

2,2',3,3',4,4'-Hexaacetato-6,6'-bis(isobutanoyl)- α,α -trehalose 0.7-hydrate

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Key indicators

Single-crystal X-ray study

$T = 150\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.017\text{ \AA}$

Disorder in solvent or counterion

R factor = 0.040

wR factor = 0.100

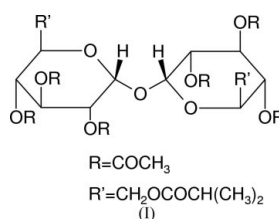
Data-to-parameter ratio = 7.7

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

In the title compound, $\text{C}_{32}\text{H}_{46}\text{O}_{19}\cdot 0.7\text{H}_2\text{O}$, all hydroxy groups on the parent compound, trehalose, have been substituted and some disordered bound water is located in the weakly diffracting crystal.

Comment

Trehalose is a naturally occurring non-reducing disaccharide which is effective in preserving the structural and functional integrity of membranes and proteins from the effects of dehydration (Crowe *et al.*, 1988). Polymorphic, amorphous and crystalline forms of trehalose have been studied (Sussich *et al.*, 1998), and the common crystalline form is the dihydrate (Brown *et al.*, 1972; Taga *et al.*, 1972). The ability of trehalose to readily form hydrates may be related to its support of anhydrobiosis (life without water). We have undertaken a study of substituted trehalose molecules in an attempt to understand the enhanced stabilizing potential of trehalose. In the title compound, (I), all eight hydroxy groups have been substituted, hence removing the possibility of hydrogen-bonding networks with water through these groups.



However, an ill defined water molecule (occupation factor close to 0.7) was found to be incorporated into the crystal lattice. Short distances here are $\text{O1}' \cdots \text{O5}$ 3.097 (15) \AA and $\text{O1}' \cdots \text{O6}'(-1+x, y, z)$ 2.965 (13) \AA , but uncertainty in location of water H atoms precludes a reliable description of the hydrogen bonding. The substitution at C1, C1' is α,α , with $\text{C1}-\text{O1} = 1.453$ (11) \AA , $\text{C1}'-\text{O1} = 1.435$ (11) \AA and $\text{C1}-\text{O1}-\text{C1}' = 115.5$ (8) $^\circ$. 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 $Q = 0.56$ (1) \AA , $\theta = 9$ (1) $^\circ$, $\varphi = 94$ (7) $^\circ$ (primed atoms) and $Q = 0.57$ (1) \AA , $\theta = 3$ (1) $^\circ$ and $\varphi = 322$ (21) $^\circ$ (unprimed atoms).

Experimental

The title compound, (I), was prepared *via* the reaction mixture sequence of trehalose and Ph_3CCl followed by addition of CH_3COCl and pyridine, then aqueous hydrobromide was added and finally a

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mixture of $\text{CH}_3\text{CH}(\text{CH}_3)\text{COCl}$ and pyridine was prepared and added. Purification was achieved through a combination of column chromatography and recrystallization. Crystals for X-ray work were obtained by slow evaporation from 95% ethanol.

Crystal data

$\text{C}_{32}\text{H}_{46}\text{O}_{19} \cdot 0.7\text{H}_2\text{O}$

$M_r = 747.30$

Orthorhombic, $P2_12_12_1$

$a = 8.857(3) \text{ \AA}$

$b = 17.813(11) \text{ \AA}$

$c = 24.085(3) \text{ \AA}$

$V = 3800(3) \text{ \AA}^3$

$Z = 4$

$D_x = 1.306 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

Cell parameters from 250 reflections

$\theta = 2.0\text{--}25.0^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 150(2) \text{ K}$

Lozenge, colourless

$0.21 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Delft Instruments FAST diffractometer

φ and ω scans to fill Ewald sphere

17 076 measured reflections

3541 independent reflections

565 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.311$

$\theta_{\text{max}} = 25.0^\circ$

$h = -10 \rightarrow 8$

$k = -20 \rightarrow 21$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.100$

$S = 0.39$

3541 reflections

460 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2)]$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Absolute structure: none

Detailed data collection procedures are described by Darr *et al.* (1993). The number of intensities with $F^2 > 2\sigma F^2$ was only 16% of the total (565 compared with a total of 3541 unique reflections). Structure solution and refinement with such a weak data led to results that were below the standard normally expected, but are adequate for a qualitative assessment of the structure. With *SIR92* (Altomare *et al.*, 1994), the number of atoms in the unit cell was lowered to $\text{C}_{100}\text{H}_{168}\text{O}_{76}$ from $\text{C}_{128}\text{H}_{192}\text{O}_{80}$ to achieve suitable phasing. All non-H atoms were refined using the *DELU* command which applies rigid bond restraints was used. The H atoms were initially placed in calculated positions and thereafter allowed to ride on their attached atoms [$U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$]. Water H atoms were not observed on a final difference map but were calculated with *CALC-OH* (Nardelli, 1999) and the coordinates fixed. Friedel pairs were merged. The absolute configuration is known for trehalose.

Data collection: *MADNES* (Plugraht & Messerschmidt, 1989); cell refinement: *MADNES*; data reduction: *ABSMAD* (Karaulov, 1992); program(s) used to refine structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to solve structure: *SHELX97* (Sheldrick, 1997);

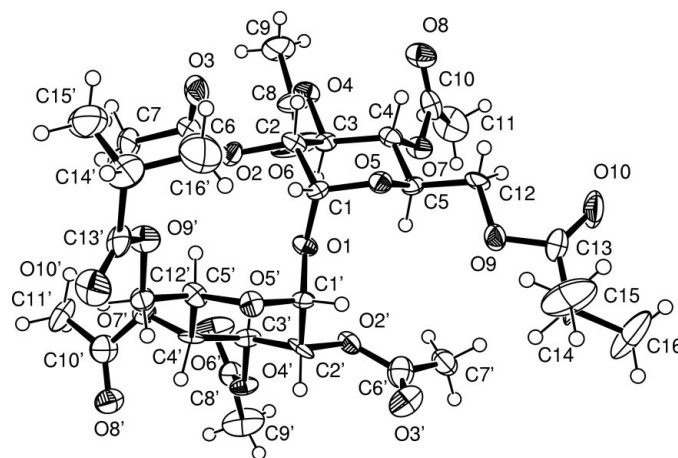


Figure 1

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

molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *ORTEP-3* (Farrugia, 1997).

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References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Brown, G. M., Rohrer, D. C., Berking, B., Bevers, C. A., Gould, R. O. & Simpson, R. (1972). *Acta Cryst.* **B28**, 3145–3158.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Crowe, J. H., Crowe, L. M. & Carpenter, J. F. (1988). *Biochem. Biophys. Acta*, **947**(2), 367–384.
- Darr, J. A., Drake, S. R., Hursthouse, M. B. & Malik, K. M. A. (1993). *Inorg. Chem.* **32**, 5704–5708.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
- Karaulov, A. I. (1992). *ABSMAD*. University of Wales, Cardiff, Wales.
- Nardelli, M. (1999). *J. Appl. Cryst.* **52**, 563–571.
- Plugraht, J. W. & Messerschmidt, A. (1989). *MADNES*. Version 11 of September, 1989. Distributed by Delft Instruments, Delft, The Netherlands.
- Sheldrick, G. M. (1997). *SHELX97*. University of Göttingen, Germany.
- Spek, A. L. (1998). *PLATON*. Version 16. University of Utrecht, The Netherlands.
- Sussich, F., Urbani, R., Princivalle, F. & Cesaro, A. (1998). *J. Am. Chem. Soc.* **120**, 7893–7899.
- Taga, T., Senma, M. & Osaki, K. (1972). *Acta Cryst.* **B28**, 3258–3263.