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(5-Methoxycarbonyl)pentyl α-D-manno-pyranoside

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.084
Data-to-parameter ratio = 6.5

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

In the crystalline state, the title compound, $C_{13}H_{24}O_8$, forms hydrogen-bonded carbohydrate bilayers with interdigitated hydrophobically interacting alkyl chains.

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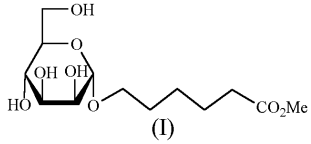
Comment

The title compound, (I), was prepared as part of a project aimed at the synthesis of potential ligands for mannose-specific binding proteins (Furneaux *et al.*, 2002).



(I)

The crystal structure of (I) contains one independent molecule of the title compound per asymmetric unit (Fig. 1). The molecules are bound into a three-dimensional lattice with mannopyranoside hydrogen-bonded bilayers (generated by the screw axis along the *b* axis) normal to the *c* axis, separated by interdigitated hydrophobically interacting alkyl chains (C7–C13; Fig. 2). This structural motif has been observed before, and is commonly associated with carbohydrate derivatives having long-chain alkyl substituents that form liquid-crystal phases (Abe *et al.*, 2000). Binding the sugar rings together are four classic hydrogen bonds which utilize all four hydroxy H atoms (H···O = 1.94–2.25 Å and O—H···O = 153–179°) (Desiraju & Steiner, 1999). The acceptor atoms are O3 (with bonds from H atoms on O2 and O6 of different molecules), and O5 and O6 (with bonds from H atoms on O3 and O4 from another adjacent molecule, respectively). The six-membered ring (O5/C1–C5) adopts the expected regular $^4C_1$

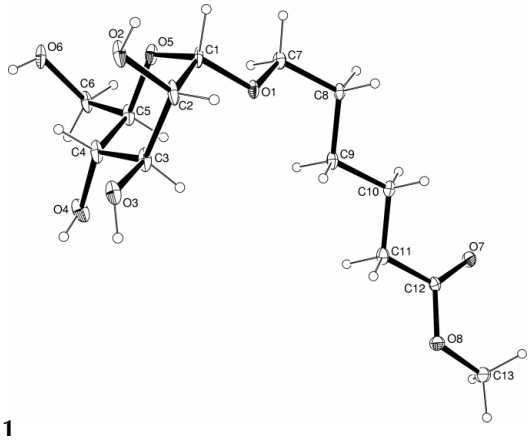


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as circles of arbitrary radii.

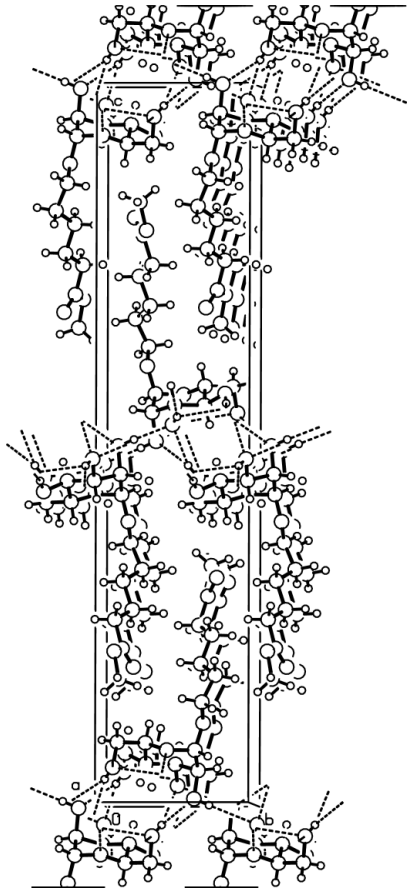


Figure 2
The packing of (I), viewed down the *a* axis, with the *c* axis vertical (Spek, 2003). Hydrogen bonds are shown as dashed lines.

chair conformation, with Q, θ and φ values of 0.548 (4) Å, 3.0 (4) and 276 (8)°, respectively (Cremer & Pople, 1975), and the substituted alkyl aglycon adopts a zigzag orientation.

There are at least 45 α-D-mannopyranoside crystal structures reported in the literature [*CONQUEST* (Bruno *et al.*, 2002); CSD version January 2004 (Allen, 2002)] but few involve compounds with alkyl aglycons. However, there has been some interest in long-chain alkyl 1-thio-α-D-mannopyranosides (Carter *et al.*, 1982; Miethchen & Hein, 2000) and alkyl α-D- and β-D-glucopyranosides (Adasch *et al.*, 1998; Hoffmann *et al.*, 2000), all of which display a liquid-crystal phase, with similar structural motifs to that reported here. Some relevant *O*-α-D-mannopyranosides which have been crystallographically analysed are the *O*-α-mannopyranosyl-(1 → 3)-L-threonine (Darbon *et al.*, 1984), *p*-nitrophenyl α-D-mannopyranoside (Fernandez-Castaño & Foces-Foces, 1996; Agianian *et al.*, 1997) and methyl 2-*O*-α-D-mannopyranosyl-α-D-mannopyranoside (Srikrishnan *et al.*, 1989). In all cases, the sugar rings adopt the $^4C_1$ conformation.

Experimental

The title compound was prepared as described previously (Furneaux *et al.*, 2002). Subsequently it was recrystallized from ethyl acetate.

Crystal data

$C_{13}H_{24}O_8$
M_r = 308.32
Orthorhombic, $P2_12_12_1$
a = 6.1389 (2) Å
b = 7.2316 (2) Å
c = 33.9218 (14) Å
V = 1505.93 (9) Å^3
Z = 4
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 969 reflections
θ = 1.0–23.8°
μ = 0.11 mm^{-1}
T = 293 (2) K
Poor quality, needle, colourless
1.0 × 0.2 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
4096 measured reflections
1283 independent reflections
1112 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 23.8°
$h = -6 \rightarrow 6$
$k = -8 \rightarrow 8$
$l = -37 \rightarrow 38$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.084
S = 1.12
1283 reflections
196 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 0.3006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.402 (4)	O8—C12	1.344 (4)
O1—C1—C2	108.1 (3)	O5—C1—C2	111.9 (3)
C7—O1—C1—C2	−178.2 (2)	C10—C11—C12—O8	157.1 (3)

All H atoms were geometrically constrained to ride on their parent atom (C—H = 0.96–0.98 Å and O—H = 0.82 Å), with U_{iso} values 1.5 and 1.2 times the U_{eq} values of the parent atoms O/C13 and the remaining C atoms, respectively. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged, and the absolute configuration cannot be determined from the crystallographic experiment; it was assumed from the synthesis.

Data collection: *KappaCCD Software* (Nonius, 1997); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; 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 and *PLATON* (Spek, 2003).

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