

C(5)—C(10)—C(19)	123.4 (2)	C(21)—C(20)—C(22)	108.5 (2)
C(5)—C(6)—C(7)	130.6 (2)	C(20)—C(22)—C(23)	124.9 (2)
C(6)—C(7)—C(8)	123.1 (2)	C(22)—C(23)—C(24)	126.5 (2)
C(7)—C(8)—C(9)	125.5 (2)	C(23)—C(24)—C(25)	110.5 (2)
C(7)—C(8)—C(14)	124.2 (2)	C(23)—C(24)—O(24)	112.2 (2)
C(9)—C(8)—C(14)	110.4 (2)	O(24)—C(24)—C(25)	106.6 (2)
C(8)—C(9)—C(11)	112.8 (2)	C(24)—C(25)—C(26)	119.8 (2)
C(9)—C(11)—C(12)	112.2 (2)	C(24)—C(25)—C(27)	120.7 (2)
C(11)—C(12)—C(13)	111.0 (2)	C(26)—C(25)—C(27)	60.0 (2)
C(12)—C(13)—C(14)	108.3 (2)	C(25)—C(26)—C(27)	60.1 (2)
C(12)—C(13)—C(18)	110.2 (2)	C(25)—C(27)—C(26)	60.0 (2)
C(12)—C(13)—C(17)	116.2 (2)		

Table 3. Torsion angles ($^{\circ}$)

C(10)—C(12)—C(2)—C(3)	54.5 (3)
C(1)—C(2)—C(3)—C(4)	-57.0 (3)
C(2)—C(3)—C(4)—C(5)	52.5 (3)
C(3)—C(4)—C(5)—C(10)	-46.8 (3)
C(4)—C(5)—C(10)—C(1)	43.9 (3)
C(10)—C(5)—C(6)—C(7)	-2.8 (5)
C(5)—C(6)—C(7)—C(8)	179.7 (3)
C(6)—C(7)—C(8)—C(9)	-6.4 (4)
C(7)—C(8)—C(9)—C(11)	-53.8 (3)
C(8)—C(9)—C(11)—C(12)	51.8 (3)
C(9)—C(11)—C(12)—C(13)	-53.8 (3)
C(11)—C(12)—C(13)—C(14)	56.8 (3)
C(12)—C(13)—C(14)—C(8)	-60.2 (3)
C(15)—C(14)—C(13)—C(17)	46.2 (2)
C(13)—C(14)—C(15)—C(16)	-34.6 (3)
C(14)—C(15)—C(16)—C(17)	9.5 (3)
C(15)—C(16)—C(17)—C(13)	19.2 (3)
C(16)—C(17)—C(13)—C(14)	-39.4 (3)
C(16)—C(17)—C(20)—C(22)	57.3 (3)
C(17)—C(20)—C(22)—C(23)	-131.5 (3)
C(20)—C(22)—C(23)—C(24)	-173.7 (3)
C(22)—C(23)—C(24)—C(25)	110.5 (3)
C(23)—C(24)—C(25)—C(26)	81.2 (3)
C(23)—C(24)—C(25)—C(27)	151.9 (3)
C(24)—C(25)—C(26)—C(27)	110.4 (3)
C(24)—C(25)—C(27)—C(26)	-109.0 (3)

Table 4. Hydrogen-bond geometry (\AA , $^{\circ}$)

D—H...A	D—A	D—H—A	H—A
O(1)—H(10)—O(W1) ^y	2.801 (3)	172 (3)	2.10 (4)
O(3)—H(30)—O(1) ⁱⁱ	2.818 (2)	148 (4)	2.23 (4)
O(24)—H(240)—O(3) ⁱⁱⁱ	2.703 (3)	169 (4)	2.01 (4)
O(W1)—H(W1)—O(24) ⁱⁱⁱ	2.871 (3)	153 (3)	2.15 (4)
O(W1)—H(W2)—O(24) ^v	2.838 (2)	170 (3)	1.95 (3)

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

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55648 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1021]

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Structure of β,β -Trehalose

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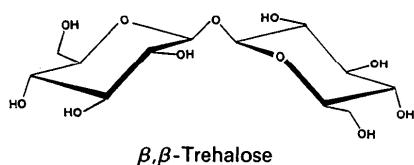
Abstract

The structure of β,β -trehalose (β -D-glucopyranosyl β -D-glucopyranoside) tetrahydrate, $C_{12}H_{22}O_{11} \cdot 4H_2O$, is described. The molecule, unlike its α,α -isomer, has exact twofold symmetry through the O(1) atom. The two glucopyranosyl residues adopt a slightly distorted chair 4C_1 conformation, as defined by the Cremer & Pople puckering parameters, $Q = 0.567$, $q_2 = 0.067$, $q_3 = 0.563 \text{ \AA}$, $\theta = 6.8^{\circ}$ and $\varphi_2 = 159.4^{\circ}$. The packing of the crystal is determined by a complex system of hydrogen bonds, each hydroxyl group of the sugar forming two hydrogen bonds and each water molecule forming four hydrogen bonds. The ring and the glycosidic O atoms are, however, not involved in any hydrogen bonding.

Comment

The structure, chemistry and biochemistry of the naturally occurring non-reducing disaccharide, α,α -trehalose (α -D-glucopyranosyl α -D-glucopyranoside), also known as mycose or mushroom sugar, has been widely studied (Birch, 1963; Lee, 1980). Little is known, however, of the synthetic isomeric β,β -trehalose and α,β -trehalose (Fischer & Delbrück, 1909; Schlubach & Maurer, 1925; Haworth & Hickenbottom, 1931; Helferich & Weis, 1956). Interest has recently been shown (Colaco, 1992) in the use of these synthetic analogues in biochemical and medical studies, hence our interest

in the X-ray crystal diffraction study of β,β -trehalose.



A *SHELXTL-Plus* (Sheldrick, 1990) *XP* plot of the title compound with the atomic numbering scheme is shown in Fig. 1. Unlike its α,α -anomer, β,β -trehalose exhibits exact twofold symmetry with atom O(1) lying on a twofold axis. The torsion angles about the C(5)—C(6) bond involving O(6) with O(5) and with O(4) are 62.1 and -59.5° , respectively, and describe a *gauche-gauche* (or $+sc$) arrangement which is the same as that for β -D-glucopyranose (Ferrier, 1963); in α,α -trehalose, one of the C(6)—O(6) bonds has the $+sc$ while the other has the $-sc$ conformation (Taga, Senna & Osaki, 1972; Brown, Rohrer, Berking, Beevers, Gould & Simpson, 1972). The torsion angles about C(1)—O(1) and C(1a)—O(1) clearly describe the *E1* conformer for each of the two glucopyranosyl—O(1) units. The torsion angle of the glycosidic bond O(5)—C(5)—O(1)—C(1a), which is 75.6° , describes the *gauche* conformation, this being the preferred conformation of methyl β -D-pyranosides (Jeffrey & Tagaki, 1977), and the angle -167.2° [C(2)—C(1)—O(1)—C(1a)] describes the *trans* or antiperiplanar conformation.

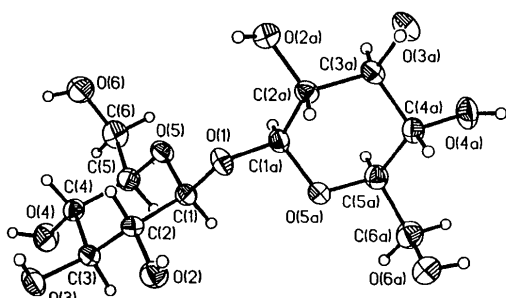


Fig. 1. Molecular conformation of β,β -trehalose.

Both the pyranose rings have the chair 4C_1 conformation but each ring is significantly more puckered and less regular (with ring torsion angles varying from 49 to 65°) than in α - and β -D-glucopyranose (Brown & Levy, 1963; Chu & Jeffrey, 1968) and their methyl glycosides (Jeffrey & Tagaki, 1977), where the torsion angles lie in the more commonly observed range of 54 – 64° . The puckering parameters of the ring (Cremer & Pople, 1975) describe a slightly dis-

torted chair, Q being only slightly less than that for an ideal cyclohexane ring [0.63 Å for $R(C-C) = 1.54$ Å], θ is very small, and $q_3 \gg q_2$ ($q_2 = 0.067$, $q_3 = 0.563$, $Q = 0.567$ Å, $\theta = 6.8^\circ$, $\varphi_2 = 159.4^\circ$). The large φ_2 value indicates that the direction of distortion is towards an inverted boat conformation. The principal reason for this distortion is probably the extensive hydrogen-bonding network and the influence of the water in determining the hydrogen-bond structure (see below).

Bond angles (Table 2), including those at the ring O atom (112.2°) and the glycosidic O (115.2°), are in the normal range for such angles in pyranose rings (Sundaralingam, 1968) and are almost identical to those found in α,α -trehalose. The latter is rather surprising since the demands of the anomeric effect and hydrogen bonding are absent. Since this is the first structure reported for a carbohydrate oligomer containing a glycosidic O atom which is equatorial with respect to two sugar rings, it is difficult to predict whether this equivalence is coincidental. However, the angle is comparable to those found in other oligosaccharides, such as sucrose (114.4°), methyl maltoside (117.6°) (Berman, 1970), cellobiose (116.1°) (Chu & Jeffrey, 1968) or planteose (116.9°) (Rohrer, 1972).

The C—C bond lengths (Table 2) agree well with each other and with accepted values, the mean being 1.522 Å and the maximum deviation 0.011 Å. These appear to be marginally shorter than those of the α,α -anomer but the mean value compares well with that of cellobiose (1.51 Å) (Jacobson, Wunderlich & Lipscomb, 1961). The C—OH bond lengths range from 1.418 to 1.428 Å with a mean value of 1.423 Å, in agreement with the accepted value of 1.42 Å (Dewar & Thiel, 1977). The C(1)—O(1) bond is identical to that found in β -D-glucopyranose (Ferrier, 1963) but is shorter than that in the α,α -anomer by 0.017 Å. There is a marked shortening of the C(1)—O(5) and C(5)—O(5) bonds which may be related to the fact that the ring O atoms of β,β -trehalose do not take part in hydrogen bonding whereas that in β -D-glucopyranose does.

The molecular packing in the crystal is shown in Fig. 2. The molecules of the sugar and water are linked by an extensive and complex network of nine crystallographically independent hydrogen bonds, the distances and angles of which are shown in Table 3. All the hydroxyl groups are involved in hydrogen bonding, each hydroxyl group acting as both donor and acceptor. Only the C(2) and C(4) hydroxyl groups are involved in intermolecular hydrogen bonding, each acting as both donor and acceptor, and each is in turn hydrogen bonded to a water molecule. Neither the ring oxygen, O(5), nor the anomeric oxygen, O(1), is involved in hydrogen bonding. Each glycosyl unit is hydrogen bonded to

two water molecules, and each of the water molecules is tetrahedrally coordinated, three with the hydroxyl groups of the sugar and the fourth with another water molecule. It is interesting to note that, unlike the α,α -anomer, there are no water molecules bridging the hydroxyl groups of the two halves of the sugar molecules. But like the α,α -anomer, there is no direct intramolecular hydrogen bonding such as that found in sucrose (Brown & Levy, 1963), methyl β -maltoside (Chu & Jeffrey, 1967), cellobiose (Chu & Jeffrey, 1968) and β -lactose monohydrate (Fries, Rao & Sundaralingam, 1971).

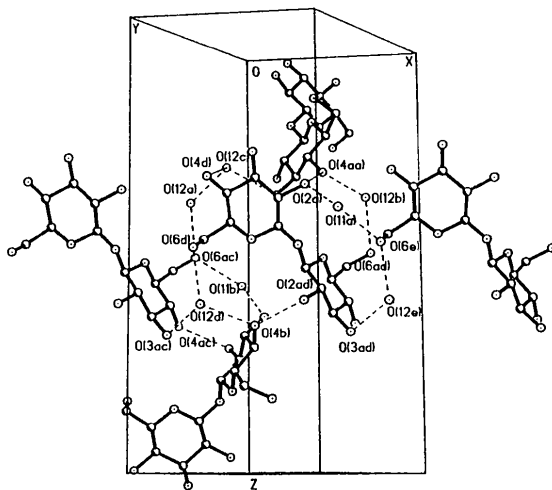


Fig. 2. Molecular packing in β,β -trehalose tetrahydrate.

Experimental

Crystal data

$C_{12}H_{22}O_{11} \cdot 4H_2O$

$M_r = 414.4$

Trigonal

$P3_221$

$a = 8.789 (2) \text{ \AA}$

$c = 21.245 (8) \text{ \AA}$

$V = 1421.3 (7) \text{ \AA}^3$

$Z = 3$

$D_x = 1.452 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 14 reflections

$\theta = 4.5\text{--}10.5^\circ$

$\mu = 0.127 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Prisms

$0.35 \times 0.3 \times 0.25 \text{ mm}$

Colourless

Data collection

Siemens R3m/V diffractometer

ω scans

Absorption correction:

none

2768 measured reflections

1690 independent reflections

1458 observed reflections

$[F > 4.0\sigma(F)]$

$R_{int} = 0.024$

$\theta_{max} = 25^\circ$

$h = -10 \rightarrow 9$

$k = 0 \rightarrow 10$

$l = 0 \rightarrow 24$

2 standard reflections

monitored every 98

reflections

intensity variation: 1.5%

Refinement

Refinement on F

Final $R = 0.029$

$wR = 0.031$

$S = 1.33$

1458 reflections

181 parameters

Only coordinates of H atoms refined

$w = 1/[\sigma^2(F) + 0.0001F^2]$

$(\Delta/\sigma)_{max} = 0.050$

$\Delta\rho_{max} = 0.24 \text{ e \AA}^{-3}$

$\Delta\rho_{min} = -0.22 \text{ e \AA}^{-3}$

Extinction correction:

$F^* = F(1 + 0.002\chi F^2 / \sin 2\theta)^{-0.25}$

Extinction coefficient:

0.0057

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

$U_{eq} = 1/3(\text{trace of the orthogonalized } U_{ij} \text{ tensor}).$

	x	y	z	U_{eq}
C(1)	0.2986 (3)	0.9094 (2)	0.1214 (1)	0.024 (1)
C(2)	0.1628 (3)	0.8579 (3)	0.0697 (1)	0.024 (1)
C(3)	0.1826 (3)	0.7410 (3)	0.0214 (1)	0.025 (1)
C(4)	0.1885 (3)	0.5885 (3)	0.0535 (1)	0.026 (1)
C(5)	0.3194 (3)	0.6534 (3)	0.1081 (1)	0.028 (1)
C(6)	0.3176 (3)	0.5075 (3)	0.1459 (1)	0.039 (1)
O(1)	0.2585 (2)	1.0000	0.1667	0.028 (1)
O(2)	0.1743 (2)	1.0070 (2)	0.0394 (1)	0.033 (1)
O(3)	0.0455 (2)	0.6783 (2)	-0.0244 (1)	0.035 (1)
O(4)	0.2348 (2)	0.4957 (2)	0.0100 (1)	0.040 (1)
O(5)	0.2803 (2)	0.7555 (2)	0.1504 (1)	0.029 (1)
O(6)	0.1498 (2)	0.3944 (2)	0.1726 (1)	0.044 (1)
O(11)	0.9504 (2)	0.0885 (2)	0.1080 (1)	0.044 (1)
O(12)	0.7197 (2)	0.5658 (2)	0.0340 (1)	0.042 (1)

Table 2. Geometric parameters ($\text{\AA}, ^\circ$)

C(1)—C(2)	1.515 (3)	C(4)—C(5)	1.529 (3)
C(1)—O(1)	1.402 (2)	C(4)—O(4)	1.420 (3)
C(1)—O(5)	1.420 (3)	C(5)—C(6)	1.506 (4)
C(2)—C(3)	1.522 (3)	C(5)—O(5)	1.428 (3)
C(2)—O(2)	1.418 (3)	C(6)—O(6)	1.421 (3)
C(3)—C(4)	1.528 (3)	C(1)—C(1a)	1.401 (2)
C(3)—O(3)	1.428 (2)		
C(1)—C(2)—C(3)	110.6 (2)	C(3)—C(4)—C(5)	111.1 (2)
C(1)—C(2)—O(2)	111.8 (1)	C(3)—C(4)—O(4)	111.0 (2)
C(1)—O(1)—C(1a)	115.2 (2)	C(4)—C(3)—O(3)	111.0 (3)
C(1)—O(5)—C(5)	112.2 (1)	C(4)—C(5)—C(6)	113.7 (2)
C(2)—C(1)—O(1)	106.4 (2)	C(4)—C(5)—O(5)	109.8 (2)
C(2)—C(1)—O(5)	109.4 (1)	C(5)—C(4)—O(4)	109.2 (2)
C(2)—C(3)—C(4)	110.9 (2)	C(5)—C(6)—O(6)	112.0 (2)
C(2)—C(3)—O(3)	111.1 (3)	C(6)—C(5)—O(5)	107.2 (2)
C(3)—C(2)—O(2)	109.8 (1)	O(1)—C(1)—O(5)	107.6 (1)
C(1)—C(2)—C(3)—C(4)	50.7 (2)		
C(1)—C(2)—C(3)—O(3)	-61.5 (3)		
C(1)—C(2)—C(3)—C(4)	174.6 (2)		
C(2)—C(3)—C(4)—C(5)	-48.9 (2)		
C(2)—C(3)—C(4)—O(4)	-170.7 (1)		
C(3)—C(4)—C(5)—C(6)	174.0 (2)		
C(3)—C(4)—C(5)—O(5)	53.9 (2)		
C(4)—C(5)—O(5)—C(1)	-63.0 (2)		
C(5)—O(5)—C(1)—C(2)	65.1 (2)		
O(1)—C(1)—C(2)—C(3)	-173.8 (1)		
O(1)—C(1)—O(5)—C(5)	-179.7 (1)		
O(5)—C(1)—C(2)—C(3)	-57.8 (2)		
O(1)—C(1)—C(2)—O(2)	63.5 (2)		
O(2)—C(2)—C(3)—O(3)	174.6 (2)		
O(3)—C(3)—C(4)—O(4)	65.3 (2)		
O(4)—C(4)—C(5)—C(6)	-63.2 (2)		
C(2)—C(1)—O(1)—C(1a)	-167.2 (1)		
C(4)—C(5)—C(6)—O(6)	-59.4 (3)		
O(5)—C(1)—O(1)—C(1a)	75.6 (1)		
O(5)—C(5)—C(6)—O(6)	62.1 (2)		

Table 3. *Hydrogen bonds in the crystal structure of β,β -trehalose*

Symmetry operations: (1) x, y, z ; (2) $y, x, -z$; (3) $x - y, -y, \frac{1}{2} - z$. The last three digits specify a lattice translation. Each O of H₂O is hydrogen bonded to four other O's. Each O of sugar OH is bonded to two other O's. O(1) and O(5) are not hydrogen bonded. O(X)→O(Y) means O(X)—H···O(Y). O(X)↔O(Y) means O(X)—H···O(Y) and O(X)···H—O(Y) (50:50) (i.e. disorder in H).

Hydrogen bond	Symmetry operation	Distances (Å)						Angles (°)	
		O—H	H—O	O···O	O···H	H···O	O—H···O	O···H—O	
O(4)↔O(2)	[2,100]	0.80	0.79	2.726	1.95	1.93	173	167	
O(11)↔O(2)	[1,110]	0.83	0.81	2.814	2.02	1.99	172	166	
O(11)→O(3)	[2,000]	0.82	—	2.848	—	2.03	176	—	
O(11)→O(6)	[1,100]	—	0.81	2.733	1.93	—	—	171	
O(11)↔O(11)	[3,000]	0.82	0.82	2.835	2.01	2.01	180	180	
O(12)→O(3)	[1,100]	—	0.81	2.808	2.00	—	—	174	
O(12)↔O(4)	[2,000]	0.81	0.82	2.736	1.94	1.95	163	163	
O(12)→O(6)	[3,110]	0.82	—	2.712	—	1.90	—	170	
O(12)↔O(12)	[2,000]	0.82	0.82	2.752	1.93	1.93	180	180	
E.s.d.'s		±0.05		±0.003		±0.05		±13	

H atoms were located from a difference map. Their positions were refined with fixed isotropic thermal parameters. Final C—H distances range from 0.97 to 1.00 Å, O—H from 0.79 to 0.83 Å. H atoms attached to O(2), O(4) and one of the H atoms of O(11) and O(12) were disordered as indicated by the difference map. Each of these were assigned two positions with site occupancy factors of 0.5. The disorder is required in order to describe the hydrogen-bonding scheme. The disordered model gave a final *R* of 0.029 against an *R* of 0.042 for the non-disordered model. The structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1990).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55632 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1014]

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Structure of *N*-(3-Phthalimidooxypropyl)-trifluoroacetamide

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Abstract

The title compound consists of a planar phthalimide ring linked via a single N—O bond [1.377 (6) Å] and a propyl chain to an amide function. The packing is stabilized by hydrogen bonds formed between the amide groups.

Comment

In connection with the synthesis of some polyamine analogues, the title intermediate was prepared (Kong Thoo Lin, 1992) from 3-amino-1-propanol by selectively *N*-trifluoroacetylating with ethyl trifluoroacetate/triethylamine in methanol followed by *O*-toluene-*p*-sulfonation in pyridine. Reaction with