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The Crystal and Molecular Structure of Maltitol

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The crystal structure of maltitol, 4-O- α -D-glucopyranosyl-D-glucitol, has been determined by X-ray diffraction method. The crystal is orthorhombic with cell parameters of $a = 8.170(1)$, $b = 12.731(1)$, $c = 13.679(3)$ Å, space group $P2_12_12_1$ and $z = 4$. The structure was solved by direct methods and refined to $R = 0.030$ for 1181 observed reflections measured on a diffractometer. The α -glucose ring has chair conformation. The carbon atom chain of the glucitol residue has the bent, *ap*, *Psc*, *Psc* conformation. The angle at the ring oxygen atom is 112.6° and the one at the glucosidic oxygen is 117.1° . The molecules are linked by very complicated hydrogen bonds, and there is an intramolecular hydrogen bond between O(1') and O(2').

Introduction

X-ray diffraction technique can be widely used in the determination of the conformations and the configurations of carbohydrates. The importance of the molecular conformations of carbohydrates has long been recognized, and therefore a number of structure investigations of monosaccharides, disaccharides, polysaccharides and their derivatives have been carried out using X-ray and neutron diffraction methods¹⁻³.

In this study, the crystal and molecular structure of maltitol (4-O- α -D-glucopyranosyl-D-glucitol) is determined from X-ray diffraction method to compare this conformation with those of maltose related compounds and derivatives⁴⁻⁸ to be determined already.

Maltitol is derived from the disaccharide maltose and can

be formed by reductive ring opening of its reducing pyranose ring. In this class of hexosylhexitols, composed of an aldopyranose linked to a hexitol by a C-O-C glycosidic bridge, the acyclic carbon chain of the molecule is much more flexible than the rigid pyranose ring.

In the work of Jeffrey and Kim⁹ on the conformations of alditols, $HOCH_2(CHOH)_nCH_2OH$, acyclic carbon chains have the extended, planar, zigzag arrangement, unless this would give rise to a steric interaction between substituents on alternate carbon atoms. If such an interaction is possible, the carbon chain adopts, by a 120° rotation about a C-C bond, a non-linear, or bent chain conformation so that these interactions are avoided. The aim of the present study is also to examine the acyclic carbon chain conformation in this structure and to compare with that observed in D-glucitol compounds.

Table 1. Final Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Thermal Parameters of Maltitol

$$U_{eq.} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j (\text{\AA}^2 \times 10^3)$$

atom	x	y	z	Ueq.
C(1)	8586(4)	9771(2)	3887(2)	20
C(2)	9046(4)	9037(2)	3049(2)	21
C(3)	8269(4)	7959(2)	3148(2)	21
C(4)	8417(4)	7530(2)	4182(2)	20
C(5)	7807(4)	8334(2)	4912(2)	20
C(6)	7932(4)	7994(3)	5968(2)	24
O(1)	6957(2)	10081(1)	3748(2)	19
O(2)	8680(3)	9509(2)	2128(2)	29
O(3)	9046(3)	7269(2)	2465(2)	31
O(4)	7470(3)	6592(2)	4265(2)	29
O(5)	8805(2)	9272(1)	4807(1)	20
O(6)	9502(3)	7661(2)	6258(2)	30
C(1')	5927(4)	13835(2)	3344(2)	26
C(2')	5562(4)	12906(2)	3999(2)	19
C(3')	6586(3)	11967(2)	3694(2)	18
C(4')	6386(3)	10966(2)	4310(2)	17
C(5')	4647(3)	10719(2)	4661(2)	18
C(6')	3553(4)	10218(2)	3899(2)	23
O(1')	4748(3)	14672(2)	3428(1)	28
O(2')	5863(3)	13212(2)	4984(2)	28
O(3')	8291(2)	12267(2)	3746(2)	25
O(5')	4704(3)	9960(2)	5439(2)	23
O(6')	3389(3)	10851(2)	3044(2)	32

*Tables for anisotropic thermal parameters of the nonhydrogen atoms, coordinates of hydrogen atoms, structure factors are available from the author(YJP).

Experimental

A maltitol was obtained from Sigma Chemical Company.

Colorless crystals were obtained by slow evaporation of the solution of aqueous ethanol and methanol at 5°C.

Preliminary X-ray Weissenberg photographs showed that the crystal system is orthorhombic with space group $P2_12_12_1$. The unit cell parameters were determined by least-squares fit of 2θ angles for 23 reflections in the range of $22^\circ < 2\theta < 28^\circ$ on the diffractometer.

Intensity data were measured on Enraf-Nonius CAD-4 diffractometer with graphite-monochromatized Mo-K α radiation with a crystal of dimension $0.3 \times 0.4 \times 0.7$ mm. The intensities were measured using $\omega-2\theta$ scan technique over a scan width of $(0.8 + 0.34 \tan \theta)^\circ$ in ω . One standard reflection (3 5 4) was remeasured every 5000 seconds and three orientation reflections (1 8 0), (3 4 4), (1 6 4) were monitored every 100 reflections, no significant loss of intensities being observed throughout data collection. The 1222 independent reflections measured with the range of $2^\circ < 2\theta < 47^\circ$ of which 1181 with the $|F| > 2\sigma |F|$ were considered to be observed. Corrections for Lorentz and polarization effects were applied to the intensity data, while no absorption or extinction correction was carried out.

The crystal data are as follows. C₁₂H₂₄O₁₁; Mol. wt. 344.2; Space Group $P2_12_12_1$; $a = 8.170$ (1), $b = 12.731$ (1),

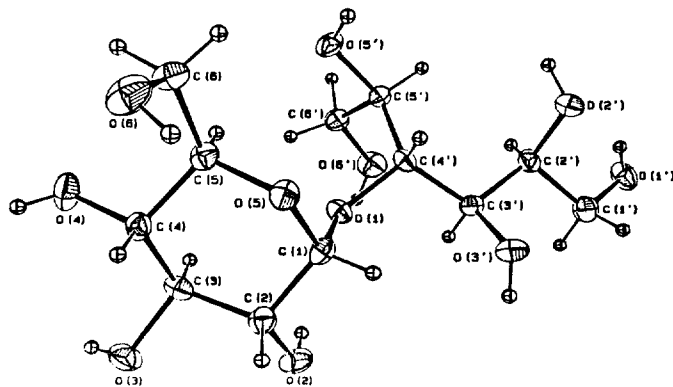


Figure 1. Molecular conformation and atomic numbering of Maltitol.

$c = 13.679$ (3) Å; $Z = 4$; $D_c = 1.607$ g/cm³, $D_m = 1.58$ g/cm³ measured by floatation method in benzene-methyl iodide mixture; $\mu(\text{Mo-K}\alpha 0.7107 \text{\AA}) = 0.94$ cm⁻¹.

Structure was solved by direct method of SHELXS-86 program¹⁰ using 156 reflections whose E values were greater than 1.30. Best E map gave recognizable all non-hydrogen atoms in an asymmetric unit.

The refinement was carried out by the least squares method using SHELX-76 program¹¹. Four cycles of isotropic and three cycles anisotropic full matrix least squares refinements reduced R to 0.12. Three more cycles of anisotropic refinements for non-hydrogen atoms lowered R value to 0.066. Reasonable all 24 hydrogen atoms could be identified on the difference map with electron densities ranging from 0.26 to 0.55 eÅ⁻³. The function $\sum w(|F_o| - |F_c|)^2$ was minimized in the refinements, where w , the weight of the reflection, was defined by $w = 0.77/[\sigma(F_o)^2 + 0.004(F_o)^2]$. In the final refinement, the positional parameters of all atoms, anisotropically for non-hydrogen atoms, isotropic thermal parameters for the hydrogen atoms were refined.

The final R and R_w values were 0.030 and 0.035, respectively for the 1181 observed reflections. The final positional and thermal parameters for C and O atoms are listed in Table 1.*

Results and Discussion

Molecular Conformation. The conformation of the molecule and the numbering of the atoms are shown in Figure 1. The glucose and glucitol moieties are denoted by unprimed and primed atom designators respectively.

Bond distances and angles involving nonhydrogen atoms are given in Table 2.

The C-C bonds range from 1.513 to 1.536 Å and average bond length is 1.520 Å. As in many pyranosides and in some alditol structures, the exocyclic C(5)-C(6) bond length and the outer terminal bonds, C(1')-C(2') and C(5')-C(6'), of the glucitol moiety are significantly shorter than the other C-C bond lengths. It has been remarked that the chemical significance of this trend is difficult to assess because of uncertainty in the treatment of the thermal motion. The C-OH bonds range from 1.408 to 1.447 Å (mean 1.430 Å). The bond lengths in the acetal sequence of bonds C(5)-O(5)-C(1)-O(1)-C(4') show the characteristic variation observed in other α -pyranosides, in that the anomeric C(1)-O(1) bond of 1.401

Table 2. Bond Lengths(Å) and Angles(°) for Maltitol

C(1)-C(2)	1.526(4)	C(2)-O(2)	1.427(4)
C(2)-C(3)	1.518(4)	C(3)-O(3)	1.430(4)
C(3)-C(4)	1.520(4)	C(4)-O(4)	1.427(4)
C(4)-C(5)	1.514(4)	C(6)-O(6)	1.408(4)
C(5)-C(6)	1.511(4)	C(1')-O(1')	1.441(4)
C(1')-C(2')	1.513(4)	C(2')-O(2')	1.423(4)
C(2')-C(3')	1.518(4)	C(3')-O(3')	1.447(3)
C(3')-C(4')	1.536(4)	C(5')-O(5')	1.439(4)
C(4')-C(5')	1.533(4)	C(6')-O(6')	1.427(4)
C(5')-C(6')	1.513(4)	mean value	1.430(11)
mean value	1.520(8)		
	C(1)-O(5)	1.421(3)	
	C(1)-O(1)	1.401(4)	
	C(5)-O(5)	1.453(3)	
	O(1)-C(4')	1.442(3)	
C(1)-C(2)-C(3)	112.5(2)	C(1')-C(2')-C(3')	110.1(2)
C(2)-C(3)-C(4)	112.0(2)	C(2')-C(3')-C(4')	116.4(2)
C(3)-C(4)-C(5)	110.2(2)	C(3')-C(4')-C(5')	116.1(2)
C(4)-C(5)-C(6)	114.5(2)	C(4')-C(5')-C(6')	114.7(2)
mean value	113.3(23)		
C(1)-C(2)-O(2)	110.7(2)	C(2')-C(1')-O(1')	113.5(2)
C(3)-C(2)-O(2)	111.9(2)	C(1')-C(2')-O(2')	108.2(2)
C(2)-C(3)-O(3)	108.1(2)	C(3')-C(2')-O(2')	112.4(2)
C(4)-C(3)-O(3)	110.6(2)	C(2')-C(3')-O(3')	108.0(2)
C(3)-C(4)-O(4)	109.4(2)	C(4')-C(3')-O(3')	107.1(2)
C(5)-C(4)-O(4)	109.5(2)	C(4')-C(5')-O(5')	109.8(2)
C(5)-C(6)-O(6)	114.7(3)	C(6')-C(5')-O(5')	104.2(2)
	mean value	110.0(26)	
C(2)-C(1)-O(5)	111.1(2)	C(1)-O(1)-C(4')	117.1(2)
C(2)-C(1)-O(1)	107.7(2)	C(1)-O(5)-C(5)	112.6(2)
O(1)-C(1)-O(5)	111.5(2)	O(1)-C(4')-C(3')	108.7(2)
C(4)-C(5)-O(5)	107.8(2)	O(1)-C(4')-C(5')	107.8(2)
C(6)-C(5)-O(5)	107.0(2)		

Å is very short and C(5)-O(5) bond length of 1.453 Å is significantly (10σ) longer than other ring C(1)-O(5) bond length of 1.421 Å. The C(4')-O(1) bridge bond is consistently significantly (13σ) longer than C(1)-O(1).

The C-C-C bond angles are in the range 110.2-116.4° (average 113.3°), the C-C-OH bond angles from 107.1 to 114.7° (average 110.0°). These results and the valence angles involving bridge and ring oxygen atoms are not significantly different from those of other known maltose related compounds.

The conformation angles of the pyranose ring range from 45.2° to 66.0° (Table 3). The ring has the distorted chair conformation. If the maximum deviations from the least-squares plane for the 'seat' sections of the various chain forms of the glucopyranosyl ring are calculated, it is evident that the form with C(2) at the foot shows least deviation (≤0.027 Å) (Table 4). This, coupled with a comparison of dihedral angles for the chair forms of various pyranose rings, suggests departure from the chair form toward a skew conformation.

The conformation of the primary alcohol group C(6)-O(6)

Table 3. Torsional Angles (°) for Maltitol

(a) within the pyranose ring		(b) outside the pyranose ring	
O(5)-C(1)-C(2)-C(3)	48.7(2)	O(5)-C(1)-C(2)-O(2)	174.7(3)
C(1)-C(2)-C(3)-C(4)	-45.2(3)	O(1)-C(1)-C(2)-C(3)	-73.7(3)
C(2)-C(3)-C(4)-C(5)	51.4(3)	O(1)-C(1)-C(2)-O(2)	52.3(2)
C(3)-C(4)-C(5)-O(5)	-59.9(2)	C(1)-C(2)-C(3)-O(3)	-167.3(3)
C(4)-C(5)-O(5)-C(1)	66.0(2)	O(2)-C(2)-C(3)-C(4)	-170.6(3)
C(5)-O(5)-C(1)-C(2)	-60.1(2)	O(2)-C(2)-C(3)-O(3)	67.3(3)
		C(2)-C(3)-C(4)-O(4)	171.8(3)
		O(3)-C(3)-C(4)-C(5)	172.1(3)
(c) 1→4 glycosidic linkage		O(3)-C(3)-C(4)-O(4)	-67.5(3)
		C(3)-C(4)-C(5)-C(6)	-178.8(3)
C(2)-C(1)-O(1)-C(4')	-165.3(3)	O(4)-C(4)-C(5)-O(5)	179.7(3)
O(5)-C(1)-O(1)-C(4')	72.5(2)	O(4)-C(4)-C(5)-C(6)	60.9(3)
C(1)-O(1)-C(4')-C(3')	94.2(3)	C(4)-C(5)-C(6)-O(6)	53.6(3)
C(1)-O(1)-C(4')-C(5')	-139.1(3)	O(5)-C(5)-C(6)-O(6)	-65.7(3)
		C(5)-O(5)-C(1)-O(1)	60.1(2)
		C(6)-C(5)-O(5)-C(1)	-170.4(3)
(d) glucitol moiety			
O(1')-C(1')-C(2')-C(3')	166.0(3)		
O(1')-C(1')-C(2')-O(2')	-70.8(3)		
C(1')-C(2')-C(3')-C(4')	178.4(3)		
C(1')-C(2')-C(3')-O(3')	58.0(2)		
O(2')-C(2')-C(3')-C(4')	57.7(3)		
O(2')-C(2')-C(3')-O(3')	-62.7(3)		
C(2')-C(3')-C(4')-C(5')	38.3(2)		
C(2')-C(3')-C(4')-O(1)	160.1(3)		
O(3')-C(3')-C(4')-C(5')	159.3(3)		
O(3')-C(3')-C(4')-O(1)	-78.9(2)		
C(3')-C(4')-C(5')-C(6')	79.4(3)		
C(3')-C(4')-C(5')-O(5')	-163.6(3)		
O(1)-C(4')-C(5')-C(6')	-42.9(2)		
O(1)-C(4')-C(5')-O(5')	74.1(2)		
C(4')-C(5')-C(6')-O(6')	-57.9(3)		
O(5')-C(5')-C(6')-O(6')	-178.1(3)		

Table 4. Least-Squares Planes with the Displacement (Å) of the Atoms from Best Planes in Maltitol. The * Atoms are Not Used to Define the Planes

(a) glucose ring					
$0.929x + 0.008y + 0.371z = 8.544$					
C(1)	.043	C(2)	-.041	C(4)	.041
C(3)*	-.591	O(5)*	.671	C(5)	-.043
$-0.857x + 0.486y - 0.169z = -1.525$					
C(2)	.074	C(3)	-.072	C(5)	.075
C(1)*	.656	C(4)*	-.680	O(5)	-.019
$0.987x - 0.069y - 0.148z = 5.307$					
C(3)	.026	C(4)	-.027	O(5)	.007
C(2)*	.577	C(5)*	-.737	C(1)	-.027
(b) glucitol residue					
$0.731x + 0.298y + 0.614z = 11.582$					
C(1')	.011	C(2')	-.009	C(3')	-.010
O(1)*	-.458	C(5)*	-.830	O(1)*	-.306
$0.301x - 0.906y + 0.297z = -9.335$					
C(4')	.000	C(5')	.000	C(6')	.000
O(1)*	.934	C(3)*	-1.356	O(6)*	-1.117

Table 5. Torsional and Bridge Angles ($^{\circ}$) about the Glycosidic bonds with (1 \rightarrow 4) Linkage

	H(C1)-C(1) -O(1)-C(4')	C(1)-O(1) -C(4')-H(C4')	C(1)-O(1)-C(4')
maltitol	-45	-24	117.1
α -maltose[4]	-5	2	120.1
β -maltose[5]	3	11	117.1
methyl β -maltoside[6]	-8	9	117.6
phenyl α -maltoside[7]			
molecule A	-10	-20	116.5
molecule B	-10	-20	116.8
β -maltose octaacetate[8]	-29	-36	117.5

Table 6. Torsion angles ($^{\circ}$) for the Glucitol Moiety of Maltitol with Corresponding values for D-glucitol Compounds

	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	<i>e</i>
O(1')-C(1')-C(2')-C(3')	166.0	176.1	-173.9	-58.7	-70.4
C(1')-C(2')-C(3')-C(4')	178.4	-69.7	-52.4	-69.5	-170.6
C(2')-C(3')-C(4')-C(5')	38.3	172.7	-178.1	161.3	173.5
C(3')-C(4')-C(5')-C(6')	79.4	74.4	-179.8	178.6	-180.0
C(4')-C(5')-C(6')-O(6')	-57.9	-177.2	-174.1	70.6	57.2

^amaltitol(4-O- α -D-glucopyranosyl-D-glucitol) [this study]. ^b4-O- β -D-glucopyranosyl-D-glucitol [14]. ^cD-glucitol [12]. ^dglucitol-pyridine [13]. ^eD-glucitol hexaacetate [15].

has gauche-gauche orientation.

The relative orientation of pyranose and glucitol is customarily described by the torsion angles around the glycosidic bonds, C(1)-O(1) and O(1)-C(4'). The values of these torsional angles are H(C1)-C(1)-O(1)-C(4') = -45° and C(1)-O(1)-C(4')-H(C4') = -24° . Another important parameter is the valence angle C(1)-O(1)-C(4') which is equal to 117.1° . The torsional and bridge angles about the glycosidic bonds, as found in the already solved structures of maltose related compounds with (1 \rightarrow 4) linkage, are compared in Table 5. The greatest difference is in the conformation about the glycosidic bonds, resulting from difference in the intramolecular hydrogen bonds. In maltitol, there is no intramolecular hydrogen bond between glucose and glucitol moieties, whereas in the other compounds, the intramolecular hydrogen bonds exist between two glucoses.

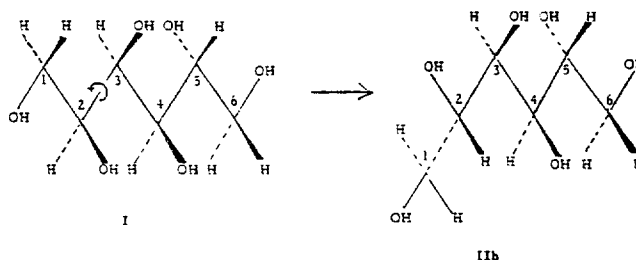
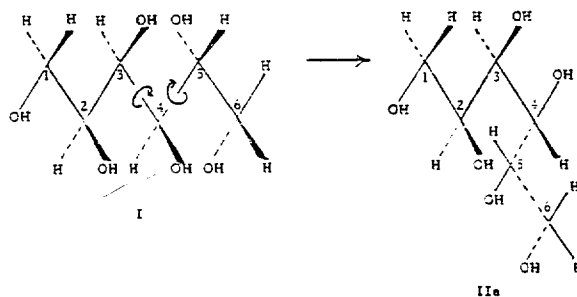
The glucitol residue has a non-planar carbon-chain conformation (Tables 3 and 6). On the basis of the hypothesis that parallel C(*n*)-OH [or C(*n*)-C] and C(*n*+2)-OH [or C(*n*+2)-C] bonds in the alditols give rise to conformation instability, Jeffrey and Kim have predicted that there should be two low energy non-planar carbon-chain conformations for D-glucitol. The parallel C(2')-OH to C(4')-OH interaction in the straight chain conformation shown in I is avoided by a rotation 120° about the C(2')-C(3') or C(3')-C(4') bond; this gives rise to the two bent chain conformations shown in IIa and IIb. The 120° rotation about C(3')-C(4') brings C(5')-OH and C(2')-C(3') into parallel alignment, requiring a further rotation of 120° about C(4')-C(5') to produce the conforma-

Table 7. Hydrogen bond Distances and Angles in Maltitol

D	H	A	D-H(\AA)	H...A(\AA)	D...A(\AA)	Angle($^{\circ}$)
O(1')-H	...	O(2')	0.78(5)	2.28(5)	2.968(3)	148(5)
O(2)-H	...	O(1') ^a	0.77(4)	2.16(5)	2.910(3)	165(4)
O(3)-H	...	O(6') ^a	0.78(4)	2.03(4)	2.775(4)	161(4)
O(4)-H	...	O(5') ^b	0.83(4)	1.89(4)	2.720(4)	175(4)
O(6)-H	...	O(4) ^b	0.86(4)	1.92(5)	2.701(4)	151(4)
O(6')-H	...	O(1') ^a	0.79(4)	2.15(4)	2.936(3)	171(4)

Symmetry code. ^a-*x* + 1, *y* - 0.5, -*z* + 0.5. ^b*x* + 0.5, -*y* + 1.5, -*z* + 1.

tion, IIa, in which C(1') to C(4') are coplanar and C(4'), C(5') and C(6') are coplanar in a different plane. Of the two bent chain conformations IIa is found in this study. The simpler conformation is observed in the A form of D-glucitol¹² and in the D-glucitol-pyridine complex¹³. However, the conformation of the glucitol residue of 4-O- β -D-glucosyl-D-glucitol¹⁴ and glucitol hexaacetate¹⁵ are different from the above



three compounds. In 4-O- β -D-glucosyl-D-glucitol, the glucitol residue has a non-planar carbon-chain conformation which is derived from the planar extended chain by a rotation of 120° about C(2')-C(3') and C(4')-C(5'), and which is unstable because of the almost parallel orientation of the C(5')-C(6') and C(3')-O(3') bonds. In D-glucitol hexaacetate, the conformation of carbon chain is zigzag planar mainly due to the six acetyl groups.

The terminal hydroxyl groups of the glucitol residue have trans-gauche conformation, whereas in D-glucitol and 4-O- β -D-glucosyl-D-glucitol these conformations are the same extended trans-trans. In D-glucitol-pyridine and D-glucitol hexaacetate, they are both bent. Clearly, environmental effects in the crystal not only determine the conformation of the terminal hydroxyl groups, but also strongly influence the carbon-chain conformation.

Molecular Packing. The molecular packing in the crystal appears to be determined mainly by hydrogen bonds, since there are six hydrogen bonds per asymmetric unit. The hydrogen bond distances and angles are listed in Table 7 and

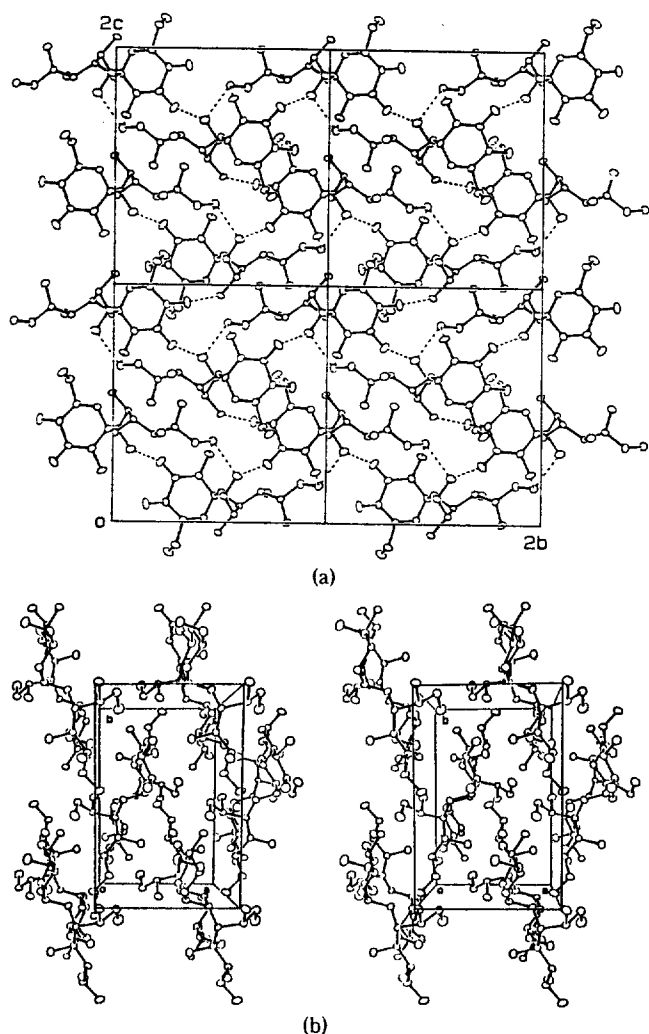


Figure 2. Molecular packing in Maltitol. (a) viewed down the *a* axis. The dotted lines are indicated the hydrogen bonds. (b) viewed down the *c* axis.

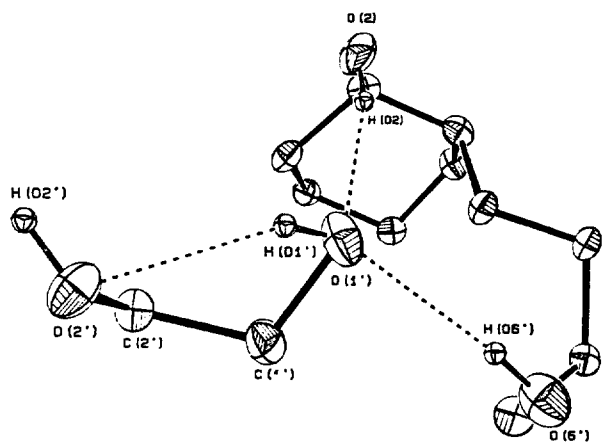
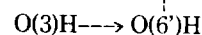


Figure 3. Nearest neighbor coordination around O(1') in Maltitol.

molecular packing is illustrated in Figure 2.

Among the nine hydroxyl groups only six hydroxyls act as hydrogen-bond donors. Three hydroxyl groups, O(4)-H, O(1')-H and O(6')-H are both donors and acceptors, whereas O(3')-H, O(4')-H are not involved in any hydrogen bond and O(5')-H takes part in one hydrogen bond as a acceptor. O(1')-H is double acceptors from O(2)-H and O(6')-H all within the same molecule and also an intramolecular hydrogen bond donor to O(2') (see Figure 3). The ring O(5) and glycosidic O(1) do not accept any hydrogen bond.

Each molecule is linked through two kinds of hydrogen bonds, a short donor-acceptor chain O(6)H→O(4)H→O(5') and a branched hydrogen bond chain O(2)H→O(1')H→O(2').



The intramolecular hydrogen bond O(1')-H...O(2'), with H...O 2.28 Å and ∠O-H...O 148° should be considered as a weak interaction.

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