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A Neutron Diffraction Refinement of the Crystal Structure of Methyl α-D-Altropyranoside*

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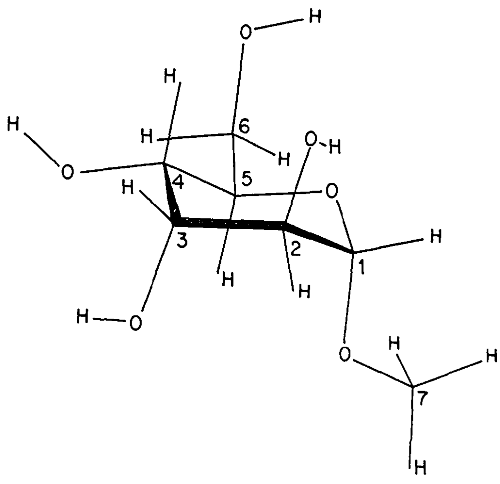
Neutron-diffraction data have been used to refine the crystal structure of methyl α-D-altropyranoside, $C_7H_{14}O_6$, $P2_12_12_1$, $Z=4$, $a=7{\cdot}486$, $b=9{\cdot}098$, $c=13{\cdot}330$ Å. The vicinal intramolecular hydrogen bond and the bifurcated hydrogen bonding reported from the X-ray analysis are confirmed. The H···O distances for the bifurcated hydrogen bonds range from 2·085 (6) to 2·185 (7) Å, compared with 1·736 (7) and 1·922 (7) Å for the normal hydrogen bonds. The C–O distances from the neutron data appear to be systematically shorter than the X-ray values by 0·007 Å (2σ). The pyranose ring is distorted $^4C_1$, with ring torsion angles ranging from 45° to 63°.

Introduction

The crystal structure of methyl α-D-altropyranoside, $C_7H_{14}O_6$, was determined by Gatehouse & Poppleton (1971*a*). In the crystal, the molecule has the $^4C_1$ pyranose ring conformation with three axial substituents, $O(1)CH_3$, O(2)H and O(3)H, as shown in (I). The alternate $^1C_4$ conformation has only two axial substituents, but one of these is the primary alcohol group. For this reason, both conformers are believed to have similar conformational free energies and to co-exist in aqueous solutions (*cf.* Reeves, 1950; Angyal, 1968; Stoddart, 1971).



(I)

The X-ray analysis indicated that in the crystal all six oxygens were hydrogen bonded by means of the four available protons. This strongly bonded structure was obtained because of an intramolecular hydrogen bond between the vicinal hydroxyls O(3)H and O(4)H and two bifurcated hydrogen bonds in the crystal structure, in addition to two normal intermolecular hydrogen bonds. Vicinal intramolecular hydrogen bonding is geometrically unfavorable in carbohydrates relative to *syn*-diaxial and intermolecular hydrogen bonding (Jeffrey, 1973). Since bifurcated hydrogen bonding is not common in carbohydrate crystal structures, we decided to investigate this structure further by neutron diffraction.

It was also of interest to study, with more accuracy, the distortion of the pyranose ring due to the interaction of the non-bonded *syn*-diaxial oxygens O(1) and O(3), and the bond lengths and conformation of the glycosidic group for comparison with the theoretical predictions of Jeffrey, Pople & Radom (1972, 1974).

Experimental

Crystals were obtained by slow evaporation of saturated aqueous solutions. The one selected for neutron diffraction weighed 63 mg and was of irregular shape with maximum dimensions 0·7 × 0·4 × 0·3 cm. It was mounted on a quartz pin, approximately about **b***, and enclosed in a quartz capsule to avoid deliquescence. 1835 intensities were collected automatically with a monochromatic neutron beam ($\lambda=1{\cdot}020$ Å) from the Brookhaven High Flux Reactor by θ–2θ step scans to $2\theta(\max)=90°$. The background correction was as described by Lehmann & Larsen (1974). Absorption corrections in the range 0·52 to 0·70 were applied by means of *DATAPH* (Bednowitz, Coppens & Hamilton, 1975) using $\mu=2{\cdot}47$ cm^{-1}. A value of 23·9 was used for the mass absorption coefficient of hydrogen corresponding to a value of 43 barns for the incoherent scattering cross-section of hydrogen (Koetzle, 1975). The corrected intensities for symmetry-related reflections were averaged and reduced to give 1475 *hkl* F^2's for the structure refinement.

Starting with the atomic coordinates from the X-ray

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data* and isotropic temperature factors, an R (for F^2) of 0·14 was obtained by least-squares minimization of $\sum\omega|F_o^2-k^2|F_c|^2|^2$ where $\omega=1/\sigma^2(F_o^2)$ with $\sigma^2(F_o^2)=\sigma^2_{\text{count}}(F_o^2)+(0{\cdot}02F_o^2)^2$. The refinement converged in six cycles with anisotropic temperature parameters for all atoms. Two models for anisotropic extinction correction (Coppens & Hamilton, 1970) were tested for the later cycles, but that using Zachariasen's (1967) isotropic g parameter appeared to be equally valid and was used to obtain the final parameters. The final R_{F2} and $R_{\omega F2}$ values are 8·9 and 10·2, respectively. The standard deviation of an observation of unit weight is 2·80. No structure factors were rejected, although seven low orders gave poor agreement. Interestingly many of these same reflections also gave similar poor agreement in the X-ray analysis. A further cycle of refinement was run giving zero weight to reflections where $\Delta F^2\geq 10\sigma^2$. This did not affect any atomic parameters by more than $0{\cdot}2\sigma$, so the parameters using the full set of data are given in Table 1.

The X-ray crystal data, $P2_12_12_1$, $Z=4$, $a=7{\cdot}486$ (4), $b=9{\cdot}098$ (7), $c=13{\cdot}330$ (7) Å of Gatehouse & Poppleton (1971*a*) were used. The unit-cell edges derived from 32 accurately centered reflections on the neutron diffractometer were about 3% greater using $\lambda_n=1{\cdot}020$ Å. We believe that this systematic discrepancy is from a combination of absorption effects and an error in the value of the neutron beam wavelength. The neutron scattering factors used were $\bar{b}_{\rm H}=-0{\cdot}3723$, $\bar{b}_{\rm O}=0{\cdot}575$, $\bar{b}_{\rm C}=0{\cdot}6626$ all $\times 10^{-12}$ cm (Shull, 1971). The computations were carried out on CDC 6600 computers with more recent versions of the programs briefly described by Schlemper, Hamilton & La Placa (1971).* The atomic notation and conformation is shown in (I) and the *ORTEP* diagram in Fig. 1. The molecular dimensions are reported in Figs. 2 and 3. The standard deviations in the cell dimensions given above were used with those from the full inverse matrix to obtain the σ's for the molecular parameters (Busing & Levy, 1964). The mean σ's for the bond lengths were: C–C, 0·003 Å; C–O, 0·0035 Å; O–H, 0·0065 Å; C–H, 0·005 Å; except for the C(7)–H bonds which were 0·015 Å; for the angles, the σ's were: C–C–C and C–C–O, 0·2°, C–C–H and C–O–H, 0·5°, except those involving the H(7)'s which were 1°.

Results and discussion

The hydrogen bonding is the same as that determined by the X-ray analysis and illustrated in Fig. 2 of Gatehouse & Poppleton (1971) except for some details. This bonding scheme is represented diagrammatically below,

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* Note: There is a typographical error in Table 2 of the X-ray coordinates (Gatehouse & Poppleton, 1971*a*). The z coordinate of C(5) should read 0·2963.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31013 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with estimated standard deviations and the components of the thermal vibration tensors* ($\text{Å}^2\times 10^4$) *with their estimated standard deviations*

The U_{ij} are the coefficients in the expression $\exp[-2\pi^2(U_{11}a^{*2}h^2+U_{22}b^{*2}k^2+U_{33}c^{*2}l^2+2U_{12}a^*b^*hk+2U_{13}a^*c^*hl+2U_{23}b^*c^*kl)]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	1490 (3)	2919 (2)	1668 (2)	260 (10)	297 (10)	305 (9)	42 (9)	38 (9)	41 (9)
C(2)	3143 (3)	2801 (2)	2332 (2)	190 (9)	275 (10)	444 (11)	−2 (8)	61 (9)	38 (9)
C(3)	2893 (3)	1736 (2)	3212 (2)	180 (9)	312 (11)	361 (10)	9 (9)	−26 (8)	12 (9)
C(4)	1092 (3)	1965 (2)	3728 (1)	220 (9)	269 (10)	273 (9)	−13 (9)	−18 (8)	−20 (8)
C(5)	−416 (3)	1999 (2)	2962 (1)	175 (9)	249 (10)	291 (9)	8 (8)	5 (7)	−25 (9)
C(6)	−2203 (3)	2346 (3)	3431 (2)	208 (10)	326 (12)	456 (12)	16 (9)	98 (11)	19 (11)
C(7)	57 (6)	1705 (5)	332 (3)	707 (26)	688 (23)	511 (18)	227 (21)	−261 (19)	−138 (18)
O(1)	1391 (4)	1652 (3)	1075 (2)	390 (14)	394 (14)	315 (12)	105 (13)	−3 (12)	−37 (11)
O(2)	3477 (4)	4230 (3)	2726 (3)	256 (13)	302 (13)	707 (19)	−77 (12)	41 (15)	−14 (14)
O(3)	3037 (4)	265 (3)	2850 (2)	235 (13)	287 (12)	460 (14)	55 (11)	39 (12)	1 (12)
O(4)	737 (4)	781 (3)	4383 (2)	363 (14)	428 (16)	301 (12)	−109 (14)	−58 (12)	66 (12)
O(5)	−80 (3)	3137 (3)	2237 (2)	213 (10)	290 (12)	313 (11)	54 (10)	28 (10)	39 (10)
O(6)	−2195 (4)	3647 (3)	4002 (2)	415 (15)	335 (14)	440 (14)	96 (14)	212 (13)	34 (12)
H(C1)	1565 (8)	3908 (5)	1188 (4)	501 (28)	407 (24)	601 (28)	68 (25)	127 (27)	158 (22)
H(C2)	4266 (7)	2434 (6)	1870 (4)	284 (22)	548 (29)	730 (34)	61 (22)	160 (24)	91 (26)
H(C3)	3978 (6)	1949 (6)	3748 (4)	312 (23)	529 (30)	654 (30)	−35 (24)	−162 (23)	−1 (26)
H(C4)	1112 (7)	3025 (5)	4123 (3)	405 (25)	430 (25)	489 (23)	−38 (24)	2 (22)	−140 (21)
H(C5)	−529 (6)	920 (5)	2586 (3)	370 (22)	313 (20)	467 (22)	12 (20)	−27 (20)	−64 (19)
H(C61)	−2557 (8)	1445 (6)	3949 (5)	472 (30)	457 (28)	800 (34)	−41 (25)	244 (29)	119 (28)
H(C62)	−3228 (7)	2377 (7)	2858 (5)	262 (23)	701 (35)	852 (36)	24 (25)	−69 (28)	−42 (32)
H(C71)	220 (17)	842 (12)	−160 (7)	1504 (96)	1391 (81)	992 (56)	551 (81)	−608 (70)	−618 (62)
H(C72)	5 (26)	2730 (16)	−36 (10)	2497 (186)	1169 (86)	1248 (77)	213 (100)	−1152 (110)	271 (67)
H(C73)	−1248 (16)	1606 (20)	652 (9)	671 (54)	2736 (184)	1335 (90)	253 (94)	−437 (64)	−493 (113)
H(O2)	4681 (7)	4517 (6)	2570 (5)	289 (26)	480 (27)	919 (38)	−148 (22)	22 (26)	−19 (30)
H(O3)	2158 (9)	−305 (6)	3205 (4)	508 (31)	375 (24)	722 (32)	−6 (26)	215 (30)	68 (24)
H(O4)	1421 (7)	931 (5)	4993 (3)	475 (25)	493 (25)	407 (21)	−76 (26)	−108 (24)	39 (21)
H(O6)	−2096 (9)	4496 (6)	3572 (4)	608 (35)	386 (25)	626 (29)	25 (26)	257 (30)	67 (23)

$$\rightarrow O(4) \rightarrow O(6) \rightarrow \left\{ \begin{array}{l} O(1) \\ \begin{array}{c} O(3) \\ \uparrow \\ O(2) \end{array} \rightarrow \left\{ \begin{array}{l} O(5) \\ O(4) \rightarrow \end{array} \right. \end{array} \right.$$

It consists of a continuous chain forming a helix in the **b** direction with a short branch. There are two normal intermolecular hydrogen bonds O(4)H → O(6), O(2)H → O(3), and two bifurcated bonds involving, respectively, the hydroxyl O(4)H and the ring oxygen O(5), the hydroxyl O(3)H and the glycosidic oxygen O(1). One of these bifurcated bonds involves the vicinal intramolecular interaction, *i.e.* O(3)H–O(4). Because of the two bifurcated hydrogen bonds, the hydrogen bonding by the four hydroxyl protons involves all six oxygens; every hydroxyl donates and accepts a hydrogen bond and both ether oxygens are acceptors. This involvement of all the oxygens in hydrogen bonding must be an important factor contributing to the stability of the conformation observed in the crystals. The geometry of these hydrogen bonds is shown in Fig. 2. As in other carbohydrate structures,

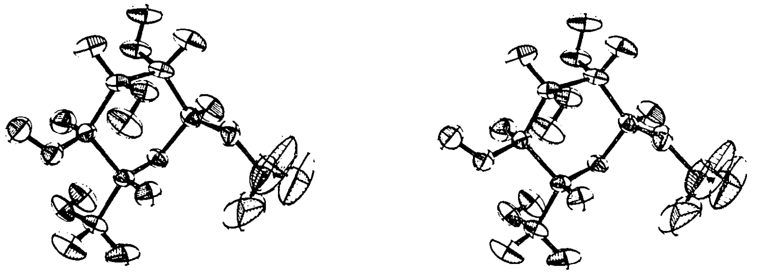


Fig. 1. Stereo *ORTEP* plot at 50% probability for methyl α-D-altropyranoside (Johnson, 1965).

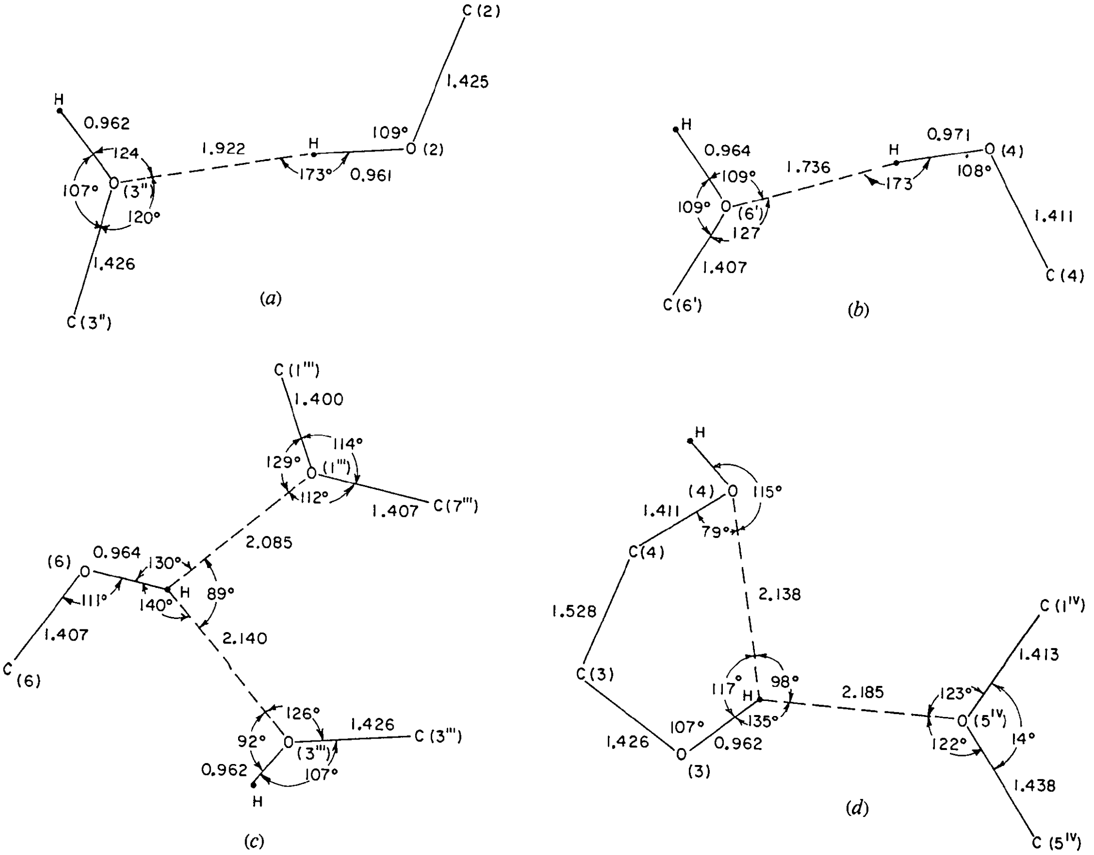
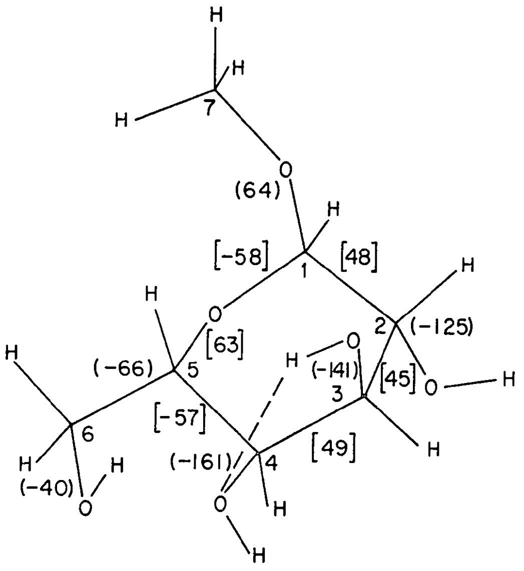


Fig. 2. The geometry of the hydrogen bonding and the C–O, O–H bond lengths in methyl α-D-altropyranoside. (*a*), (*b*) The intermolecular hydrogen bonds O(2)–H···O(3''), O(4)–H···O(6^{i}). (*c*) The intermolecular bifurcated hydrogen bonds $O(6)\text{–}H\begin{array}{l}\cdots O(1''')\\ \cdots O(3'')\end{array}$. (*d*) The intra-intermolecular bifurcated hydrogen bond $O(3)\text{–}H\begin{array}{l}\cdots O(4)\\ \cdots O(5^{iv})\end{array}\begin{array}{l}\text{intramolecular}\\ \text{intermolecular}\end{array}$. [Notation is as in Gatehouse & Poppleton (1971*a*) except that (i) is $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$.]

the hydrogen bond H···O distances vary over a wide range without any correlation with the O–H···O angles or O–H distances. The two normal intermolecular hydrogen bonds have H···O distances of 1·736 and 1·922 Å with the same O–H···O angle of 173°. The H···O distances of the bifurcated bonds are more nearly equal, 2·09 to 2·19 Å. The angles of 89° and 98° at the hydrogens of the bifurcated bonds correspond to separations of 2·95 and 3·25 Å between the acceptor oxygens. The C–O–H angles are close to tetrahedral in the normal hydrogen bonds, but are 107° and 111° in the bifurcated cases. The acceptor C–O···H angles vary more widely, being as small as 79° in the vicinal intramolecular bond. As shown by the angles around the hydrogens involved in the bifurcated bonds, H(O6) is much closer to the plane of O(6), O(1) and O(3), *i.e.* 0·10 Å, than H(O(3) is to that of O(3), O(4) and O(5), *i.e.* 0·25 Å. The pyranose ring is distorted, as shown in Fig. 3, by the variation of the ring torsion angles outside the customary range of 52–65°. The systematic differences in the torsion angles, observed in simple unstrained monosaccharides *i.e.* C–C–O–C > C–C–C–O > C–C–C–C (Jeffrey, 1973), are not observed. The biggest distortions occur across C(1)–C(2) and C(2)–C(3). These are a consequence of the *syn*-diaxial repulsion of O(1) and O(3), which are separated by 2·952 Å rather than the 2·50 Å required by an undistorted ring and tetrahedral angles. This distortion is such that the C(1)–O(2) and C(3)–O(3) bonds remain coplanar, indicating that it requires less energy to distort the ring by displacements normal to a four-atom ring plane than by a twist of opposite bonds of the ring. This is therefore the most likely route for the ring conformational change to the $^4C_1 \rightleftharpoons {}^1C_4$ equilibrium mixture in solution. The ring torsion angle about C(3)–C(4) is also lessened so as to bring the bonds C(3)–O(3), C(4)–O(4) more nearly parallel; presumably this is a result of the vicinal intramolecular hydrogen bond. The torsion angle O(3)–C(3)–C(4)–O(4) is 46°, rather than the usual 55–60°. The ring is closest to a $^2C_5$ chair with displacements of 0·568 Å for C(2) and −0·67 Å for C(5) from the reference plane, or $^3C_0$ with displacements of 0·591 Å for C(3) and −0·638 Å for O(5).



C(1) - C (2)	1.525Å
C(2) - C (3)	1.533
C(3) - C (4)	1.528
C(4) - C (5)	1.524
C(5) - C (6)	1.510
C(1) - O (1)	1.400
C(2) - O (2)	1.425
C(3) - O (3)	1.426
C(4) - O (4)	1.411
C(5) - O (5)	1.438
C(1) - O (5)	1.413
C(6) - O (6)	1.407

C(1) -H(1)	1.105
C(2) -H(2)	1.094
C(3) -H(3)	1.100
C(4) -H(4)	1.099
C(5) -H(5)	1.105
C(6) -H(6)	1.104
C(6) -H(6')	1.083
C(7) -H(7)	1.031
C(7) -H(7')	1.054
C(7) -H(7'')	1.070

Fig. 3. Torsion angles C–C and C–H bond distances in methyl α-D-altropyranoside. The ring torsion angles are in square brackets. Other torsion angles are according to Klyne–Prelog (1960) priority. The intramolecular hydrogen bond is dotted.

The bond distances are included in Figs. 2 and 3. When compared with the X-ray data, there appears to be a small, but systematic, shortening of the neutron diffraction C–O bond lengths on average by 0·007 Å (2σ). This was also observed in the comparison of the X-ray and neutron analyses for sucrose (Brown & Levy, 1973; Hanson, Sieker & Jensen, 1972, 1973), potassium gluconate monohydrate (Panagiotopoulos, Jeffrey, La Placa & Hamilton, 1975; Jeffrey & Fasiska, 1972), D-glucitol (Jeffrey, Park & Hamilton, 1971). In the sucrose data, which are the most accurate, the differences ranged from −0·0014 to −0·0086 Å, with one exception of +0·0017 in 14 C–O bond lengths. In the 32 C–O bonds in this and the three structures quoted above, there were only three exceptions and the mean shift was −0·0060 Å. This suggests that, where X-ray

Table 2. *Deviations of some* C–O *bond lengths from a standard value* (*experimental* −1·428) ×10³

The deviation tabulated refers to the bond between the two atoms flanking the top of the column. θ and φ refer to the torsion angles.

		θ	ϕ				
	C(5)–O(5)–C(1)–O(1)–CH$_3$				θ	φ	References
Methyl α-glucopyranoside	+5	−15	−17	+1	+59°	+63°	Berman & Kim (1968)
Methyl α-galactopyranoside	+11	−10	−23	+8	+62	+63	Gatehouse & Poppleton (1971*b*)
Methyl α-mannopyranoside	+21	−8	−22	+15	+60	+61	Gatehouse & Poppleton (1970)
Methyl α-altropyranoside (X-ray)	+12	−12	−23	0	+64	+64	Gatehouse & Poppleton (1971*a*)
Methyl α-altropyranoside (neutrons)	+10	−15	−28	−21	+64	+64	This work
Theory (relative to 1·437 Å)		−20	−16	+7	60	60	Jeffrey, Pople & Radom (1974)

C–O bond lengths are compared with values from experimental nuclear separations or from theoretical calculations, this small correction should be applied. This correction is consistent with the displacement of the lone-pair electron density from the nuclear center and the consequent use of an incorrect model of isotropic scattering factors for the oxygen atoms. No similar systematic difference is observed in the C–C bonds from the same structures.

With the exception of C(5)–C(6), the C–C bond lengths are within 2σ of the mean value of 1·524 Å. The primary alcohol C–C bond is shorter by 3σ and was also short in the X-ray results (1·495 Å). Although distances of 1·510 Å or less have been observed for this bond in the structures of several other pyranoses and methyl pyranosides, this bond is normal (1·523 Å) in the sucrose work.

The C–OH bonds are within 2σ of the mean of 1·417 Å. The geometry of the acetal bond sequence C(5)–O(5)–C(1)–O(1)–C(7) is of interest for comparison with the quantum mechanical calculations on methanediol and methoxymethanol (Jeffrey, Pople & Radom, 1971, 1974). This comparison is shown in Table 2 together with data from three other methyl α-D-pyranosides. The shortening of the bonds adjacent to the anomeric carbon C(1) predicted by the theory, is well reproduced both by this neutron data and the X-ray results. However, the smaller lengthening of the methoxy C–O, which is predicted and observed in the other structures, is not observed in this structure and we have no explanation for this at present.

The torsion angles of the primary alcohol group are −66° and 54° with respect to O(5) and C(4) respectively (*i.e.* g_0g), corresponding to a O(6) to O(5) approach of 2·874 Å. The C–C–C bond angles are unremarkable and range from 110° to 113°. The ring and glycosidic C–O–C angles are 114°.

The O–H bonds range from 0·961 to 0·971 Å, and there is no evidence of sensitivity to the hydrogen bonding. The C–H distances are 1·083 to 1·105 Å, except for those on the methyl group which are shorter by 0·05 Å. A rigid-body motion analysis using the procedure of Schomaker & Trueblood (1968) gave a good fit for the six pyranose ring atoms, with an average value of $U_{ij} - U_{ij\,\mathrm{RBM}} = 0{\cdot}0003$ $[0{\cdot}3\sigma(u_{ij})]$. This average difference increased to 0·0014 Å when O(1), O(2), O(3), O(4) and C(6) were included and to 0·0031 Å when the five hydrogens on C(1) to C(5) were added.* The rigid-body corrections on the C–H distances were small, < 0·005 Å, but the riding-motion corrections (Busing & Levy, 1964) ranged from 0·017 Å for C(5)–H(5) to a mean of 0·115 Å for C(7)–H, and were probably overestimated, although they brought all C–H distances within the range of 1·110 Å [C(6)–H(6)] to 1·132 Å [C(1)–H].

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* A table giving the rigid-body motion parameters based on the six-atom and sixteen-atom molecular fragments has been deposited with the structure factor table. See footnote on p. 2401.