

contrasts with the well documented (Glushkov & Granik, 1969) reactivity of lactams towards $\text{Et}_3\text{O}^+\text{BF}_4^-$ which regularly yield the corresponding *O*-alkylated derivatives.

We thank the Deutsche Forschungsgemeinschaft for financial support and the Fonds der Chemischen Industrie for the award of a fellowship (to MB-P), as well as Dr J. Engel for preparing the crystals.

References

- BADGER, G. M., JONES, R. A. & LASLETT, R. L. (1964). *Aust. J. Chem.* **17**, 1157–1163.
- BONNETT, R. (1977). *The Porphyrins*, edited by D. DOLPHIN. New York: Academic Press. In the press.
- BONNETT, R., DAVIES, J. E. & HURSTHOUSE, M. B. (1976). *Nature, Lond.* **262**, 326–328.
- BONNETT, R., HURSTHOUSE, M. B. & NEIDLE, S. (1972a). *J. Chem. Soc. Perkin II*, pp. 902–906.
- BONNETT, R., HURSTHOUSE, M. B. & NEIDLE, S. (1972b). *J. Chem. Soc. Perkin II*, pp. 1335–1340.
- BURKE, M. J., PRATT, D. C. & MOSCOWITZ, A. (1972). *Biochemistry*, **11**, 4025–4031.
- CAILLET, A., BAUER, D., FROYER, G. & SEKIGUCHI, H. (1973). *C. R. Acad. Sci. Paris*, **277**, 1211–1214.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- CULLEN, D. L., BLACK, P. S., MEYER, E. F. JR, LIGHTNER, D. A., QUISTAD, G. B. & PAK, C. S. (1977). *Tetrahedron*, **33**, 477–483.
- FALK, H., GRUBMAYR, K., HERZIG, U. & HOFER, O. (1975). *Tetrahedron Lett.* pp. 559–562.
- FLITSCH, W. & NEUMANN, U. (1971). *Chem. Ber.* **104**, 2170–2176.
- GLUSHKOV, R. G. & GRANIK, V. G. (1969). *Russ. Chem. Rev.* **38**, 913–925.
- GOSSAUER, A., BLACHA, M. & SHELDRIK, W. S. (1976). *Chem. Commun.* pp. 764–765.
- GOSSAUER, A. & KÜHNE, G. (1977). *Liebigs Ann.* In the press.
- GOSSAUER, A. & MIEHE, D. (1974). *Liebigs Ann.* pp. 352–362.
- GOSSAUER, A., MIEHE, D. & INHOFFEN, H. H. (1970). *Liebigs Ann.* **738**, 31–41.
- GRAMBEIN, S., RÜDIGER, W. & ZIMMERMANN, H. (1975). *Z. physiol. Chem.* **356**, 1709–1714.
- HERZ, W. (1949). *J. Amer. Chem. Soc.* **71**, 3982–3984.
- HUDSON, M. F. & SMITH, K. M. (1976). *Quart. Rev.* **30**, 363–399.
- JACKSON, A. H. & SMITH, K. M. (1973). *The Total Synthesis of Natural Products*, Vol. 1, edited by J. AP SIMON, pp. 145–146. New York: Wiley-Interscience.
- JONES, R. A. (1970). *Advanc. Heterocycl. Chem.* **11**, 383–472.
- JONES, R. A. & LINDNER, J. A. (1965). *Aust. J. Chem.* **18**, 875–885.
- JONES, R. A., POJARLIEVA, T. & HEAD, R. J. (1968). *Tetrahedron*, **24**, 2013–2017.
- NYGAARD, L., NIELSEN, J. T., KIRCHHEINER, J., MALTESEN, G., RASTRUP-ANDERSEN, J. & SØRENSEN, G. O. (1969). *J. Mol. Struct.* **3**, 491–506.
- RATUSKÝ, J. & ŠORM, F. (1958). *Coll. Czech. Chem. Commun.* **23**, 467–478.
- SHELDRIK, W. S. (1976). *J. Chem. Soc. Perkin II*, pp. 1457–1462.
- YAGIL, G. (1967). *Tetrahedron*, **23**, 2855–2861.

Acta Cryst. (1977). **B33**, 3635–3640

The Crystal and Molecular Structure of DL-Mannitol at -150°C

BY J. A. KANTERS, G. ROELOFSEN AND D. SMITS

Structural Chemistry Group, Rijksuniversiteit Utrecht, Transitorium 3, Padualaan 8, Utrecht, The Netherlands

(Received 12 April 1977; accepted 14 May 1977)

DL-Mannitol, $\text{C}_6\text{H}_{14}\text{O}_6$, is orthorhombic, space group $Pna2_1$, with $a = 9.048$ (7), $b = 4.870$ (3), $c = 18.262$ (13) Å, $Z = 4$ at -150°C . The structure was refined to $R = 0.030$ with 956 counter reflexions collected at -150°C . In DL-mannitol the D-mannitol molecule has the same conformation as that in the B and K forms of D-mannitol. The molecule has a planar C atom chain and nearly a twofold axis of symmetry. All OH groups are involved in intermolecular hydrogen bonds. The hydrogen-bond pattern is similar to that of DL-arabinitol, but differs markedly from that in the B and K forms of D-mannitol.

Introduction

Mannitol, $\text{C}_6\text{H}_{14}\text{O}_6$, is an acyclic polyalcohol, of which the D-enantiomer is widely distributed in nature. The L

and DL forms do not occur in nature. D-Mannitol is unusual in the class of alditols in that it exists in at least three crystalline polymorphs. The B form (Berman, Jeffrey & Rosenstein, 1968) and the K form (Kim,

Jeffrey & Rosenstein, 1968) have been studied in detail and the A' form (Berman, Jeffrey & Rosenstein, 1968) in two dimensions. In these three forms D-mannitol has the same molecular conformation in different hydrogen-bond environments.

Though it is known that a mixture of rotamers exists in solution and in the melt of alditols (Jeffrey & Kim, 1970), as yet no conformational polymorphism has been observed in crystalline polymorphs of alditols.

The aim of the present study is to examine the conformational stability in a structure in which both enantiomers are present and also to compare the hydrogen-bond pattern with that observed in the B and K forms of D-mannitol and in the closely related DL-arabinitol (Hunter & Rosenstein, 1968).

The structure of DL-mannitol was determined at room and liquid-nitrogen (-150°C) temperatures with the same crystal. As the results (bond lengths and angles) of both analyses do not differ by more than 2.5σ , only the low-temperature structure will be reported.

Experimental

Large colourless crystals were obtained by slow evaporation of an equimolar solution of the D- and L-enantiomers in aqueous ethanol. Preliminary cell dimensions and the space group were determined from photographs. Accurate cell dimensions and intensities were measured on an automatic Nonius CAD-4 diffractometer at liquid-nitrogen temperature with Zr-

filtered Mo $K\alpha$ radiation ($\lambda = 0.71069 \text{ \AA}$) and the ω - 2θ scan technique. The crystal data are summarized in Table 1. The intensities were collected up to $\sin \theta/\lambda = 0.65 \text{ \AA}^{-1}$; 21 reflexions with $I < 2.5\sigma(I)$ were considered unobserved. The equivalent reflexions were averaged to give 935 independent intensities after which Lorentz-polarization corrections were applied. No correction for absorption was made.

Determination and refinement of the structure

The structure was solved with *MULTAN* of the XRAY system (1972). The data were placed on an absolute scale by Wilson's (1942) method. The initial value of the overall temperature factor was 1.09 \AA^2 .

For the structure determination, 128 normalized structure factors with $E > 1.30$ were used. Full-matrix least-squares refinement (*CRYLSQ* of the XRAY system, 1972) of the positions and isotropic thermal parameters of the C and O atoms followed by anisotropic thermal refinement gave $R = 0.063$ for 935 reflexions. A difference map showed 14 maxima which could be assigned to the H atoms. The electron densities of these peaks ranged from 0.30 to 0.77 e \AA^{-3} . The positions of the H atoms, with constant iso-

Table 1. *Crystal data for DL-mannitol*

Molecular formula: $\text{C}_6\text{H}_{14}\text{O}_6$		
FW 182.17		
Crystal system: orthorhombic		
	20°C	-150°C
a (Å)	9.149 (8)	9.048 (7)
b (Å)	4.869 (4)	4.870 (3)
c (Å)	18.237 (12)	18.262 (13)
Systematic absences	$0kl, k + l = 2n + 1$	$h0l, h = 2n + 1$
Space group*	$Pna2_1$	
V (Å ³)	812.40 (20°C), 804.69 (-150°C)	
Z	4	
D_m (g cm ⁻³)	1.489 (20°C)	
D_x (g cm ⁻³)	1.489 (20°C), 1.504 (-150°C)	
μ (Mo $K\alpha$) (cm ⁻¹)	1.46	
Crystal dimensions (mm)	0.6 × 0.3 × 0.2	
λ (Mo $K\alpha$) (Å)	0.71069	

* The noncentrosymmetric alternative, $Pna2_1$, rather than the centrosymmetric $Pnam$, which has the same systematic absences, was chosen on the basis of the molecular symmetry. In $Pnam$, with $Z = 4$, each molecule would occupy a special position with m symmetry, contrary to its known optical activity.

Table 2. *Fractional atomic coordinates* ($\times 10^4$, for H $\times 10^3$) *for DL-mannitol*

The estimated standard deviations are given in parentheses and refer to the last decimal position of respective values.

	x	y	z
C(1)	4208 (2)	7113 (4)	1050 (1)
C(2)	4249 (2)	8806 (4)	1751 (2)
C(3)	4205 (2)	7025 (3)	2437 (2)
C(4)	4135 (2)	8711 (4)	3144 (2)
C(5)	4188 (2)	6881 (4)	3824 (1)
C(6)	4261 (2)	8496 (4)	4535 (1)
O(1)	2880 (2)	5539 (3)	980*
O(2)	3083 (2)	10793 (3)	1753 (1)
O(3)	5518 (1)	5389 (3)	2416 (1)
O(4)	5286 (1)	10712 (3)	3166 (1)
O(5)	2911 (1)	5171 (3)	3796 (1)
O(6)	3088 (2)	10461 (3)	4575 (1)
H(C1)	427 (3)	831 (5)	61 (2)
H'(C1)	517 (3)	597 (5)	101 (2)
H(C2)	518 (3)	982 (5)	173 (1)
H(C3)	334 (2)	579 (5)	241 (2)
H(C4)	312 (3)	968 (5)	316 (2)
H(C5)	505 (3)	577 (5)	381 (1)
H(C6)	414 (3)	733 (5)	494 (1)
H'(C6)	520 (3)	941 (5)	453 (1)
H(O1)	301 (3)	417 (6)	120 (2)
H(O2)	220 (3)	1010 (6)	190 (2)
H(O3)	540 (3)	391 (6)	264 (1)
H(O4)	601 (3)	1007 (5)	331 (1)
H(O5)	308 (3)	388 (6)	405 (2)
H(O6)	275 (3)	1036 (6)	499 (2)

* In space group $Pna2_1$, the origin is not defined in the z direction; to fix the origin the z coordinate of O(1) was kept constant at 0.098 during the entire refinement.

tropic thermal parameters equal to those of the carrier atoms, were included in the refinement. The quantity minimized was $\sum w(F_o - F_c)^2$ with weights $w = \sigma^{-2}(F_o)$. Scattering factors for C and O were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965). The refinement was terminated at $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.030$ and $R_w = [\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/2} = 0.042$ for 935 observed reflexions. The goodness-of-fit is 2.52, the maximum shift/error ratio 0.14 and the average shift/error ratio 0.018. A final difference synthesis showed no peaks above $0.36 \text{ e } \text{Å}^{-3}$. Positional parameters are listed in Table 2.*

Description of the structure

The numbering of the atoms and the conformation of the D-enantiomer are shown in Fig. 1. As in the B and K forms of D-mannitol and DL-arabinitol the C atoms and two terminal H atoms form a planar zigzag chain almost parallel to (100) (Table 3). The molecular symmetry is approximately 2, as far as the C and O atoms are concerned (Table 3). The mean distance of atom pairs from the twofold axis is 0.042 Å ; for the B and K forms of D-mannitol this is 0.017 and 0.007 Å respectively.

The fact that the molecular symmetry of D-mannitol in the three mannitol structures is the same suggests that this particular rotamer, *i.e.* *Msc, ap, Psc, ap, Msc*, is the stable one, although it would appear that the synclinal (*Psc*) conformation of the terminal C—C

bonds should be energetically equally favourable (Berman, Jeffrey & Rosenstein, 1968).

Bond lengths and angles involving C and O atoms are given in Table 4, with corresponding values for the B and K forms of D-mannitol. The C—C bonds range from 1.519 to 1.531 Å (mean 1.525 Å). As in many alditol structures the two terminal C—C bonds are shorter than those within. It has been remarked (Hunter & Rosenstein, 1968; Kim & Jeffrey, 1969; Jeffrey & Kim, 1970) that the chemical significance of this trend is difficult to assess because of uncertainty in the treatment of the thermal motion.

The C—O bonds range from 1.426 to 1.432 Å (mean 1.429 Å). The C—C—C angles range from 111.8 to 113.2° (mean 112.6°), and the C—C—O angles from 105.9 to 112.7° (mean 110.2°). The mean values of bond lengths and angles are in good agreement with those (Jeffrey & Kim, 1970) of nine alditols: C—C 1.522 (3), C—O 1.428 (6) Å; C—C—C 113.0 (6), C—C—O 109.6 (4)°.

Bond lengths and angles involving H atoms are given in Table 5. The conformation angles of D-mannitol in the three crystal structures (Table 6) differ little from those for the ideal planar chain conformation *Msc, ap, Psc, ap, Msc*. Though the conformations are the same, in DL-mannitol the three intramolecular distances between O atoms in the *gauche* position have a mean value of 2.94 Å , whereas in both the B and K forms it

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32755 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

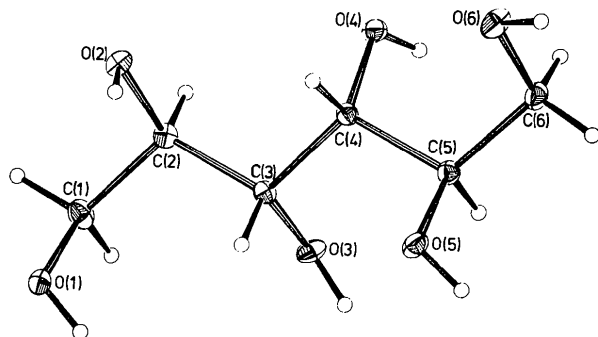


Fig. 1. Molecular conformation and the atomic numbering of the D-enantiomer of mannitol. C and O atoms are represented at the 50% probability level. The H atoms are represented by artificial small spheres.

Table 3. Least-squares plane and twofold axis in DL-mannitol

Equation for plane: $Ax + By + Cz = D$, where x, y, z are in Å, and $A = 0.9999$, $B = -0.0142$, $C = 0.0002$ and $D = 3.7537$. The angle between this plane and (100) is 0.83° . Asterisks indicate atoms which were not included in the calculation of the plane.

Deviations (Å) of atoms from the plane

C(1)	+0.005	C(5)	-0.011
C(2)	+0.030	C(6)	+0.044
C(3)	+0.003	*H(C1)	+0.052
C(4)	-0.072	*H(C6)	-0.061

Parametric equation of the best twofold axis (Å)

$$\begin{aligned} x &= 3.612 + 0.999t \\ y &= 3.859 - 0.001t \\ z &= 5.089 - 0.014t \end{aligned}$$

Distance of mid-point of atom pairs from molecular twofold axis

Atom pairs	
C(1) C(6)	0.059 Å
C(2) C(5)	0.039
C(3) C(4)	0.028
O(1) O(6)	0.035
O(2) O(5)	0.028
O(3) O(4)	0.064

The angle between the best plane and the best axis is 88.9° .

Table 4. Bond distances (\AA) and angles ($^{\circ}$) of DL-mannitol involving C and O atoms, with corresponding values for B-D-mannitol and K-D-mannitol

The e.s.d.'s are given in parentheses.

	DL-Mannitol ^a	B-D-Mannitol ^b	K-D-Mannitol ^c	
C(1)–C(2)	1.522 (3)	1.505 (10)	1.511 (6)	
C(2)–C(3)	1.525 (4)	1.522 (10)	1.519 (5)	
C(3)–C(4)	1.531 (4)	1.513 (10)	1.524 (5)	
C(4)–C(5)	1.531 (3)	1.537 (10)	1.517 (5)	
C(5)–C(6)	1.519 (3)	1.503 (10)	1.523 (5)	
C(1)–O(1)	1.431 (2)	1.425 (9)	1.421 (5)	
C(2)–O(2)	1.432 (2)	1.440 (9)	1.423 (5)	
C(3)–O(3)	1.430 (2)	1.433 (9)	1.433 (4)	
C(4)–O(4)	1.427 (2)	1.448 (9)	1.427 (4)	
C(5)–O(5)	1.426 (2)	1.444 (9)	1.431 (4)	
C(6)–O(6)	1.430 (2)	1.436 (9)	1.415 (5)	
C(1)–C(2)–C(3)	112.5 (2)	111.6 (6)	113.0 (3)	
C(2)–C(3)–C(4)	112.9	112.8	113.4	
C(3)–C(4)–C(5)	111.8	113.3	113.4	
C(4)–C(5)–C(6)	113.2	113.2	113.6	
O(1)–C(1)–C(2)	112.7	112.9	111.6	
O(2)–C(2)–C(1)	110.5	107.7	110.6	
O(2)–C(2)–C(3)	111.3	109.8	109.7	
O(3)–C(3)–C(2)	105.9	109.3	109.3	
O(3)–C(3)–C(4)	110.8	108.7	110.9	
O(4)–C(4)–C(3)	111.1	109.9	109.6	
O(4)–C(4)–C(5)	110.6	108.7	109.1	
O(5)–C(5)–C(4)	106.6	109.5	109.6	
O(5)–C(5)–C(6)	111.6	106.5	110.2	
O(6)–C(6)–C(5)	111.0	111.8	111.2	
Mean values				
C–C	1.525 (5)	1.516 (14)	1.519 (5)	1.522 (3) ^d
C–O	1.429 (1)	1.438 (8)	1.426 (7)	1.428 (6) ^d
C–C–C	112.6 (6)	112.7 (8)	113.4 (1)	113.0 (6) ^d
C–C–O	110.1 (22)	109.5 (18)	110.2 (9)	109.6 (4) ^d

(a) This article. (b) Berman, Jeffrey & Rosenstein (1968). (c) Kim, Jeffrey & Rosenstein (1968). (d) Mean values of bond distances and angles of nine alditols (Jeffrey & Kim, 1970).

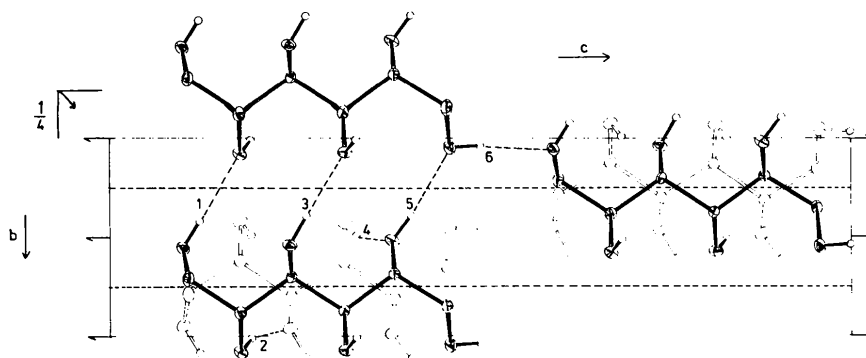


Fig. 2. A view of the structure of DL-mannitol down **a**. Hydrogen bonds, numbered according to Table 7, are indicated by dashed lines.

Table 5. Bond distances (Å) and bond angles (°) of DL-mannitol involving H atoms

The e.s.d.'s are given in parentheses.

C(1)–H(C1)	1.00 (4)	C(1)–H'(C1)	1.03 (3)
C(2)–H(C2)	0.98 (3)	C(3)–H(C3)	0.99 (2)
C(4)–H(C4)	1.03 (2)	C(5)–H(C5)	0.95 (3)
C(6)–H(C6)	0.94 (3)	C(6)–H'(C6)	0.96 (3)
O(1)–H(O1)	0.79 (3)	O(2)–H(O2)	0.91 (3)
O(3)–H(O3)	0.83 (3)	O(4)–H(O4)	0.77 (3)
O(5)–H(O5)	0.80 (3)	O(6)–H(O6)	0.83 (3)
H(O1)–O(1)–C(1)	106 (2)	H(O2)–O(2)–C(2)	113 (2)
H(O3)–O(3)–C(3)	111 (2)	H(O4)–O(4)–C(4)	111 (2)
H(O5)–O(5)–C(5)	106 (2)	H(O6)–O(6)–C(6)	106 (2)
H(C1)–C(1)–O(1)	107 (1)	H(C1)–C(1)–C(2)	111 (2)
H(C1)–C(1)–H'(C1)	102 (2)	H'(C1)–C(1)–O(1)	114 (1)
H'(C1)–C(1)–C(2)	109 (2)	H(C2)–C(2)–O(2)	107 (1)
H(C2)–C(2)–C(1)	106 (2)	H(C2)–C(2)–C(3)	110 (2)
H(C3)–C(3)–O(3)	109 (1)	H(C3)–C(3)–C(2)	109 (2)
H(C3)–C(3)–C(4)	109 (2)	H(C4)–C(4)–O(4)	110 (1)
H(C4)–C(4)–C(3)	108 (2)	H(C4)–C(4)–C(5)	106 (2)
H(C5)–C(5)–O(5)	109 (2)	H(C5)–C(5)–C(4)	109 (2)
H(C5)–C(5)–C(6)	107 (2)	H(C6)–C(6)–O(6)	106 (2)
H(C6)–C(6)–C(5)	110 (2)	H(C6)–C(6)–H'(C6)	113 (2)
H'(C6)–C(6)–O(6)	110 (2)	H'(C6)–C(6)–C(5)	106 (2)

Mean values

C–H	0.99 (4)
O–H	0.82 (5)
C–O–H	109 (3)
C–C–H	108 (2)
O–C–H	109 (2)
H–C–H	108 (8)

Number of contributors

8
6
6
12
8
2

is 2.84 Å, equal to the value expected for the ideal unstrained conformation with average bond lengths and angles. Comparison of the relevant O...O torsion angles shows that the larger separation in DL-mannitol

is primarily due to the enlargement of the O...O torsion angles beyond the ideal value of 60°.

The molecular packing and hydrogen bonds are shown in Fig. 2; the hydrogen-bond geometry is given in Table 7. As in the structures of nine other alditols (Jeffrey & Kim, 1970) the molecules are arranged in a very regular pattern, which utilizes their full potential for hydrogen-bond formation. The hydrogen bonds are intermolecular; each O–H group acts as a donor and each O atom accepts one hydrogen bond. Both the range and the mean of the six O...O distances, 2.666 to 2.714 Å and 2.691 Å respectively, have a minimal value in the alditols.

The enantiomers are related by the *a* glide plane to form DL pairs which are connected by two hydrogen bonds, O(2)...O(3') and O(4)...O(5'), forming infinite chains along *a*. The D enantiomer is also joined to another D enantiomer at a distance of a unit-cell translation along *b* by three hydrogen bonds, O(1)...O(2'), O(3)...O(4') and O(5)...O(6'), thus constituting an infinite sheet of hydrogen-bonded molecules. The last hydrogen bond, O(6)...O(1'), links the terminal O atoms of molecules related by the *n* glide plane. This hydrogen-bond scheme is very similar to that of DL-arabinitol (Hunter & Rosenstein, 1968) which also crystallizes in *Pna*2₁. Here two hydrogen bonds connect two molecules related by the *a* glide plane, and two link molecules related by a translation along *b*. However, the hydrogen-bonded terminal O(1) and O(5') belong to molecules that are related by the 2₁ axis and not by the *n* glide plane as in DL-mannitol.

The hydrogen-bond patterns of the B and K forms of D-mannitol (both *P*2₁2₁2₁) are different from this. Their schemes are very similar and can be characterized by an infinite spiral in which two hydrogen bonds

Table 6. Conformation angles (°) of DL-mannitol, B-D-mannitol and K-D-mannitol

The conformation angle A(1)–A(2)–A(3)–A(4) is viewed along A(2)–A(3), with a clockwise rotation of A(1) to A(4) taken to be positive.

	DL-Mannitol ^a	B-D-Mannitol ^b	K-D-Mannitol ^c
O(1)–C(1)–C(2)–C(3)	+62.0	+56.2	+65.4
O(1)–C(1)–C(2)–O(2)	–63.0	–64.5	–58.0
C(1)–C(2)–C(3)–C(4)	+184.0	+184.7	+185.2
C(1)–C(2)–C(3)–O(3)	+62.6	+63.6	+60.9
O(2)–C(2)–C(3)–O(3)	–172.7	–177.0	–175.3
O(2)–C(2)–C(3)–C(4)	–51.3	–55.9	–51.0
O(3)–C(3)–C(4)–C(5)	–57.9	–62.8	–60.8
O(3)–C(3)–C(4)–O(4)	+66.1	+59.0	+58.9
C(2)–C(3)–C(4)–C(5)	+183.5	+175.8	+175.8
C(2)–C(3)–C(4)–O(4)	–52.4	–62.4	–64.5
C(3)–C(4)–C(5)–C(6)	+174.8	+180.2	+183.5
O(4)–C(4)–C(5)–O(5)	+173.5	+176.5	+186.2
C(3)–C(4)–C(5)–O(5)	–62.1	–61.1	–52.8
O(4)–C(4)–C(5)–C(6)	+50.4	+57.8	+62.4
O(5)–C(5)–C(6)–O(6)	–66.9	–64.9	–60.5
C(4)–C(5)–C(6)–O(6)	+53.4	+55.5	+62.9

(a) This article. (b) Berman, Jeffrey & Rosenstein (1968). (c) Kim, Jeffrey & Rosenstein (1968).

Table 7. *Geometry of the hydrogen bonds in DL-mannitol and some short intermolecular O...O contacts*

		O—H	H...O	O...O	O—H...O	Symmetry operation*
1	O(1)—H(O1)...O(2')	0.79 (3) Å	1.93 (3) Å	2.714 (2) Å	173 (3) ^o	545.1
2	O(2)—H(O2)...O(3')	0.91	1.81	2.681	161	465.4
3	O(3)—H(O3)...O(4')	0.83	1.84	2.666	175	545.1
4	O(4)—H(O4)...O(5')	0.77	1.94	2.673	159	565.4
5	O(5)—H(O5)...O(6')	0.80	1.92	2.704	167	545.1
6	O(6)—H(O6)...O(1')	0.83	1.89	2.711	172	555.3

Intermolecular contacts less than 3.5 Å

		Symmetry operation
O(3)...O(2')	3.366 (2)	545.1
O(5)...O(4')	3.265 (2)	545.1

* The symmetry operation is performed on atom O'. The first set of numbers specifies the lattice translations, e.g. 564.2 is $+b - c$ from 555.2. The last digit refers to one of the following symmetry operations: (1) x, y, z ; (2) $-x, -y, \frac{1}{2} + z$; (3) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$; (4) $\frac{1}{2} + x, \frac{1}{2} - y, z$.

participate and a four-link closed loop involving four hydrogen bonds. In the two forms both the spiral and the loop involve the same O atoms, but the donor-acceptor sequence in the loops runs in opposite directions.

References

- BERMAN, H. M., JEFFREY, G. A. & ROSENSTEIN, R. D. (1968). *Acta Cryst.* **B24**, 442–449.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- HUNTER, F. D. & ROSENSTEIN, R. D. (1968). *Acta Cryst.* **B24**, 1652–1660.
- JEFFREY, G. A. & KIM, H. S. (1970). *Carbohydr. Res.* **14**, 207–216.
- KIM, H. S. & JEFFREY, G. A. (1969). *Acta Cryst.* **B25**, 2607–2613.
- KIM, H. S., JEFFREY, G. A. & ROSENSTEIN, R. D. (1968). *Acta Cryst.* **B24**, 1449–1455.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 151–152.
- XRAY system (1972). Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland; implemented and extended by the Dutch X-ray System Group (1973).