Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Acetoxymethyl-2-(2-methoxyphenoxy)-tetrahydropyran-3,4,5-triyl triacetate

Feng-Yan Zhou[a,b] and Jian-Hua Zhong[a]*

[a]Department of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of China, and [b]Department of Chemistry, Zaozhuang University, Zaozhuang 277160, People's Republic of China

Correspondence e-mail: zhoufy1968@163.com

Received 4 July 2005
Accepted 18 July 2005
Online 23 July 2005

Key indicators

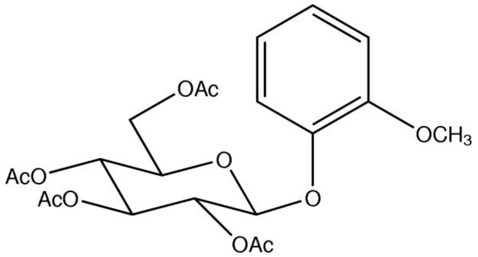
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.118
Data-to-parameter ratio = 10.0

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, $C_{21}H_{26}O_{11}$, possesses normal geometric parameters. Intermolecular C—H···O hydrogen bonds link adjacent molecules to form one-dimensional chains along the crystallographic *a* axis.

Comment

Carbohydrates often exist on cell surfaces as glycoprotein or glycolipid conjugates, and play important structural and functional roles in numerous biological recognition processes (Ernst *et al.*, 2000). Many drugs also incorporate carbohydrates in order to reduce toxicity (Jung *et al.*, 2000; Seeberger & Haase, 2000; Sears & Wong, 2001). In order to find new compounds with high biological activity, our group has synthesized several glucosides. We report here the crystal structure of one of them, the title compound, (I).



(I)

Fig. 1 shows the structure of (I). Selected geometric parameters and the hydrogen-bond parameters are listed in Tables

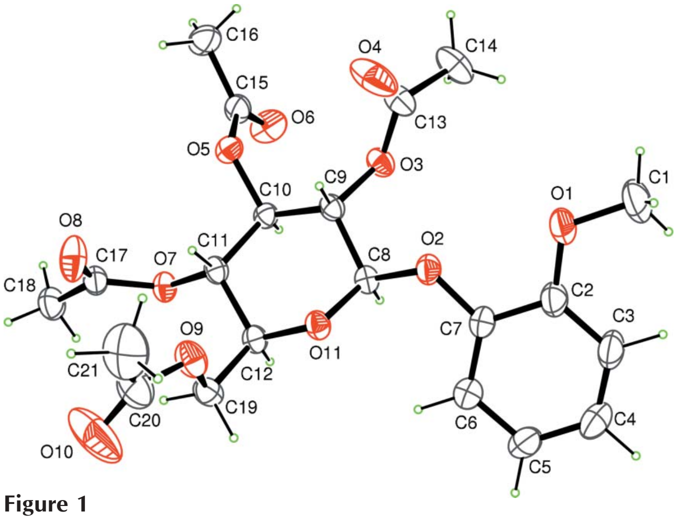


Figure 1
The molecule of compound (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

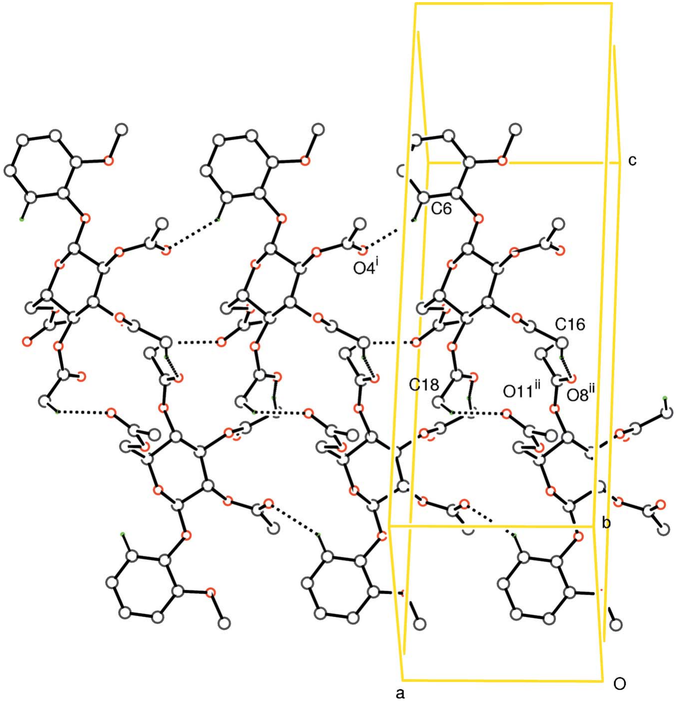


Figure 2
The molecular packing of (I). Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonding have been omitted (see Table 2 for symmetry codes).

1 and 2, respectively. The compound crystallizes in the orthorhombic space group $P2_12_12_1$, with one molecule in the asymmetric unit. In the crystal structure, non-classical C—H⋯O hydrogen bonds play an important role in the formation of polymeric chains running along the crystallographic *a* axis (Fig. 2).

Experimental

A solution of 2-methylphenol (1.08 g, 10 mmol) in $CHCl_3$ (20 ml) and water (15 ml) was stirred at 328 K for 30 min. A solution of NaOH (0.5 g, 12.5 mmol) in water (15 ml) and 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyronoside bromide (4.3 g, 10.5 mmol) in $CHCl_3$ (15 ml) were added dropwise to the reaction solution synchronously, maintaining pH = 8–9 and a reaction temperature of 328 K. The reaction was continued for another 2 h and then cooled. The organic phase was separated, washed with aqueous NaOH (5%) and water, and dried with anhydrous $MgSO_4$. 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 71%). Colorless crystals were obtained from ethyl acetate solution after allowing it to stand for 6 d. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.12 (*t*, *J* = 6.0 Hz, 1H), 7.07 (*dd*, J_1 = 0.5 Hz, J_2 = 6.5 Hz, 1H), 6.89 (*m*, 2H), 5.28 (*m*, 2H), 5.17 (*dd*, J_1 = 3.5 Hz, J_2 = 6.0 Hz, 1H), 4.96 (*dd*, J_1 = 2.0 Hz, J_2 = 5.0 Hz, 1H), 4.28 (*m*, 1H), 4.16 (*m*, 1H), 3.81 (*d*, *J* = 6.0 Hz, 3H), 3.76 (*m*, 1H), 2.08 (*s*, 3H), 2.07 (*s*, 3H), 2.04 (*s*, 3H), 2.03 (*s*, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.7, 170.4, 169.6, 169.5, 150.8, 146.3, 124.9, 121.0, 120.4, 112.9, 101.0, 72.8, 72.1, 71.4, 68.6, 62.1, 56.1, 20.82, 20.77, 20.72. HRMS calculated for $C_{21}H_{26}O_{11}Na$ (*M* + Na): 477.1373; found: 477.1383.

Crystal data

$C_{21}H_{26}O_{11}$
M_r = 454.43
Orthorhombic, $P2_12_12_1$
a = 8.1321 (2) Å
b = 10.6922 (3) Å
c = 26.8033 (7) Å
V = 2330.5 (1) Å^3
Z = 4
D_x = 1.295 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 7872 reflections
θ = 2.0–27.5°
μ = 0.11 mm^{-1}
T = 295 (1) K
Block, colorless
0.45 × 0.40 × 0.35 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.928, T_{max} = 0.964
16634 measured reflections
2893 independent reflections
2377 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.032
θ_{max} = 27.5°
h = −10 → 10
k = −13 → 12
l = −34 → 34

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.118
S = 1.01
2893 reflections
290 parameters
H-atom parameters constrained
$w = 1/[0.0019F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 4.0 (7) × 10^2

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.410 (8)	O3—C13	1.345 (3)
O1—C2	1.358 (4)	O4—C13	1.183 (4)
O2—C7	1.383 (3)	O12—C8	1.409 (3)
O2—C8	1.396 (3)	O12—C12	1.433 (3)
O3—C9	1.427 (3)		
C2—O1—C1	116.0 (3)	C13—O3—C9	117.9 (2)
C8—O2—C7	117.6 (2)	C12—O12—C8	112.6 (2)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C6—H6⋯O4^i	0.98	2.53	3.338 (4)	140
C16—H163⋯O8^{ii}	0.96	2.40	3.323 (4)	160
C18—H181⋯O11^{ii}	0.96	2.36	3.159 (5)	140

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

The absolute stereochemistry could not be established from the diffraction experiment, because of the lack of significant anomalous dispersion effects; Friedel pairs in the data set were merged. The absolute configuration was, therefore, chosen on the basis of the known configuration of the synthetic precursor. Atoms H222 and H555 were found in a difference Fourier map and fixed in position. The methyl H atoms were constrained to an ideal geometry [C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$] and were allowed to rotate freely about the C—C bonds. The other H atoms were placed in calculated positions (C—H = 0.97 and 0.98 Å), with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atoms, and included in the final cycles of refinement in the riding-model approximation.

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* (Better-

o2702 Zhou *et al.* • $C_{21}H_{26}O_{11}$ *Acta Cryst.* (2005). E**61**, o2701–o2703

idge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

We thank the National Natural Science Foundation of China (No. 20272051) as well as the Teaching and Research Award Program for Outstanding Young Teachers in Higher Education Institutions of MOE, People's Republic of China.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Ernst, B., Hart, G. W. & Sinày, P. (2000). Editors. *Carbohydrates in Chemistry and Biology*, Vols. 1–4. Weiheim: Wiley-VCH.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Jung, K. H., Müller, M. & Schmidt, R. R. (2000). *Chem. Rev.* **100**, 4423–4442.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Rigaku (1998). *PROCESS-AUTO*. Version 1.06. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Sears, P. & Wong, C. H. (2001). *Science*, **291**, 2344–2350.

Seeberger, P. H. & Haase, W. C. (2000). *Chem. Rev.* **100**, 4349–4394.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Acta Cryst. (2005). E**61**, o2701–o2703 Zhou *et al.* • $C_{21}H_{26}O_{11}$ **o2703**