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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
Disorder in main residue
 R factor = 0.058
 wR factor = 0.171
Data-to-parameter ratio = 11.1

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

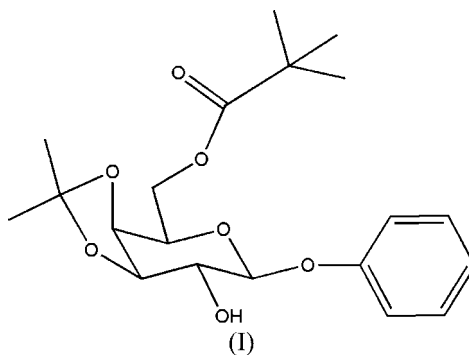
Phenyl 3,4-*O*-isopropylidene-6-*O*-pivalyl- β -D-galactopyranoside

In the title compound, $\text{C}_{20}\text{H}_{28}\text{O}_7$, the six-membered sugar ring adopts a distorted chair conformation. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into zigzag chains extending along the a axis. The crystal packing is further stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Carbohydrates play a central role in the synthesis of various biological natural products (Berlin *et al.*, 1969; Berlin *et al.*, 1974). In particular, galactose is a monosaccharide exploited as a building block in various biological structures (Du *et al.*, 2000; Knuhr *et al.*, 2001). In this paper, we report the crystal structure of the title compound, (I).



In compound (I) (Fig. 1), the bond lengths and angles are normal and comparable with those in published related structures (Abboud *et al.*, 1997; Brito-Arias *et al.*, 2002). The six-membered sugar ring adopts a distorted chair conformation. The conformation of the 1,3-dioxolane ring is a half chair, with a total puckering amplitude (Cremer & Pople, 1975) $Q_T = 0.364$ (2) Å and a pseudo-twofold axis running in the direction through C6 and the mid-point of the C2—C3 bond.

Intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1) link the molecules into zigzag chains extending along the a axis. The crystal packing (Fig. 2) is further stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1).

Experimental

To an ice-cold solution of phenyl 3,4-*O*-isopropylidene- β -D-galactopyranoside (1.0 mmol) and pyridine (3.0 mmol) in dichloromethane (20 ml), pivalyl chloride (1.1 mmol) was added dropwise. The reaction mixture was then stirred at ambient temperature overnight. The organic layer was washed with dilute HCl and water, dried with anhydrous sodium sulfate and evaporated *in vacuo*. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a

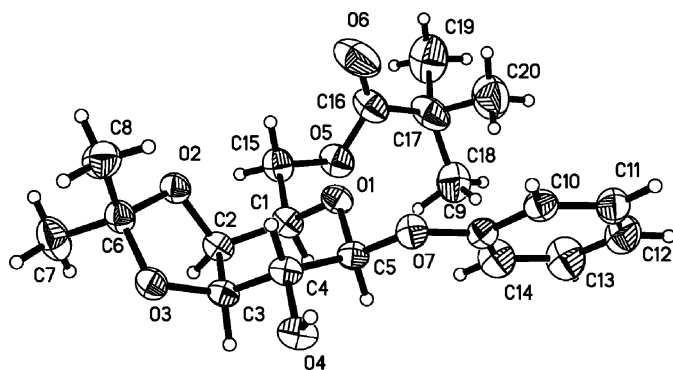


Figure 1

A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major component of the disordered *tert*-butyl group is shown.

solution of (I) in ethyl acetate and petroleum ether (1:4 v/v) at room temperature for one week.

Crystal data

$C_{20}H_{28}O_7$	$Z = 4$
$M_r = 380.42$	$D_x = 1.178 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 5.8611 (11) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 18.529 (3) \text{ \AA}$	$T = 298 (2) \text{ K}$
$c = 19.756 (4) \text{ \AA}$	Block, colourless
$V = 2145.5 (7) \text{ \AA}^3$	$0.5 \times 0.34 \times 0.2 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	12805 measured reflections
φ and ω scans	2897 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1993 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.970$, $T_{\max} = 0.981$	$R_{\text{int}} = 0.037$
	$\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1046P)^2 + 0.091P]$
$R[F^2 > 2\sigma(F^2)] = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.171$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
2897 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
260 parameters	
H-atom parameters constrained	

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O4-H4A\cdots O3^i$	0.82	1.94	2.754 (3)	173
$C3-H3A\cdots O7^{ii}$	0.98	2.59	3.570 (4)	175
$C10-H10A\cdots O4^{iii}$	0.93	2.61	3.437 (4)	148

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

All H atoms were placed in calculated positions, with $C-H = 0.93-0.98 \text{ \AA}$, and $O-H = 0.82 \text{ \AA}$, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the aryl and methane H atoms, or $1.5U_{\text{eq}}(\text{O}, \text{C})$ for the hydroxyl and methyl H atoms. The *tert*-butyl group (atoms C17, C18, C19 and C20 with their attached H atoms) was treated as disordered over two orientations, with refined occupancies of 0.630 (7) and 0.370 (7). Due to the absence

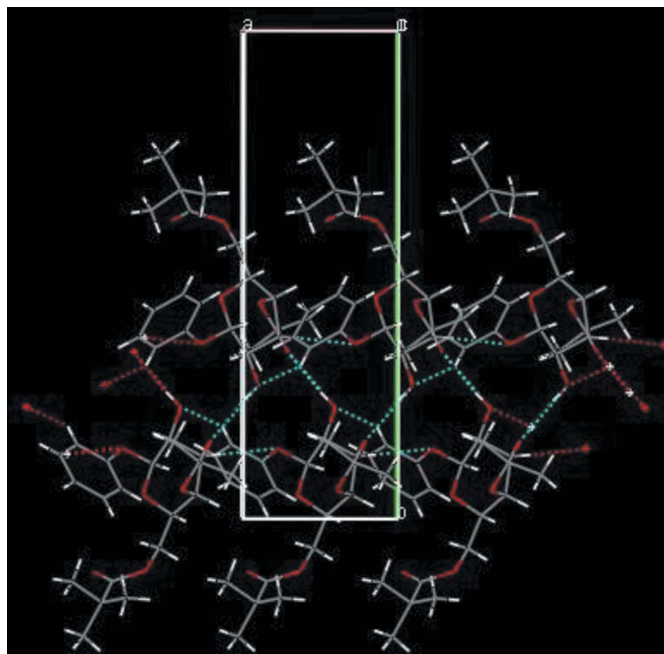


Figure 2

A packing diagram for (I), viewed down the c axis. Hydrogen bonds are shown as dashed lines. Only the major component of the disordered *tert*-butyl group is shown.

of any significant anomalous scatterers in the molecule, the 2901 Friedel pairs were merged before the final refinement. The absolute configuration of the title compound was assigned from a knowledge of the stereochemistry of its synthetic precursor.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999) and *MERCURY* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

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