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2-Bromoethyl 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranoside

Ivan Halasz,[a]* Renata Odžak,[a] Srdanka Tomić[a] and Dubravka Matković-Čalogović[b]

[a]Laboratory of Organic Chemistry, Department of Chemistry, Faculty of Science, University of Zagreb, Strossmayerov trg 14, 10000 Zagreb, Croatia, and [b]Laboratory of General and Inorganic Chemistry, Department of Chemistry, Faculty of Science, University of Zagreb, Zvonimirova 8, 10002 Zagreb, Croatia

Correspondence e-mail: ihalasz@chem.pmf.hr

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.047
wR factor = 0.142
Data-to-parameter ratio = 15.6

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

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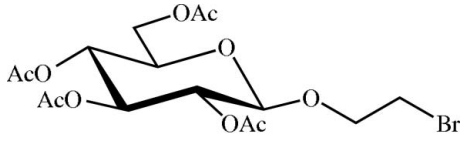
The six-membered ring of the title compound, $C_{16}H_{23}BrO_{10}$, adopts a chair conformation. The molecules are interconnected by weak C—H···O hydrogen bonds into a three-dimensional network.

Comment

The synthesis of oligosaccharides has played an important role in the development of carbohydrate chemistry. Today, many different procedures are known, and these are constantly being improved and further developed (Lindhorst, 2003). Neighbouring-group participation has frequently been used in organic synthesis. In carbohydrate chemistry, it is generally thought that glycosyl donors possessing an *O*-acyl group with a participating function at C2 give exclusively the corresponding 1,2-*trans*-glycoside with quite high stereoselectivity in glycosylation reactions. In our investigations of monosaccharides, the OAc group was used as the protective group for further glycosylation reactions. The 1-OAc group in the glycosyl donor was transformed to a good leaving group using activated $BF_3{\cdot}Et_2O$. Thus, we synthesized the title compound, (I), and determined its crystal structure.



(I)

The six-membered ring of (I) adopts a chair conformation (Fig. 1). The absolute configuration, known from the synthetic pathway, is fully confirmed by refinement of the Flack parameter (Flack & Bernardinelli, 2000).

Since all its hydroxyl groups are either acylated or alkylated, compound (I) has many possible hydrogen-bond acceptors and, at the same time, no hydrogen-bond donors other than C—H groups. A number of possible weak hydrogen bonds can be found, but only those which satisfy the usual geometrical conditions are listed in Table 1. Six C—H···O hydrogen bonds are intermolecular and form a three-dimensional network. Among these, four link the molecules along the *b* axis, whereas the other two link the molecules along the *a* and *c* axes. It is interesting to note that only acetyl O atoms and the anomeric O atom are involved in hydrogen bonding in (I), even though, in general, the ester O atom is known to be involved in hydrogen bonding in small organic molecules (Molčanov *et al.*, 2004). The reason for this could be steric hindrance of the ester O atoms in the crystal structure of (I).

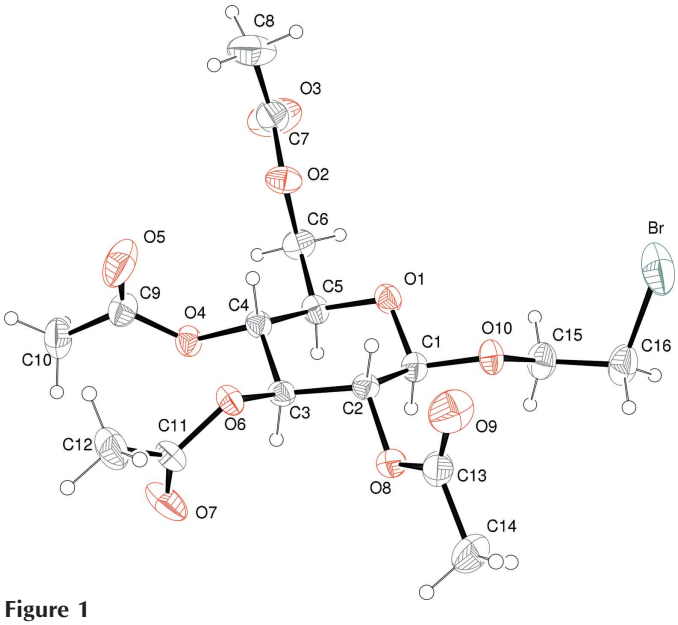


Figure 1
A view of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitary radii.

The packing of compound (I) is different from that observed in (2-ethyl)-2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranoside [Cambridge Structural Database (Allen, 2002; version 5.26) refcode ZETNOB (Prawat *et al.*, 1995)]. The Br atom itself forms no contacts other than those of a van der Waals nature. It can be speculated that the differences in packing arise from the larger radius of the Br atom and its effect on the H-atom donor properties of the methylene group to which it is bonded. The C—H group of this methylene group is involved in the shortest hydrogen bond in the crystal structure of (I), whereas in ZETNOB it forms no hydrogen bonds.

Experimental

The title compound was synthesized according to the method of Dahmén *et al.* (1983). ^{1}H and ^{13}C NMR spectra are in agreement with data presented by Lindhorst (2003).

Crystal data

$C_{16}H_{23}BrO_{10}$
M_r = 455.25
Monoclinic, $P2_1$
a = 9.4153 (12) Å
b = 9.8326 (14) Å
c = 11.2069 (19) Å
β = 96.391 (12)°
V = 1031.1 (3) Å^3
Z = 2
D_x = 1.466 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2233 reflections
θ = 2.8–18.3°
μ = 2.04 mm^{-1}
T = 295 (2) K
Prism, colourless
0.45 × 0.10 × 0.08 mm

Data collection

Oxford Xcalibur 3 CCD area-detector diffractometer
ω scans
Absorption correction: analytical (Alcock, 1970)
T_{min} = 0.686, T_{max} = 0.871
13961 measured reflections
3873 independent reflections
3044 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 26.1°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.142
S = 1.03
3873 reflections
249 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0929P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Absolute structure: Flack & Bernardinelli (2000), with 1722 Friedel pairs
Flack parameter = 0.005 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O5^{i}	0.98	2.55	3.479 (6)	157
C6—H6B···O9^{i}	0.97	2.73	3.697 (6)	173
C10—H10B···O9^{i}	0.96	2.63	3.497 (8)	154
C8—H8A···O7^{ii}	0.96	2.60	3.459 (8)	149
C12—H12C···O1^{iii}	0.96	2.66	3.581 (6)	158
C16—H16B···O3^{iv}	0.97	2.45	3.367 (7)	157

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

Methine and methylene H atoms were positioned geometrically, with C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for methine H, and C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms. Methyl H atoms were refined using a riding model, with C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C).

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2003); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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