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2-Phenyl-6-(*p*-tolylsulfanyl)-4,4a,6,7,8,8a-hexahydro-2*H*-pyrano[3,2-*d*][1,3]dioxane-7,8-diyl diacetate

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Key indicators

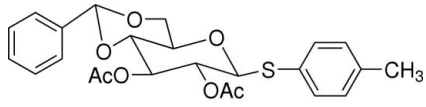
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
R factor = 0.088
wR factor = 0.163
Data-to-parameter ratio = 11.8

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

The molecule of the title compound, $C_{24}H_{26}O_7S$, (I), is an important building block in the synthesis of oligosaccharides. Non-classical C—H···O hydrogen bonds link the molecules in the crystal structure into infinite sheets.

Comment

Carbohydrates often exist on cell surfaces as glycoprotein or glycolipid conjugates, and play important structural and functional roles in numerous biological recognition processes. The synthesis of saccharides has attracted much more interest in recent years. As part of a program to construct a phenylpropanoid glycoside library, our group has synthesized several monosaccharide building blocks. We report here the crystal structure of one of them, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. In the crystal structure, non-classical C—H···O hydrogen bonds link the molecules into infinite sheets (Fig. 2).

Experimental

To a vigorously stirred solution of *p*-tolylsulfanyl-β-D-glucopyranoside (0.286 g, 1 mmol) in acetonitrile (20 ml) were added dropwise

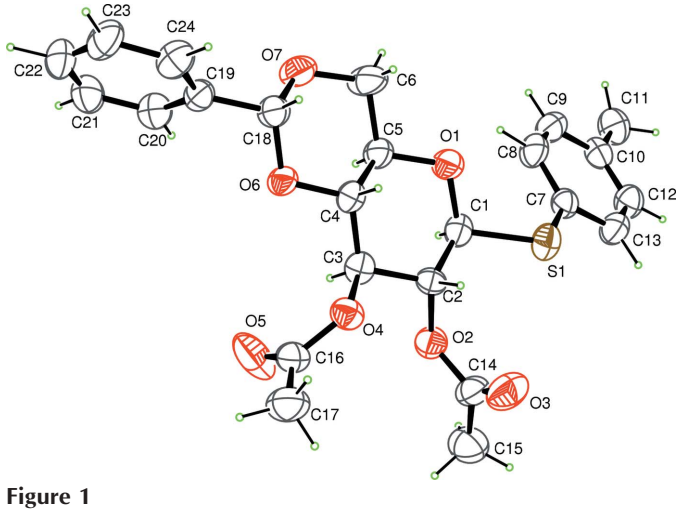


Figure 1
The molecular structure of compound (I). Displacement ellipsoids are drawn at the 40% probability level.

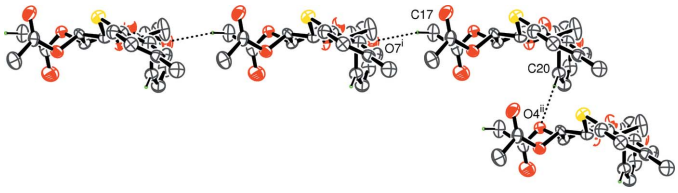


Figure 2
Part of the packing arrangement for (I). Dashed lines indicate hydrogen-bonding interactions. H atoms not involved in hydrogen bonds have been omitted. [Symmetry codes: (i) $-1+x, -1+y, z$; (ii) $1+x, y, z$.]

benzaldehyde dimethyl acetal (0.30 ml, 2 mmol) and *p*-tolylsulfonic acid (0.018 g, 0.1 mmol) in turn at room temperature. The reaction solution was stirred for 30 min at this temperature and concentrated. The resulting mixture was dissolved in CH_2Cl_2 (20 ml), washed with saturated $NaHCO_3$ and water, then dried over anhydrous Na_2SO_4 and concentrated. The crude residue was dissolved in pyridine (10 ml) and acetic anhydride (7.5 ml) and stirred overnight at room temperature. The resulting mixture was concentrated, dissolved in CH_2Cl_2 (20 ml), washed with saturated aqueous $NaHCO_3$ and water. The organic phase was dried over Na_2SO_4. After removal of the solvent *in vacuo*, the solid was purified by silica-gel column chromatography (hexane/ethyl acetate 4:1) to give the desired product (yield 83% over two steps). Colorless crystals were obtained from an acetone solution after allowing it to stand for 4 d. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 (*m*, 8H), 7.16 (*d*, J = 8.0 Hz, 2H), 5.50 (*s*, 1H), 5.34 (*t*, J = 9.5 Hz, 1H), 4.99 (*t*, J = 9.0 Hz, 1H), 4.75 (*d*, J = 10.0 Hz, 1H), 4.39 (*m*, 1H), 3.79 (*t*, J = 10.0 Hz, 1H), 3.65 (*t*, J = 9.5 Hz, 1H), 3.57 (*m*, 1H), 2.37 (*s*, 3H), 2.12 (*s*, 3H), 2.04 (*s*, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.3, 169.7, 139.0, 137.0, 133.9, 130.0, 129.4, 128.5, 127.9, 126.4, 101.7, 87.0, 78.3, 73.2, 71.0, 70.9, 68.7, 21.4, 21.0. MS (EI): m/z = 481 $[M + Na]^+$.

Crystal data

$C_{24}H_{26}O_7S$
M_r = 458.53
Monoclinic, $P2_1$
a = 5.6530 (7) Å
b = 8.0292 (9) Å
c = 25.490 (3) Å
β = 90.906 (5)°
V = 1156.8 (2) Å^3
Z = 2
D_x = 1.316 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8131 reflections
θ = 2.5–27.3°
μ = 0.18 mm^{-1}
T = 295 (1) K
Block, colorless
0.31 × 0.19 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.893, T_{max} = 0.975
8608 measured reflections
4688 independent reflections
3140 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.097
θ_{max} = 27.3°
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.163
S = 1.00
4583 reflections
390 parameters
H-atom parameters constrained
$w = 1/[0.0006F_o^2 + 3\sigma(F_o^2) + 0.5]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: Flack (1983), 1922 Friedel pairs
Flack parameter: 0.04 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C17—H19···O7^{i}	0.96	2.54	3.487 (8)	168
C20—H22···O4^{ii}	0.98	2.59	3.550 (6)	166

Symmetry codes: (i) $x-1, y-1, z$; (ii) $x+1, y, z$.

All H atoms were placed in calculated positions, with C—H = 0.96–0.98 Å, and were refined as riding on their carrier atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku,1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 1996); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

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