

## The Crystal and Molecular Structure of $\alpha$ -Maltose\*

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The crystal structure of  $\alpha$ -maltose [4-*O*-( $\alpha$ -D-glucopyranosyl)- $\alpha$ -D-glucopyranose] has been determined by single-crystal X-ray techniques. The unit cell is orthorhombic, space group  $P2_12_12_1$ , with dimensions  $a = 12.667$  (3),  $b = 13.830$  (5) and  $c = 8.400$  (2) Å. The structure was solved using a direct method and refined by full-matrix least-squares procedures. Difference syntheses showed all H atoms and also indicated a partial (18%) random substitution of  $\alpha$ -maltose molecules by the  $\beta$  anomer. The final discrepancy factor,  $R$ , is 0.043 for 1379 observed reflections. Bond lengths and angles are in good agreement with those of other disaccharides. The pyranosyl rings are slightly distorted from the C1 chair form and the O(1)–O(4) distance of 4.052 Å is shorter than found previously. The primary alcohol groups have *gauche*–*trans* orientations. All the O atoms in both the  $\alpha$  and  $\beta$  anomers, except the bridge O atoms, take part in a hydrogen-bonding network.

### Introduction

In connection with X-ray diffraction studies of selected carbohydrates in this laboratory (James, French & Rundle, 1959; Hybl, Rundle & Williams, 1965; Jacobson, Wunderlich & Lipscomb, 1961; Hackert & Jacobson, 1971), we have determined the crystal and molecular structure of  $\alpha$ -maltose. Maltose is obtained, along with other products, from the partial hydrolysis of starch in aqueous acidic solution. Maltose is also formed in the fermentation of starch to ethyl alcohol, when catalyzed by the enzyme diastase, which is present in malt. The importance of the molecular conformations of carbohydrates has long been recognized, and therefore a number of crystal investigations of monosaccharides, disaccharides, polysaccharides and their derivatives have been carried out using X-ray and neutron diffraction techniques. Just as the molecular structure of cellobiose is important in order to better understand the properties and reactions of cellulose, the molecular conformation of maltose is important in basic studies of starch. The crystal structures of  $\beta$ -maltose monohydrate (Quigley, Sarko & Marchessault, 1970), methyl  $\beta$ -maltopyranoside monohydrate (Chu & Jeffrey, 1967), and isomaltose monohydrate (Dreissig & Luger, 1973) have been determined by other workers. We decided to carry out a molecular structure investigation of  $\alpha$ -maltose in order to elucidate its molecular conformation and to compare the  $\alpha$  and  $\beta$  forms and the corresponding hydrogen bonding in these solids.

### Experimental

#### Crystal data

$C_{12}H_{22}O_{11}$ ,  $M_r = 342.3$ , orthorhombic,  $P2_12_12_1$ ,  $a = 12.667$  (3),  $b = 13.830$  (5),  $c = 8.400$  (2) Å,  $V = 1471.5$  (7) Å<sup>3</sup>,  $D_c = 1.546$  g cm<sup>-3</sup>,  $Z = 4$ ,  $\mu = 1.65$  cm<sup>-1</sup> for Mo  $K\alpha$  radiation ( $\lambda = 0.71069$  Å), m.p. 185–187°C.

Crystalline samples of  $\alpha$ -maltose containing approximately 20% of the  $\beta$  anomer and whose preparation has been described by Hodge, Rendleman & Nelson (1972) were kindly supplied by J. E. Hodge and D. French. The unit-cell parameters and their estimated standard deviations were obtained by a least-squares fit to the  $\pm 2\theta$  values of 12 independent high-angle reflections.

For data collection a crystal in the shape of a parallelepiped formed with the (100), (010) and (001) planes as faces and of dimensions 0.3 × 0.3 × 0.5 mm was selected. Data were collected at room temperature using an automated four-circle diffractometer designed and built in the Ames Laboratory and previously described by Rohrbaugh & Jacobson (1974). For data collection, reflected-beam graphite-monochromated radiation was used ( $\lambda = 0.70954$  Å). Using eleven reflections selected from oscillation pictures taken on the diffractometer, our automatic indexing program (Jacobson, 1976) indicated an orthorhombic cell which was subsequently verified. Nine high-angle reflections were selected for standards, were remeasured every 75 reflections, and were not observed to vary throughout the entire period of data collection. Within a  $2\theta$  sphere of 50° ( $\sin \theta/\lambda = 0.5986$  Å<sup>-1</sup>), all data in the  $hkl$  and  $hkl$  octants were measured using an  $\omega$ -scan method with

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backgrounds measured by offsetting  $0.5^\circ$  in  $\omega$ . Examination of the 2851 reflections recorded in this manner revealed systematic absences for the  $h00$ ,  $0k0$  and  $00l$  reflections when  $h$ ,  $k$  or  $l$  was odd, respectively. These absences uniquely determined the space group as  $P2_12_12_1$ .

The intensity data were corrected for polarization\* and Lorentz effects, and the maximum and minimum transmission factors were estimated to be 0.952 and 0.921; hence an absorption correction was not deemed necessary. The estimated variance in each intensity was calculated by  $\sigma_i^2 = C_i + 2C_b + (0.03C_i)^2 + (0.03C_b)^2$ , where  $C_i$  and  $C_b$  represent the total and background counts, respectively, and the factor 0.03 represents an estimate of non-statistical errors. The estimated standard deviations in the structure factor amplitudes were calculated by the finite-difference method (Lawton & Jacobson, 1968). Equivalent data were averaged and 1379 reflections with  $|F_o| \geq 3\sigma_{F_o}$  were retained for use in subsequent calculations. The discrepancy factor between equivalent data was 0.039 and was computed via  $\Sigma \langle ||F_i| - \bar{F}| \rangle_i / \Sigma F$ , where  $i$  denotes the symmetry-equivalent reflections and  $F$  is the average of the corresponding structure factor magnitudes. In this calculation ten weak reflections with individual discrepancy factors  $>0.2$  were removed from the data set.

#### Structure determination and refinement

The crystal and molecular structure of  $\alpha$ -maltose was solved using a modification of the multi-solution direct method described by Woolfson & Germain (1968). The positions of all non-hydrogen atoms were easily located from an  $E$  map calculated using the phases from the most consistent set.

Three cycles of isotropic full-matrix least-squares refinement (Busing, Martin & Levy, 1962), followed by two cycles of anisotropic refinement yielded a crystallographic discrepancy factor,  $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$ , of 0.07. A difference electron density map (Hubbard,

Quicksall & Jacobson, 1971) revealed all the H atom positions. The difference map also indicated that the electron densities of the H atoms attached to C atoms were always larger by about  $0.15 \text{ e } \text{\AA}^{-3}$  than those attached to O atoms. Therefore isotropic temperature factors of 3.5 and  $5.0 \text{ \AA}^2$ , respectively, were assigned to the H atoms.

A second difference map was computed after two cycles of anisotropic least-squares refinement in which the H atoms were included, but only their positional parameters were refined. This map revealed residual electron density ( $1.4 \text{ e } \text{\AA}^{-3}$ ) near the position assigned to the H attached to C(1'). The peak was located approximately  $1.30 \text{ \AA}$  from C(1') and formed a torsional angle with C(1'), C(2'), and O(2') of  $-62.2^\circ$ ,

Table 1. *Positional parameters of the non-hydrogen atoms* ( $\times 10^4$ )

In this and succeeding tables, estimated standard deviations are given in parentheses for the least significant figures.

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	401 (2)	8617 (2)	8417 (4)
C(2)	124 (3)	9245 (3)	7007 (4)
C(3)	925 (3)	10050 (3)	6824 (4)
C(4)	1116 (3)	10593 (3)	8371 (4)
C(5)	1203 (2)	9932 (2)	9815 (4)
C(6)	1060 (3)	10496 (3)	11329 (4)
O(1)	1422 (2)	8237 (1)	8141 (3)
O(2)	110 (2)	8658 (2)	5615 (3)
O(3)	591 (3)	10743 (3)	5671 (3)
O(4)	2087 (2)	11101 (2)	8307 (3)
O(5)	409 (2)	9194 (2)	9816 (3)
O(6)	1053 (2)	9905 (2)	12722 (3)
C(1')	3305 (3)	5863 (2)	7931 (4)
C(2')	2518 (3)	5941 (2)	6606 (4)
C(3')	2045 (3)	6943 (2)	6573 (4)
C(4')	1596 (2)	7211 (2)	8195 (4)
C(5')	2379 (3)	7015 (2)	9544 (4)
C(6')	1864 (3)	7086 (3)	11160 (4)
O(1')	4132 (2)	6483 (2)	7645 (4)
O(2')	3011 (2)	5746 (2)	5113 (3)
O(3')	1262 (2)	6963 (2)	5358 (3)
O(5')	2778 (2)	6049 (2)	9409 (3)
O(6')	2665 (2)	7042 (2)	12359 (3)
O(1'')	3696 (12)	5001 (10)	8051 (19)

\* Via  $(\cos^2 2\theta_M + \cos^2 2\theta) / (\cos^2 2\theta_M + 1)$ .

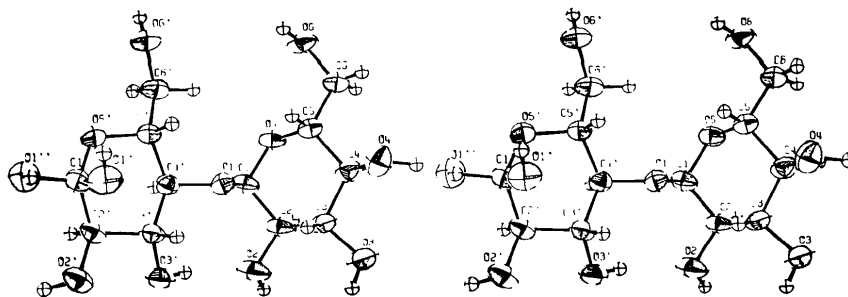


Fig. 1. A stereoscopic view of  $\alpha$ -maltose involving the  $\beta$  anomer. For clarity H atoms are depicted with reduced thermal parameters. In this and succeeding drawings 50% probability ellipsoids are depicted.

Table 2. *Positional and isotropic thermal parameters of the hydrogen atoms ( $\times 10^3$ )*

	x	y	z	$B$ ( $\text{\AA}^2$ )*	Distance ( $\text{\AA}$ )		x	y	z	$B$ ( $\text{\AA}^2$ )*	Distance ( $\text{\AA}$ )
H(C1)	-11 (3)	804 (3)	856 (6)	3.5	1.04 (8)	H(C1')	376 (-)*	522 (-)*	797 (-)*	3.5	1.18 (-)
H(C2)	-62 (3)	946 (3)	736 (3)	3.5	1.08 (5)	H(C2')	191 (3)	541 (3)	682 (6)	3.5	1.08 (5)
H(C3)	165 (3)	975 (3)	656 (6)	3.5	1.03 (5)	H(C3')	267 (3)	748 (3)	631 (6)	3.5	1.09 (5)
H(C4)	47 (4)	1119 (3)	863 (6)	3.5	1.22 (5)	H(C4')	91 (3)	689 (3)	836 (6)	3.5	1.00 (5)
H(C5)	189 (4)	961 (3)	991 (6)	3.5	0.99 (5)	H(C5')	306 (4)	751 (3)	951 (5)	3.5	1.11 (5)
H1(C6)	164 (3)	1096 (3)	1133 (6)	3.5	0.97 (5)	H1(C6')	145 (3)	661 (3)	1133 (6)	3.5	0.86 (5)
H2(C6)	33 (3)	1090 (3)	1137 (6)	3.5	1.09 (5)	H2(C6')	142 (3)	779 (3)	1130 (6)	3.5	1.09 (5)
H(O2)	-54 (4)	877 (3)	510 (6)	5.0	0.95 (6)	H(O1')	460 (4)	654 (3)	826 (6)	5.0	0.71 (8)
H(O3)	89 (4)	1047 (4)	476 (6)	5.0	0.99 (6)	H(O2')	289 (4)	517 (3)	491 (6)	5.0	0.87 (6)
H(O4)	200 (4)	1171 (4)	809 (6)	5.0	0.93 (6)	H(O3')	94 (4)	738 (4)	553 (6)	5.0	0.76 (6)
H(O6)	179 (4)	956 (4)	1294 (7)	5.0	1.05 (6)	H(O6')	234 (4)	688 (4)	1327 (6)	5.0	0.92 (5)
						H(C1'')	394 (-)*	634 (-)*	771 (-)*	3.5	1.06 (-)

\* These parameters were fixed during refinement. The occupancy factors of H(C1'), H(O1') and H(C1'') are 0.85, 0.85 and 0.15, respectively.

and therefore seemed very plausible as evidence for the presence of some  $\beta$  anomer, *i.e.* the O in the equatorial position. Therefore, in the next least-squares cycle, O(1'') of the  $\beta$  anomer was added and the occupancy parameters of this oxygen and O(1') of the  $\alpha$  anomer

were refined independently. Also the H atoms appropriate for the  $\alpha$  and  $\beta$  anomers were assigned occupancy factors of 0.85 and 0.15 and were fixed at 1.06  $\text{\AA}$  from C(1'). This refinement yielded occupancy parameters of  $0.820 \pm 0.008$  and  $0.173 \pm 0.007$  for O(1') and O(1''), respectively, hence indicating the presence of approximately 18%  $\beta$  anomer in the crystal. These values are in good agreement with the  $\alpha$ : $\beta$  value of 80:20 as inferred from optical rotatory measurements (Hodge, Rendleman & Nelson, 1972).

Finally, three cycles of anisotropic refinement on the heavy-atom parameters, followed by two cycles of refinement on the H atoms and O(1'') reduced the discrepancy factor to 0.043 and the corresponding weighted discrepancy factor to 0.060. The function refined was  $\sum \omega(|F_o| - |F_c|)^2$ , where  $\omega = 1/\sigma_{F_o}^2$ .

The final atomic positional parameters are listed in Tables 1 and 2. The atomic scattering factors used for C and O were those of Hanson, Herman, Lea & Skillman (1960), and for H, those of Stewart, Davidson & Simpson (1965).\*

## Discussion

A stereoscopic view of the molecule as obtained from the ORTEP program (Johnson, 1971) and including the  $\beta$  anomer is shown in Fig. 1. The bond lengths involving the non-hydrogen atoms are given in Fig. 2(a). The C—C distances found in the unprimed ring range from 1.511 to 1.524  $\text{\AA}$ , with an average value of 1.518  $\text{\AA}$ , while those found in the primed ring range from 1.498 to 1.530  $\text{\AA}$ , with an average value of 1.515  $\text{\AA}$ . The exocyclic C—C bond lengths are significantly shorter

\* Lists of structure factors, anisotropic thermal parameters, and dihedral angles of chair forms of some disaccharides have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32919 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

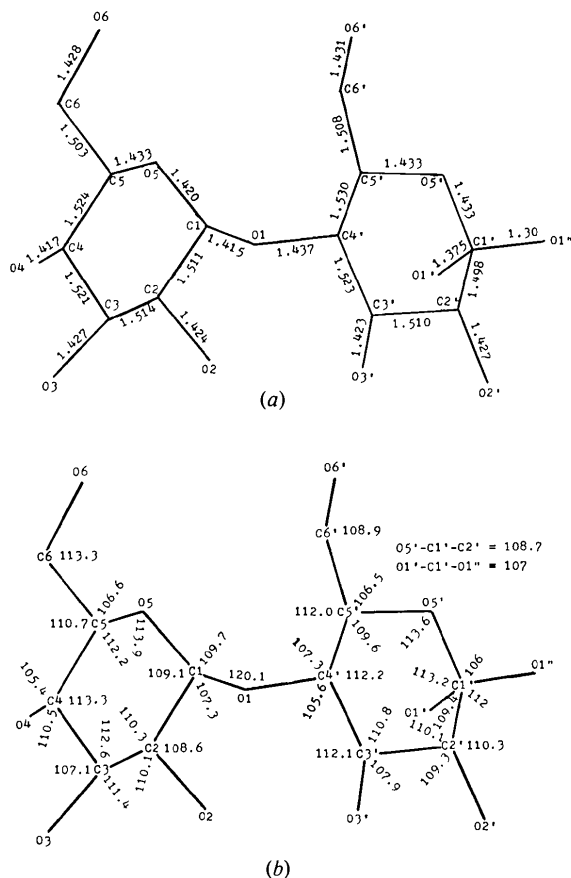


Fig. 2. (a) Bond lengths ( $\text{\AA}$ ) and (b) bond angles ( $^\circ$ ).

than those in the ring in agreement with the observation of Ham & Williams (1970). The exocyclic C—O distances excluding the anomeric and bridge C—O bonds range from 1.424 to 1.427 Å, averaging 1.425 Å. The C(1')—C(2') distance in  $\alpha$ -maltose appears to be slightly shorter ( $4\sigma$ ) than the corresponding distances in the other accurately determined disaccharides (Arnott & Scott, 1972). The C(1)—O(5) distance in the unprimed ring (1.420 Å) is found to be  $3\sigma$  shorter than the C(5)—O(5) distance (1.433 Å). In the primed ring the corresponding lengths are equal (1.433 Å). A similar trend was observed in methyl  $\beta$ -maltopyranoside (Chu & Jeffrey, 1967),  $\beta$ -lactose (Hirotsu & Shimada, 1974), and  $\beta$ -cellobiose (Chu & Jeffrey, 1968). However, in methyl  $\beta$ -cellobioside (Ham & Williams, 1970), these distances are nearly equal ( $<1\sigma$ ) in both rings. In  $\alpha$ -lactose (Fries, Rao & Sundaralingam, 1971), the same pattern of C(1)—O(5)  $<$  C(5)—O(5) is found for the unprimed ring, while the reverse trend C(1')—O(5')  $>$  C(5')—O(5'), is found in the primed ring. In both rings of  $\alpha,\alpha$ -trehalose (Brown, Rohrer, Berking, Beevers, Gould & Simpson, 1972), the distance is found to be somewhat shorter ( $>3\sigma$ ) than the C(5)—O(5) distance. If the anomeric effect is to play a very important role, one would expect that  $\alpha$ -maltose would show the same trends as  $\alpha$ -lactose, with C(1)—O(5)  $<$  C(5)—O(5) and C(1')—O(5')  $>$  C(5')—O(5'). However, our results for  $\alpha$ -maltose indicate a greater similarity to the  $\beta$ -form disaccharides. Admittedly, the presence of a small amount (18%) of the  $\beta$ -anomer in  $\alpha$ -maltose complicates such comparisons. The C(4')—O(1) bridge bond is consistently significantly ( $10\sigma$ ) longer than C(1)—O(1). There is also a significant ( $10\sigma$ ) difference between the C(1)—O(1) and C(1)—O(1') distances, the latter being shortened and in good agreement with previous observations by Berman, Chu & Jeffrey (1967).

The bond angles involving non-hydrogen atoms are given in Fig. 2(b). The differences between the corresponding angles in the primed and unprimed ring are not significant (less than  $2\sigma$ ) with the exception of those between C(4)—C(5)—O(5) and C(4')—C(5')—O(5') ( $3.5\sigma$ ), and between C(2)—C(3)—C(4) and C(2')—C(3')—C(4') ( $9\sigma$ ). Aside from the O(5)—C(1)—C(2) angle, all internal ring angles involving O atoms are significantly larger ( $15\sigma$ ) than the tetrahedral value, the C—O—C ring angles being among the largest. Similar trends have been observed in  $\alpha$ -lactose,  $\beta$ -lactose and  $\beta$ -cellobiose, but not in the primed ring of methyl  $\beta$ -cellobioside and methyl  $\beta$ -maltopyranoside. The exocyclic angles range from 105.4 to 113.2°, and, as has been observed in many other disaccharides, the differences between the corresponding angles in both glucopyranosyl groups are larger than one would expect from their e.s.d.'s. It appears that intermolecular forces, especially those arising from hydrogen-bonding effects, strongly affect these angles in contrast to the internal ring angles. The angle in the bridge, C(1)—O(1)—C(4'),

of 120.1(2)° is slightly larger than the corresponding angles in  $\alpha$ -lactose (117.1),  $\beta$ -lactose (116.5),  $\beta$ -cellobiose (116.1), methyl  $\beta$ -cellobioside (115.8) and methyl  $\beta$ -maltopyranoside (117.6°).

The torsional angles of the molecule are given in Table 3. If the maximum deviations from the least-squares plane for the 'seat' sections of the various chair forms of the glucopyranosyl rings are calculated,\* it is evident that the forms with C(1) or C(1') at the foot show least deviation ( $\leq 0.011$  Å). The ring conformational angles are as follows: C(3)→C(4), 42.5°; C(4)→C(5), -43.9°; C(3')→C(4'), 49.3°; and C(4)→C(5'), -50.5°. This, coupled with a comparison of dihedral angles for the chair forms of various disaccharides,\* suggests departure from the chair form toward a skew conformation consistent with that proposed by Bentley (1959). Such a distortion has not been observed in  $\beta$ -maltose or methyl  $\beta$ -maltopyranoside. The O(1)—O(4) distance in  $\alpha$ -maltose is especially short (4.052 Å), and indicates that a more extended range of ring con-

\* See deposition footnote on p. 215.

Table 3. Torsional angles (°) involving the non-hydrogen atoms

Within the pyranose ring

O(5)—C(1)—C(2)—C(3)	59.7	O(5')—C(1')—C(2')—C(3')	60.1
C(1)—C(2)—C(3)—C(4)	-50.1	C(1')—C(2')—C(3')—C(4')	-53.6
C(2)—C(3)—C(4)—C(5)	42.5	C(2')—C(3')—C(4')—C(5')	49.1
C(3)—C(4)—C(5)—O(5)	-43.9	C(3')—C(4')—C(5')—O(5')	-50.4
C(4)—C(5)—O(5)—C(1)	55.9	C(4')—C(5')—O(5')—C(1')	59.2
C(5)—O(5)—C(1)—C(2)	-63.9	C(5')—O(5')—C(1')—C(2')	-64.7

Outside the pyranose ring

C(4')—O(1)—C(1)—C(2)	-125.5	C(1)—O(1)—C(4')—C(3')	122.2
C(4')—O(1)—C(1)—O(5)	116.1	C(1)—O(1)—C(4')—C(5')	-118.0
O(6)—C(6)—C(5)—C(4)	175.6	O(6')—C(6')—C(5')—C(4')	-171.3
O(6)—C(6)—C(5)—O(5)	53.3	O(6')—C(6')—C(5')—O(5')	68.9

Table 4. Torsional angles (°) about bridge bonds of disaccharides with ( $\alpha$ -1,4) linkage

A =  $\alpha$ -maltose (this study).

B =  $\beta$ -maltose (Quigley, Sarko & Marchessault, 1970).

C = methyl  $\beta$ -maltopyranoside (Chu & Jeffrey, 1967).

$\psi_1, \psi_1', \psi_2, \psi_2', \psi, \psi'$  are defined according to Sundaralingam (1968).

		A	B	C
$\psi_1$	O(5)—C(1)—O(1)—C(4')	116.1	123.1	109.9
$\psi_2$	C(2)—C(1)—O(1)—C(4')	-125.5	-116.6	-128.2
$\psi_1'$	C(1)—O(1)—C(4')—C(3')	122.2	132.4	129.2
$\psi_2'$	C(1)—O(1)—C(4')—C(5')	-118.0	-109.7	-108.9
$\psi$	O(4)—C(1)—O(1)—C(4')	172.1	173.3	169.3
$\psi'$	O(1')—C(4')—O(1)—C(1)	-177.6	-164.5	-165.7
	O...O*	2.770 Å	2.79 Å	2.825 Å

\* Intrahydrogen bonding distances. O(2)...O(3').

formation angles C(2)–C(3)–C(4)–C(5) and C(3)–C(4)–C(5)–O(5) is possible for  $\alpha$  residues than had been assumed by French & Murphy (1973) in their discussion of the effects of changes in ring geometry on computer models of amylose.

Selected torsional angles in  $\alpha$ -maltose,  $\beta$ -maltose and methyl  $\beta$ -maltopyranoside are given in Table 4. The torsional angles O(4)–C(1)–O(1)–C(4') and O(1')–C(4')–O(1)–C(1) in this study are 172.1 and  $-177.6^\circ$  respectively. These small twists in the 'backbone' of the molecule are such as to favor intramolecular hydrogen bonding between O(2) and O(3'). The corresponding angles in methyl  $\beta$ -maltopyranoside are 169.3 and  $-165.7^\circ$ . Despite these larger angles, the hydrogen-bonding distance between O(2) and O(3') is longer in the latter compound than in  $\alpha$ -maltose (Table 5).

Table 5. Hydrogen-bonding distances (Å) and angles ( $^\circ$ )

	O...O	H...O	$\angle$ O–H...O
O(3')–H(O3')...O(2)–C(2) <sup>a</sup>	2.770 (4)	2.06 (5)	169 (5)
...O(2')–C(2') <sup>b</sup>	2.959 (4)	2.36 (5)	124 (4)
O(6')–H(O6')			
...O(3')–C(3') <sup>b</sup>	3.085 (4)	2.23 (5)	160 (6)
O(3)–H(O3)...O(6)–C(6) <sup>c</sup>	2.797 (4)	1.89 (5)	162 (5)
O(2')–H(O2')...O(5')–C(1') <sup>d</sup>	2.741 (4)	1.93 (4)	165 (5)
O(6)–H(O6)...O(4)–C(4) <sup>e</sup>	2.780 (4)	1.72 (5)	174 (5)
O(4)–H(O4)...O(6')–C(6') <sup>f</sup>	2.707 (4)	1.88 (6)	158 (5)
O(2)–H(O2)...O(2')–C(2') <sup>g</sup>	2.850 (4)	1.96 (5)	156 (4)
O(1')–H(O1')...O(5)–C(1) <sup>h</sup>	2.836 (4)	2.17 (5)	143 (4)
O(1'')...O(5)–C(1) <sup>h</sup>	3.03 (2)	—	—

Symmetry code

(a) $x, y, z$	(e) $-x + 0.5, -y + 2.0, z + 0.5$
(b) $x, y, z + 1.0$	(f) $-x + 0.5, -y + 2.0, z - 0.5$
(c) $x, y, z - 1.0$	(g) $x - 0.5, -y + 1.5, -z + 1.0$
(d) $-x + 0.5, -y + 1.0, z - 0.5$	(h) $x + 0.5, -y + 1.5, -z + 2.0$

The conformational angles of the primary alcohol groups, C(6)–O(6) and C(6')–O(6'), have *gauche-trans* (–synclinal) orientations. In  $\beta$ -maltose and methyl  $\beta$ -maltopyranoside, the  $\alpha$ -D-glucose residue has a *gauche-trans*, while the  $\beta$ -D-glucose residue has a *gauche-gauche* (+synclinal) orientation.

The molecular packing in the crystal appears to be determined mainly by hydrogen-bonding effects, since there are nine hydrogen bonds per asymmetric unit. Hydrogen-bonding distances and angles are listed in Table 5. Fig. 3 is a stereoview showing the hydrogen bonding. Each oxygen, O(2), O(4), O(6) and O(3'), is associated with two hydrogen bonds, one as a donor and one as an acceptor. O(3) and O(1') [or O(1'')] each take part in one hydrogen bond as a donor, while O(2') and O(6') participate in three hydrogen bonds, one as donor and two as acceptors, and one as acceptor and two as donors, respectively. Each of the ring oxygen atoms, O(5) and O(5'), acts as a hydrogen-bond acceptor. The H(O1')...O(5) and the H(O2')...C(5') vectors make angles of 18 and  $33^\circ$ , respectively, with the C(1), O(5) and C(5) type of plane. As can be seen, some of the angles deviate significantly from half of the tetrahedral value. The ring C–O–C angles are also much larger than the tetrahedral value, as discussed earlier. These anomalies suggest that the distributions of lone-pair electrons on O(5) and O(5') are distorted from ideal  $sp^3$  hybridization.

Each molecule is linked through five kinds of hydrogen bonds, O(2)–H(O2)...O(2'), O(1')–H(O1')...O(5), O(3)–H(O3)...O(6), O(6)–H(O6)...O(2') and O(6)–H(O6)...O(3'), to form sheets parallel to the (010) plane. Hydrogen bonds between the sheets are arranged in a helical fashion; the helices at  $x = \frac{1}{4}, y = 0$  and  $x = \frac{1}{4}, y = \frac{1}{2}$  are left-handed (donor to acceptor) and right-handed respectively.

The difference between the hydrogen-bonding scheme in the  $\alpha$  and  $\beta$  anomers is mainly the difference between the angles which the H(O1')...O(5) and H(O1'')...O(5) vectors of the hydrogen bonds make with the plane of the C(1), O(5) and C(5) atoms. The former angle is  $-9.3^\circ$  while the latter is  $+26.3^\circ$ . Anhydrous crystalline  $\beta$ -D-maltose, if it exists, might well be isomorphous with the  $\alpha$  anomer.

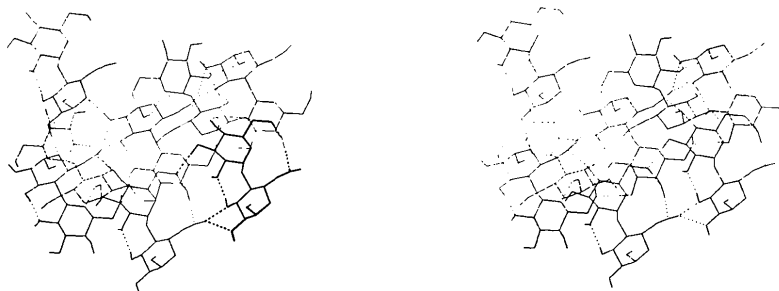


Fig. 3. A stereoscopic view down the  $a$  axis illustrating the hydrogen bonding in  $\alpha$ -maltose.

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## Structure Cristalline du Succinylsulfathiazol Monohydraté

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Crystals of sulfasuxidine, *p*-[*N*-(1,3-thiazol-2-ylidene)sulphamoyl]succinanic acid monohydrate, (SC<sub>3</sub>NH<sub>3</sub>)·NSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NHCOCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H·H<sub>2</sub>O, are monoclinic, space group *P*2<sub>1</sub>/*b*, *a* = 16.28 (1), *b* = 19.49 (1), *c* = 5.143 (4) Å,  $\gamma$  = 94.10 (4)°, *Z* = 4. The intensities were measured using Mo *K* $\alpha$  radiation and an automatic diffractometer. The structure was solved by the symbolic addition procedure and refined by least-squares methods to *R* = 0.038. Neighbouring molecules are hydrogen bonded.

### Introduction

Le produit étudié est issu de la recristallisation du succinylsulfathiazol monohydraté du commerce dans l'acétone à 37°C. Le monocristal choisi pour réaliser l'étude structurale peut, en première approximation, être assimilé à un parallélépipède; il mesure environ

0,45 × 0,20 × 0,10 mm. Les spectres d'oscillation et de Weissenberg révèlent une maille monoclinique de dimensions *a* = 16,28 (1), *b* = 19,49 (1), *c* = 5,143 (4) Å,  $\gamma$  = 94,10 (4)°. En admettant la présence de quatre molécules monohydratées par maille, la masse volumique calculée ( $\mu_c$  = 1,52 g cm<sup>-3</sup>) est très voisine de la masse volumique mesurée par McCrone & Corvin