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2,3,4,6-Tetra-*O*-acetyl-*O*-(*p*-*tert*-butylphenyl)-β-D-glucopyranoside

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.128
Data-to-parameter ratio = 10.6

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

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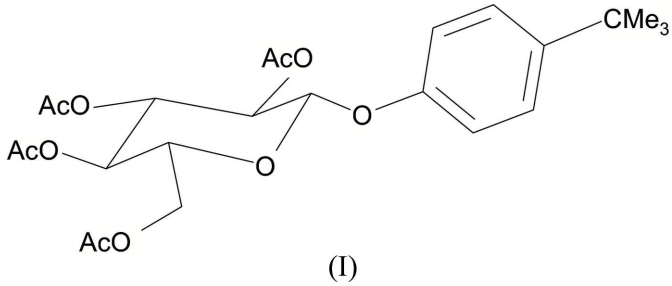
The crystal structure of the title compound, $C_{24}H_{32}O_{10}$, is stabilized by weak C—H···O hydrogen bonds. The six-membered glucopyranosyl ring adopts a chair conformation.

Comment

A variety of efficient methods are available for the synthesis of both *O*– and *C*-glycosides. They have contributed enormously to the flexible design of artificial glycoconjugates possessing high medicinal potential (Sears & Wong, 1999; Gruner *et al.*, 2002; Chiara & Sesmilo, 2002). A method used for the synthesis of aryl *C*-glycosides involves the coupling of 2-deoxysugars with phenols in the presence of a trifluoromethylsilyl trifluoromethanesulfonate–silver perchlorate (TMSOTf–$AgClO_4$) catalyst system (Toshima *et al.*, 1998). In an attempt to synthesize aryl *C*-glycosides by this method, an anomeric mixture of aryl *O*-glycosides was obtained instead of *C*-glycosides. Here, we report the structure of one of the anomers of 2,3,4,6-tetra-*O*-acetyl-*O*-(*p*-*tert*-butylphenyl)-β-D-glucopyranoside, (I). The structures of nitrophenyl-2,3,4,6-tetra-*O*-acetyl-β-D-gluco- and -D-galactopyranosides have been reported recently (Temeriusz *et al.*, 2005).



(I)

The molecular structure of (I) contains two six-membered rings linked by an ether O atom (Fig. 1). The six-membered O1/C1–C5 ring adopts a chair conformation with absolute values of torsion angles in the range 48.1 (3)–67.4 (3)°, which are quite different from the torsion angles of a free cyclohexyl ring (Bucourt, 1974). However, they are similar in range to those reported for the analogous compounds *p*-nitrophenyl-2,3,4,5-tetra-*O*-acetyl-β-D-glucopyranoside (Abboud *et al.*, 1997), with endocyclic torsion angles in the range 47.2 (2)–71.5 (2)°, and 2,3,4,5-tetra-*O*-acetyl-3,4-dinitrophenyl-β-D-glucopyranoside (Jones *et al.*, 1982), with absolute torsion angles in the range 45.7 (3)–63.2 (3)°. The large deviation in torsion angles in (I) is likely to be a consequence of the steric effect of the *para*-*tert*-butylphenyl ring that is connected to C5 *via* the ether linkage, similar to the deviation resulting from the *p*-nitrophenyl group in the above-mentioned glucopyran-

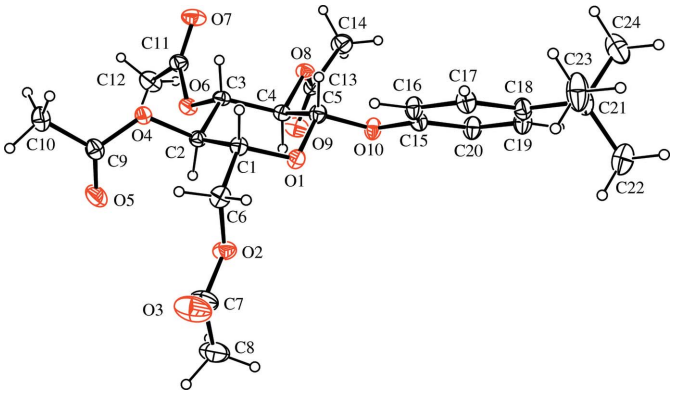


Figure 1
The molecular structure of (I), with displacement ellipsoids plotted at the 25% probability level. Atom O3′ has been omitted. H atoms are shown as small spheres.

oside. The molecular dimensions in (I) agree well with the corresponding dimensions reported in the structures quoted above.

The crystal structure of (I) is stabilized by weak inter- and intramolecular C—H···O hydrogen bonds; details are provided in Table 2.

Experimental

To a mixture of 1,2,3,4,6-penta-*O*-acetyl-*β*-D-glucopyranose (0.75 g, 1.92 mmol), *p*-*tert*-butylphenol (0.57 g, 3.8 mmol) and silver perchlorate (0.19 g, 0.096 mmol) in dry acetonitrile (20 ml) in a flask containing molecular sieves, trifluoromethylsilyl trifluoromethanesulfonate (TMSOTf; 0.17 ml, 0.096 mmol) was added dropwise at 273 K in an inert atmosphere. The reaction mixture was warmed slowly to room temperature. After stirring for 2–3 h, the reaction was monitored by thin-layer chromatography, and when no unreacted sugar was found the mixture was quenched with triethylamine at 273 K. A light-blue precipitate was obtained, which disappeared on addition of water. After filtration, the mixture was extracted with ethyl acetate and washed with brine. The organic layer was dried over sodium sulfate and concentrated *in vacuo*. A crude mixture of two anomers was obtained in the form of a light-yellow oil, which was separated through preparative TLC on pre-coated silica-gel plates (0.25 mm E; Merck). The *β*-anomer was obtained in 45% yield (m.p. 411–0413 K). EIMS: 480.

Crystal data

$C_{24}H_{32}O_{10}$
M_r = 480.50
Orthorhombic, $P2_12_12_1$
a = 8.093 (2) Å
b = 10.932 (4) Å
c = 29.318 (11) Å
V = 2593.8 (15) Å^3
Z = 4
D_x = 1.230 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
Block, colourless
0.14 × 0.12 × 0.08 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.987, T_{max} = 0.992
5849 measured reflections
3374 independent reflections
2317 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.128
S = 1.02
3374 reflections
318 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.45P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.012 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.423 (3)	O6—C11	1.357 (4)
O1—C5	1.425 (4)	O6—C3	1.448 (3)
O2—C7	1.327 (4)	O7—C11	1.200 (4)
O2—C6	1.442 (4)	O8—C13	1.354 (4)
O3—C7	1.238 (14)	O8—C4	1.437 (4)
O3′—C7	1.222 (16)	O9—C13	1.197 (4)
O4—C9	1.362 (4)	O10—C15	1.383 (4)
O4—C2	1.445 (3)	O10—C5	1.394 (4)
O5—C9	1.192 (4)		
C1—O1—C5	112.0 (2)	C11—O6—C3	118.8 (2)
C7—O2—C6	117.6 (3)	C13—O8—C4	117.6 (2)
C9—O4—C2	118.3 (2)	C15—O10—C5	118.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2	0.96	2.54	2.932 (4)	104
C2—H2···O5	0.96	2.30	2.702 (4)	104
C4—H4···O9	0.96	2.30	2.697 (4)	104
C10—H10B···O3^{i}	0.98	2.36	3.263 (16)	154
C10—H10B···O3′^{i}	0.98	2.42	3.241 (17)	142
C12—H12B···O5^{i}	0.98	2.38	3.358 (5)	173
C16—H16···O1	0.96	2.55	3.063 (4)	114
C16—H16···O9^{ii}	0.96	2.52	3.368 (4)	147
C24—H24C···O10^{iii}	0.98	2.49	3.303 (5)	140

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{5}{2}, -z$; (ii) $x+1, y, z$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Due to the absence of significant anomalous dispersion effects, the absolute configuration was not determined in this analysis and Friedel pairs were merged. The absolute configuration presented in this paper has been assigned by reference to the chiral centre in the starting material. One of the carbonyl O atoms is disordered over two sites, O3 and O3′, with equal site-occupancy factors. The H atoms were located in difference Fourier syntheses and were included in the refinement in geometrically idealized positions, with C—H = 0.95 and 0.98 Å and U_{iso} = 1.5 (methyl H) or 1.2 (other H) times U_{eq} of the atoms to which they were bonded.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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