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2,3,4,6-Tetra-*O*-acetyl-2-chloroethyl-β-D-galactopyranoside

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.141
Data-to-parameter ratio = 15.7

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

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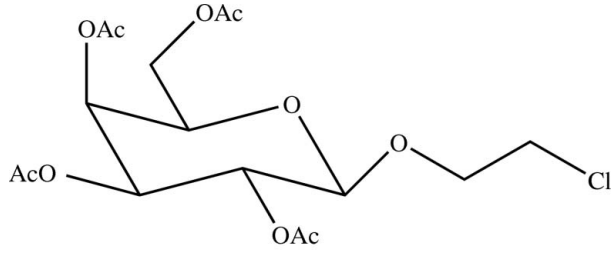
In the title compound, $C_{16}H_{23}ClO_{10}$, the galactopyranoside ring adopts the $^4C_1$ chair conformation with the acetyl group at C4 positioned axially and all other substituents equatorially. In the crystal structure, molecules are linked into chains along the *b* axis by C—H···O interactions.

Comment

Mammalian hepatocytes (parenchymal cells) have a large number of asialoglycoprotein receptors (ASGPr) that can bind desialylated proteins from the serum and internalize them into a cell interior. The ASGPr can recognize terminal β-D-galactose or *N*-acetylgalactosamine residues, which makes it a particularly attractive target in many drug carrier studies (Wang *et al.*, 2006). In an attempt to further study the interaction between glycolipids and the receptor (Sliedregt *et al.*, 1999), a new galactopyranoside, 2,3,4,6-tetra-*O*-acetyl-2-chloroethyl-β-D-galactopyranoside, (I), was synthesized and its structure determined using single-crystal X-ray methods.



(I)

All bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The galactopyranoside ring adopts a chair conformation. The (−)-synclinal conformation for the atom sequence O10—C15—C16—Cl1 [−69.8 (4)°] is observed. There exist three intramolecular hydrogen bonds (Table 1), forming three five-membered rings. These interactions contribute to the planarity of each substituent. In the crystal structure, molecules are linked into chains along the *b* axis by C5—H5*A*···O5^{i} interactions (Fig. 2 and Table 1). A rather weak hydrogen bond, C12—H12*A*···O2^{ii}, is also present.

Experimental

β-D-Galactose pentaacetate (5.0 g, 12.8 mmol) was dissolved in 25 ml of anhydrous CH_2Cl_2 and 2-chloroethanol (1.5 ml, 22.3 mmol) was added with a syringe. The resulting solution was stirred under argon and cooled to 273 K. $BF_3{\cdot}Et_2O$ (2.1 ml, 16.7 mmol) was then added dropwise. The reaction was stirred for 1 h at 273 K, continuing overnight at room temperature. After being diluted with 50 ml

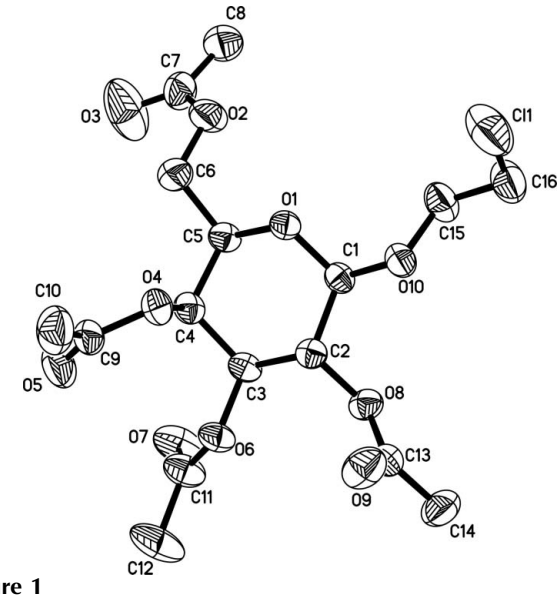


Figure 1
The molecular structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

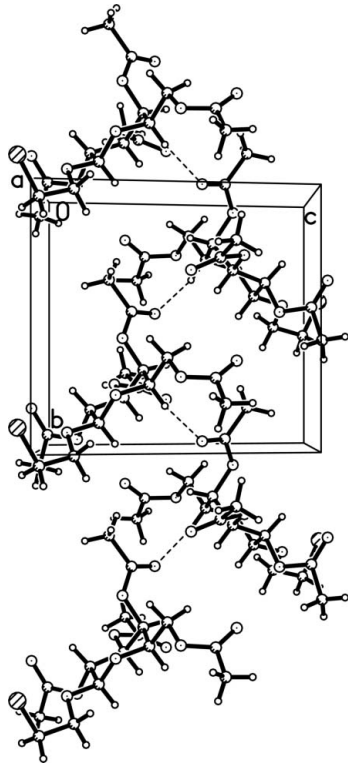


Figure 2
Packing diagram of (I), showing C—H···O hydrogen bonds (dashed lines) forming a chain along the *b* axis.

CH_2Cl_2, cold water and saturated aqueous $NaHCO_3$ were added. The resulting solution was allowed to stand over anhydrous Na_2SO_4. By evaporation of the solvent a crude white solid was obtained. After chromatography on a silica-gel column using an ethyl acetate-petroleum ether mixture (4:7 *v*/*v*) as eluant, pure (I) was obtained. Colourless single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of an ethyl actate solution of (I).

Crystal data

$C_{16}H_{23}ClO_{10}$
M_r = 410.79
Monoclinic, $P2_1$
a = 10.554 (2) Å
b = 9.708 (2) Å
c = 10.599 (3) Å
β = 103.299 (3)°
V = 1056.8 (4) Å^3
Z = 2
D_x = 1.291 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 (2) K
Block, colourless
0.44 × 0.28 × 0.19 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.907, T_{max} = 0.958
5996 measured reflections
3826 independent reflections
3238 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 26.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.141
S = 1.04
3826 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0789P)^2 + 0.1466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 1608 Friedel pairs
Flack parameter: 0.09 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O9	0.98	2.27	2.681 (4)	104
C4—H4A···O5	0.98	2.28	2.662 (4)	102
C5—H5A···O5^{i}	0.98	2.35	3.226 (4)	149
C6—H6A···O3	0.97	2.24	2.636 (5)	103
C12—H12A···O2^{ii}	0.96	2.56	3.510 (5)	172

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+3$; (ii) $x-1, y, z$.

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H = 0.96–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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