.3 (2) Å^3
Z = 4
D_x = 1.179 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 22 991 reflections
θ = 2.7–27.5°
μ = 0.16 mm^{-1}
T = 295 (1) K
Block, colorless
0.31 × 0.28 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min}$ = 0.921, $T_{\max}$ = 0.982
31 396 measured reflections

8173 independent reflections
4014 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int}$ = 0.065
$\theta_{\max}$ = 27.5°
$h = -14 \rightarrow 14$
$k = -20 \rightarrow 20$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.134
S = 1.01
4014 reflections
398 parameters
H-atom parameters constrained

$w = (4F_o^2)/[0.0024F_o^2 + \sigma(F_o^2)]$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.57 e Å^{-3}
$\Delta\rho_{\min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), 3568 Friedel pairs
Flack parameter = 0.1 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O9$^{\rm i}$	0.97	2.63	3.479 (4)	147
C8—H7···O7$^{\rm ii}$	0.97	2.47	3.379 (5)	156
C15—H11···O3$^{\rm iii}$	0.97	2.62	3.403 (7)	138

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

H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the final cycles of refinement in the riding model approximation, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$ of the carrier atoms.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

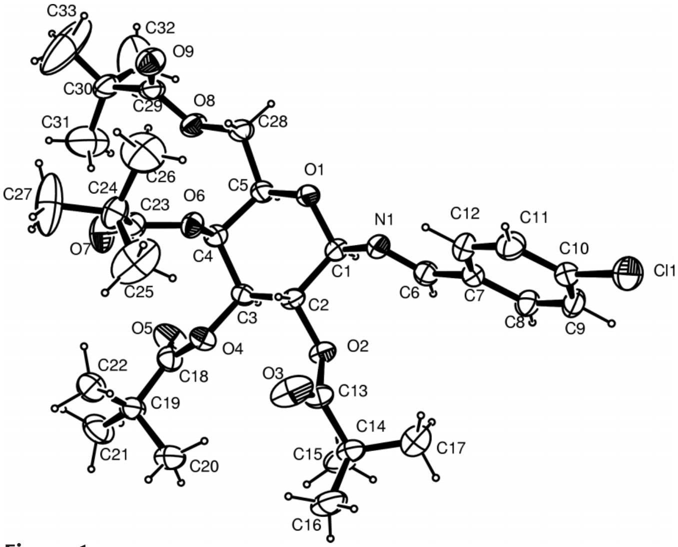


Figure 1
A view of the title molecule, showing the atomic numbering scheme and 30% probability displacement ellipsoids.

The authors thank the National Natural Science Foundation of China (No. 203760160) and the Natural Science Foundation of Zhejiang Province (No. 202075).

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