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D-Mannitol-1,2,6-tribenzoate

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.087
Data-to-parameter ratio = 10.2

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

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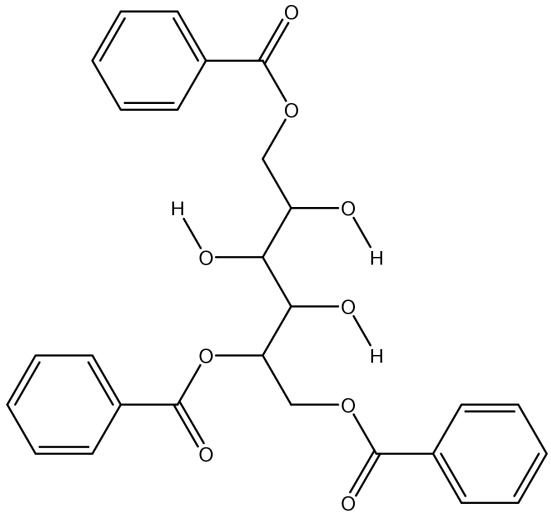
D-Mannitol-1,2,6-tribenzoate, $C_{27}H_{26}O_9$, was obtained as a side product from the reaction of D-mannitol with benzoyl chloride in hot pyridine. The major product of the reaction was D-mannitol-1,6-dibenzoate.

Comment

The application of boronic acids as labile protective agents in the selective functionalization of polyols is currently one of the focus areas of our research (Bhaskar *et al.*, 2001). The title compound, (I), was obtained from a control experiment, which was performed in order to determine the yield and selectivity of the reaction of D-mannitol with benzoyl chloride in the absence of a boronic acid. It is interesting to note that the third acylation of D-mannitol occurs at 2-OH, rather than at 3-OH.



(I)

Experimental

D-Mannitol (20.03 g, 0.11 mol) was treated with benzoyl chloride (12.30 ml, 0.11 mol) in hot pyridine (75 ml), following the procedure of Morpain & Tisserand (1979). After the reaction mixture was cooled, a white precipitate was obtained, which was recrystallized from hot methanol to afford pure D-mannitol-1,6-dibenzoate (4.29 g, 20%) (Bhaskar *et al.*, 2001). The addition of water to the recrystallization filtrate, followed by prolonged cooling, produced a crop of colourless needles that were collected by filtration, washed with diethyl ether and dried in air. Spectroscopic and X-ray crystallographic characterization of the needles revealed the second product to be D-mannitol-1,2,6-tribenzoate (0.31 g, 2%); m.p. 438–441 K [literature 439–440 K (Hockett & Fletcher, 1944)].

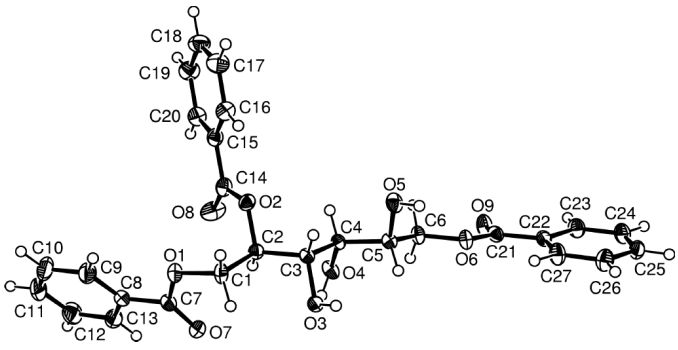


Figure 1
View of (I) (50% probability displacement ellipsoids).

Crystal data

$C_{27}H_{26}O_9$
$M_r = 494.48$
Orthorhombic, $P2_12_12_1$
$a = 5.572$ (1) Å
$b = 15.530$ (1) Å
$c = 27.221$ (2) Å
$V = 2355.5$ (5) Å^3
$Z = 4$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35667 reflections
$\theta = 2.6$–28.3°
$\mu = 0.11$ mm^{-1}
$T = 123$ (2) K
Acicular, colourless
$0.30 \times 0.22 \times 0.18$ mm

Data collection

KappaCCD diffractometer
CCD (φ and ω) scans
26379 measured reflections
3347 independent reflections
2894 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 28.3^\circ$
$h = -7 \rightarrow 7$
$k = -20 \rightarrow 17$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.087$
$S = 1.08$
3347 reflections
328 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.3834P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O7^{i}	0.84	2.15	2.9641 (19)	163
O4—H2A···O3^{ii}	0.84	2.15	2.9164 (18)	152
O4—H2A···O3	0.84	2.43	2.8636 (18)	113
O5—H3A···O9^{iii}	0.84	1.94	2.7668 (19)	169

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

As the absolute structure cannot be reliably determined for this light-atom study, all Friedel pairs (2368) have been merged. The H atoms were included in the riding-model approximation. The torsion angles about the C—O bonds of the hydroxyl groups have been refined.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL, DENZO* (Otwinowski & Minor, 1997) 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 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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