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4-Methoxyphenyl 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranoside

Yan Zhang, Gui-Long Zhao, Xi-Zhao Wang, Jiong Jia and Jian-Wu Wang*

School of Chemistry and Chemical Engineering, Shandong University, Jinan 250100, People's Republic of China

Correspondence e-mail: yugp2005@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.043
wR factor = 0.105
Data-to-parameter ratio = 7.7

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

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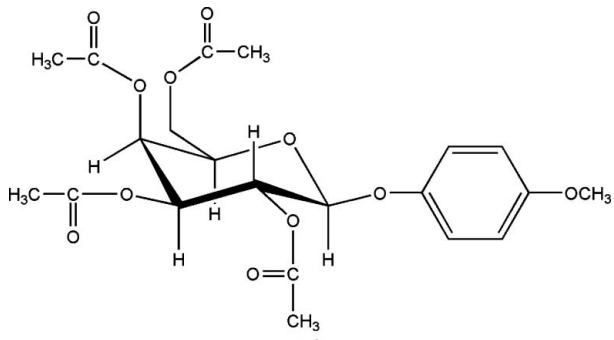
The six-membered sugar ring of the title compound, $C_{21}H_{26}O_{11}$, adopts a chair conformation. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds and van der Waals forces.

Comment

4-Methoxyphenyl 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranoside, (I), is an important intermediate in the synthesis of oligosaccharides (Chen *et al.*, 2002; Polat & Linhardt, 2003). Its crystal structure is reported here.



(I)

The absolute configuration of the title compound was assigned from a knowledge of the stereochemistry of its synthetic precursor. Bond lengths of the ring are normal (Table 1) and comparable with those in published structures (Abboud *et al.*, 1997; Temeriusz *et al.*, 2005). The ring adopts a $^3C_5$ chair conformation, with atoms C3 and O1 at distances of 0.55 (4) and 0.70 (4)Å, respectively, on opposite sides of the C1/C2/C4/C5 plane. It can be seen that the C—H of the ring and C═O bonds point in the same direction and are approximately coplanar.

The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds and van der Waals forces (Table 2).

Experimental

A mixture of tetra-*O*-acetyl-α-D-galactopyranosyl bromide (5 mmol) in chloroform (10 ml) and the sodium salt of 4-methoxyphenol (5 mmol) in water was stirred overnight. The organic layer was then separated, washed with water, and dried with anhydrous sodium sulfate. Evaporation on a rotary evaporator gave a residue, which was recrystallized from absolute ethanol to afford the pure title compound as colourless prisms (85%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution at room temperature.

Crystal data

$C_{21}H_{26}O_{11}$
M_r = 454.42
Monoclinic, $P2_1$
a = 11.935 (3) Å
b = 5.7061 (14) Å
c = 17.873 (4) Å
β = 108.884 (3)°
V = 1151.7 (5) Å^3
Z = 2
D_x = 1.310 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
Plate, colourless
0.40 × 0.18 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.959, T_{max} = 0.995
6093 measured reflections
2261 independent reflections
1837 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.105
S = 1.06
2261 reflections
292 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.1697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.412 (4)	O8—C13	1.346 (5)
O1—C5	1.431 (4)	O8—C2	1.434 (4)
O2—C7	1.335 (5)	O9—C13	1.181 (5)
O2—C6	1.441 (4)	O10—C1	1.391 (4)
O3—C7	1.193 (5)	O10—C15	1.391 (4)
O4—C9	1.357 (4)	O11—C18	1.375 (5)
O4—C4	1.440 (4)	O11—C21	1.402 (7)
O5—C9	1.175 (5)	C1—C2	1.514 (5)
O6—C11	1.342 (5)	C2—C3	1.517 (5)
O6—C3	1.442 (4)	C3—C4	1.523 (5)
O7—C11	1.206 (6)	C4—C5	1.526 (5)
C1—O1—C5	111.0 (2)	C13—O8—C2	118.9 (3)
C7—O2—C6	116.6 (3)	C1—O10—C15	118.4 (3)
C9—O4—C4	116.9 (3)	C18—O11—C21	118.6 (4)
C11—O6—C3	118.0 (3)		
C5—O1—C1—C2	65.8 (4)	C11—O6—C3—C4	−71.6 (4)
C13—O8—C2—C1	−117.8 (3)	C9—O4—C4—C3	136.8 (3)
C13—O8—C2—C3	120.6 (3)	C9—O4—C4—C5	−103.8 (4)
C11—O6—C3—C2	165.0 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O9	0.98	2.29	2.713 (5)	105
C4—H4A···O5	0.98	2.27	2.664 (4)	103
C4—H4A···O7	0.98	2.58	3.044 (5)	109
C6—H6B···O3	0.97	2.29	2.666 (5)	102
C16—H16A···O1	0.93	2.48	2.987 (4)	115
C3—H3A···O9^{i}	0.98	2.52	3.364 (5)	145
C10—H10A···O7^{ii}	0.96	2.59	3.479 (5)	154
C10—H10C···O3^{iii}	0.96	2.54	3.370 (5)	145
C21—H21C···O7^{iv}	0.96	2.47	3.356 (6)	153

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

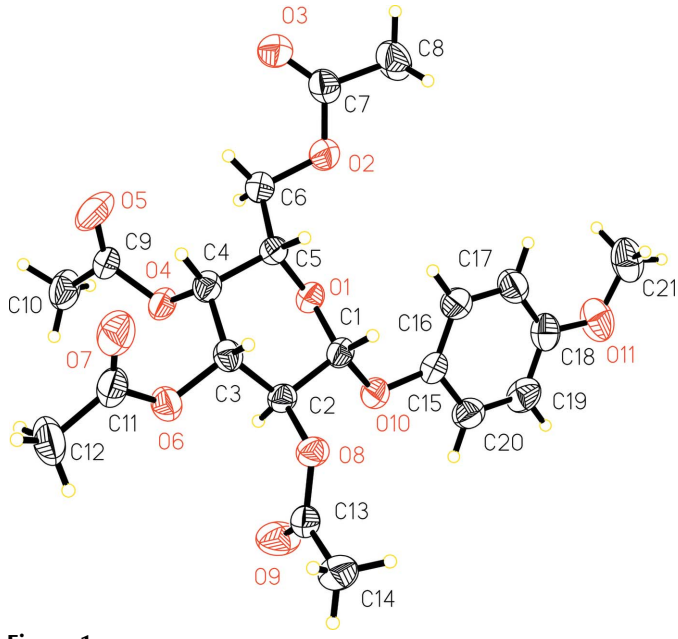


Figure 1
A view of compound (I), with displacement ellipsoids drawn at the 40% probability level.

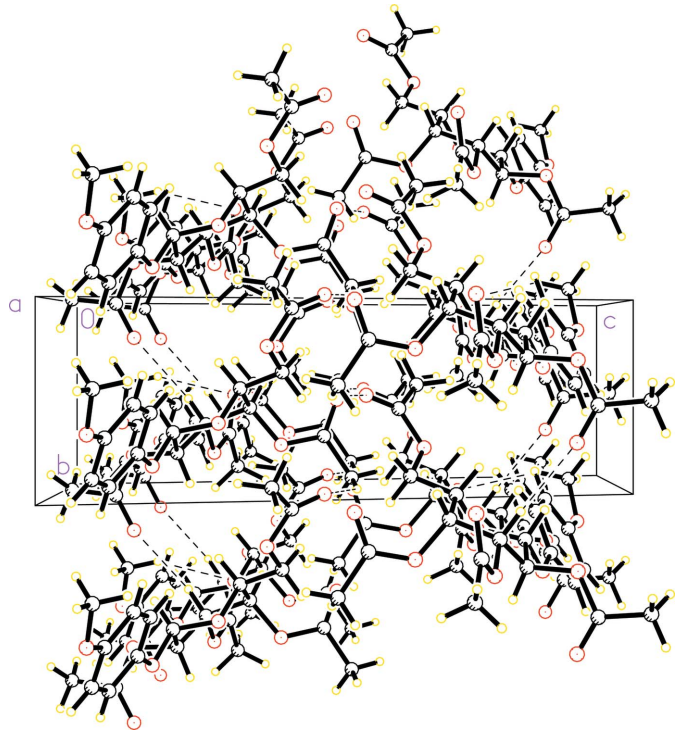


Figure 2
A packing diagram, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

All H atoms were placed in calculated positions, with C—H = 0.93–0.98Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or1.5U_{eq}(methyl C). In the absence of significant anomalous dispersion effects, Friedel pairs were merged and the absolute configuration was assumed from the synthesis.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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