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α,α-Trehalose octaacetate ethyl acetate solvate

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All hydroxy groups on the parent compound, trehalose, have been substituted with acetate groups and the solvent of recrystallization, ethyl acetate, has been incorporated into the crystal lattice to give the title compound, $C_{28}H_{38}O_{19}\cdot C_4H_8O_2$.

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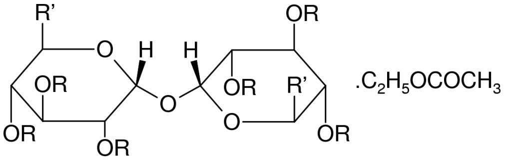
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.096
Data-to-parameter ratio = 9.9

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

Comment

Trehalose is implicated in the preservation of life without water (Branca *et al.*, 1999) and it is used in certain drug formulations (Hatley & Blair, 1999). The role of hydrogen bonding in trehalose and its derivatives is of continuing interest (Clow *et al.*, 2001). Polymorphic forms of α,α-trehalose octaacetate monohydrate have been reported previously (Park & Shin, 1993) and the crystal structure of sucrose octaacetate has been studied (Oliver & Strickland, 1984). We now report the crystal structure of the α,α-trehalose octaacetate ethyl acetate solvate, (I) and base the stereochemistry of the molecule on the known absolute stereochemistry of trehalose.



R=$COCH_3$
R'=CH_2OCOCH_3
(I)

No hydrogen bonds are present in (I). The substitution at C1/C1′ is α,α, with C1—O1 = C1′—O1 = 1.416 (3) Å and C1—O1—C1′ = 113.4 (2)°. The absolute configuration is R at the C atoms C1, C2, C4 and C5 (also at C1′, C2′, C4′ and C5′) and S at C3 and C3′. The two six-membered rings adopt chair conformations with puckering parameters (Cremer & Pople, 1975) calculated with *PLATON* (Spek, 1998) of Q = 0.549 (2) Å, θ = 4.5 (3)°, ø = 14 (3)° (primed atoms) and Q = 0.563 (2) Å, θ = 4.3 (3)° and ø = 106 (4)° (unprimed atoms).

Experimental

The title compound was prepared by reacting anhydrous α,α-trehalose with acetic anhydride. Purification was achieved through a combination of column chromatography and recrystallization from methanol. Crystals for X-ray work were obtained by slow evaporation from ethyl acetate.

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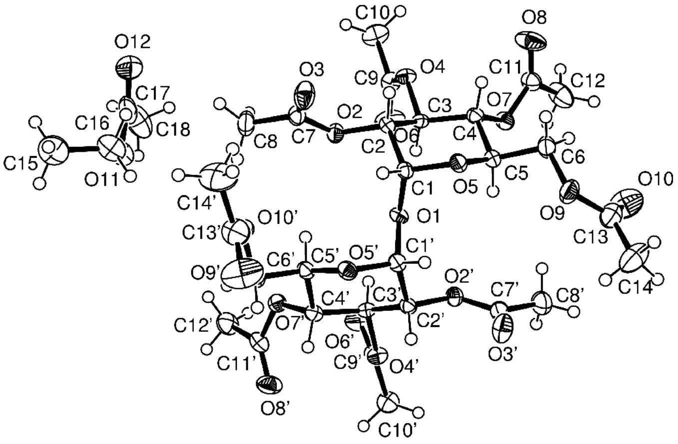

Figure 1
The atomic arrangement in the title molecule. Displacement ellipsoids are shown at the 50% probability level.

Crystal data

$C_{28}H_{38}O_{19}{\cdot}C_4H_8O_2$
$M_r = 766.69$
Orthorhombic, $P2_12_12_1$
$a = 14.1406$ (1) Å
$b = 15.2215$ (1) Å
$c = 17.8879$ (2) Å
$V = 3850.21$ (6) Å^3
$Z = 4$
$D_x = 1.323$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 4833 reflections
$\theta = 2.0$–$27.3°$
$\mu = 0.11$ mm^{-1}
$T = 150$ (2) K
Prism, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
φ and ω scans to fill Ewald sphere
Absorption correction: multi-scan using multiple and symmetry-related data measurements *via* the program *SORTAV* (Blessing, 1995)
$T_{\min} = 0.957$, $T_{\max} = 0.978$

41 400 measured reflections
4833 independent reflections
3409 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.084$
$\theta_{\max} = 27.5°$
$h = -17 \rightarrow 18$
$k = -18 \rightarrow 18$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.096$
$S = 1.00$
4833 reflections
488 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.54$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1′	1.416 (3)	O5—C5	1.438 (3)
O1—C1	1.416 (3)	O5′—C1′	1.410 (3)
O5—C1	1.415 (3)	O5′—C5′	1.439 (3)
C1′—O1—C1	113.36 (17)	O4′—C3′—C2′	105.02 (18)
C1—O5—C5	113.81 (19)	O8′—C11′—C12′	127.2 (3)
C1′—O5′—C5′	113.63 (17)		

The H atoms were initially placed in calculated positions and thereafter allowed to ride on their attached atoms. Each H atom was given an equivalent isotropic displacement parameter 1.2 greater than the attached atom. The H atoms of the methyl groups were allowed to rotate about the local threefold axes to maximize the sum of the electron density at the calculated H-atom positions.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELX*97–2 (Sheldrick, 1998); molecular graphics: *ORTEP*-3 (Farrugia, 1997).

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