References

Borgen, O. & Mestvedt, B. (1966*a*). *LSFIV*4, Technical Report 45. Inst. for Fysikalsk Kjemi, NTH, Trondheim.

Borgen, O. & Mestvedt, B. (1966*b*). *FOUAL* 3, Technical Report 46. Inst. for Fysikalsk Kjemi, NTH, Trondheim.

Chothia, C. & Pauling, P. (1969). *Chem. Comm.* p. 746.

Hamilton, W. C. (1964). *Statistics in Physical Science*. New York: Ronald Press.

Hanson, H. P., Herman, F., Lea, J. D. & Skillman, S. (1964). *Acta Cryst.* **17**, 1040.

Hess, H. (1969). *Acta Cryst.* B**25**, 2338.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Pauling, P. (1968). In *Structural Chemistry and Molecular Biology*, p. 555. Edited by A. Rich & N. Davidson. San Francisco: Freeman.

Senko, M. E. & Templeton, D. H. (1960). *Acta Cryst.* **13**, 281.

Singh, P. & Ahmed, F. R. (1969). *Acta Cryst.* B**25**, 1901.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

Sutton, L. E. (1965). *Tables of Interatomic Distances*. Supplement. Special Publication No 18. London: The Chemical Society.

Tolbert, B. M., Adams, P. T., Bennett, E. L., Hughes, A. M., Kirk, M. R., Lemmon, R. M., Noller, R. M., Ostwald, R. & Calvin, M. (1953). *J. Amer. Chem. Soc.* **75**, 1867.

Acta Cryst. (1971). B**27**, 1323

The Structure of α-Lactose Monohydrate

By C. A. Beevers and Hans N. Hansen*

Chemistry Department, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, *Scotland*

(*Received* 18 *May* 1970)

α-Lactose monohydrate, $C_{12}H_{22}O_{11}.H_2O$, is monoclinic with $a=7\cdot815$, $b=21\cdot567$, $c=4\cdot844$ Å, $\beta=106\cdot2°$. The structure has been found by Patterson methods and refined to an R value of 15%. There is one intramolecular O–O distance of 2·82 Å and there are no less than sixteen hydrogen bonds linking one lactose molecule to its neighbours in the crystal.

Introduction

We have now completed work on the crystal structure of α-lactose monohydrate, $C_{12}H_{22}O_{11}.H_2O$. Lactose occurs in the milk of mammals and in the pollen of the Forsythia plant. It is a reducing sugar which hydrolyses into equal quantities of glucose and galactose. In the α form it can be more completely described as α-4-(β-D-galactopyranosido)-D-glucopyranose. The molecule thus contains two six-membered rings with a 1,4 linkage and its conformation and numbering are shown in Fig. 1. X-ray photography was carried out with a Weissenberg camera of 10 cm diameter using normal-beam methods with copper $K\alpha$ radiation from a Raymax continuously evacuated tube. One crystal of dimensions about 0·8 mm was used for all photographs. Layers 0–3 were photographed about the a axis, 0–10 about the b axis, and 0–2 about the c axis.

Crystal data

The $0k0$ reflexions were absent for k odd. Since the molecules are optically active in solution they are likely to be of the one form and thus the space group must be $P2_1$. Crystal system: Monoclinic, $a=7\cdot815$ $b=21\cdot567$ $c=4\cdot844$ Å, $\beta=106\cdot2°$. It is thought that these dimensions are correct to $\pm0\cdot2$% and the angle to 0·2°. The values in Groth (1910) are not considered to be very accurate. Our values were obtained by extrapolation of high order spots. $D_x=1\cdot52$, $D_m=1\cdot53$ g.cm^{-3}, cell contents $2(C_{12}H_{22}O_{11}.H_2O)$.

Determination of the structure

The intensities of the reflexions were estimated visually, corrected for Lorentz and polarization factors, and all brought to a common scale. 1379 intensities were collected in all. Patterson projections were computed using a procedure to sharpen the peaks to 'point atoms at rest' and a three-dimensional sharpened Patterson map was also calculated. A model was made of the value of the function on the surface of a sphere of radius 2·5 Å, this vector distance being of special importance in the sugars (Beevers & Ehrlich, 1959). Having decided on the vectors for each ring it was possible to build up the molecule with the correct orientation. The position of this molecule in the cell was found by consideration of the Harker section at $y=\frac{1}{2}$. Taking chance peaks into account as well as the true Harker peaks a trial structure was found which had one atom

---

* Present address: Royal Veterinary and Agricultural University, Department of General and Inorganic Chemistry, Thorvaldsensvej, DK 1871 København V, Denmark.

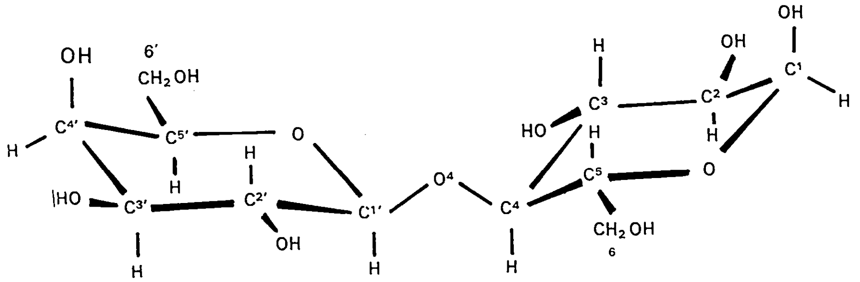

Fig. 1. The molecule of α-lactose from chemical considerations. The glucose moiety is to the right (undashed numbering) the left portion is the galactose.

incorrect. This atom was found from a three-dimensional Fourier map. The water molecule was found from packing considerations. Refinement was by Bhuiya & Stanley's (1963) method, and by least-square calculations using programs written by J. Danielsen and by Trueblood. The final R value reached was 15%, using isotropic vibrational motions and with no hydrogens in the calculations. The final parameters obtained were as in Table 1.

Table 1. *Atomic parameters* ($\times 10^4$) *and thermal parameter*

	x	y	z	B
C(1)	1956	3556	5988	4·35
C(2)	0330	3208	4120	3·50
C(3)	0523	2503	4686	3·81
C(4)	2363	2299	4500	3·65
C(5)	3871	2667	6437	3·82
C(6)	5672	2499	6076	5·12
O(1)	1916	3538	8856	5·34
O(2)	8659	3444	4494	4·18
O(3)	9116	2204	2731	4·76
O(4)	2675	1663	5459	3·38
O(5)	3532	3318	5542	3·97
O(6)	7054	2864	8090	5·11
C(1′)	2283	1187	3365	3·21
C(2′)	3629	0677	4323	3·68
C(3′)	3044	0111	2411	3·80
C(4′)	1152	9943	2090	4·05
C(5′)	0003	0499	1181	3·56
C(6′)	8013	0378	0871	4·30
O(2′)	5306	0896	4297	4·42
O(3′)	4251	9622	3588	4·46
O(4′)	0925	9337	4815	4·35
O(5′)	0524	0992	3173	3·47
O(6′)	6965	0919	0049	4·62
W	5292	4033	1195	4·67

Description and discussion of the structure

This determination shows that in lactose monohydrate both of the pyranose rings are in the strainless *trans* configuration. The galactose unit has the β configuration and the glucose unit the α configuration. Both rings thus have the conformation which Hassel & Ottar (1947) describe as the most favourable. The bond lengths and their estimated standard deviations are shown in Table 2.

The mean C–C bond lengths are 1·53 Å in the glucose unit and 1·51 Å in the galactose. All bond angles at the carbon atoms are within 4° of the tetrahedral angle, and about half of them are within 2°. The angles C–O–C in the rings are 113 and 111°, whilst the angle at the bridge oxygen O(4) is 118°. These agree well with the corresponding angles in other sugars.

The molecules are held together by a large number of hydrogen bonds. Fig. 2 is a projection down the c axis and shows the molecule near the origin and its environment. The hydrogen bond lengths are shown in this Figure. All are of reasonable length (*i.e.* between 2·72 and 3·00 Å) except O(1)–O(4′) at 3·45 Å. This is rather large but is included in order to give O(1) two hydrogen bonds. The OH groups thus all have two intermolecular bonds except that O(2) has three. The hydrogen atoms are undoubtedly on or very near to

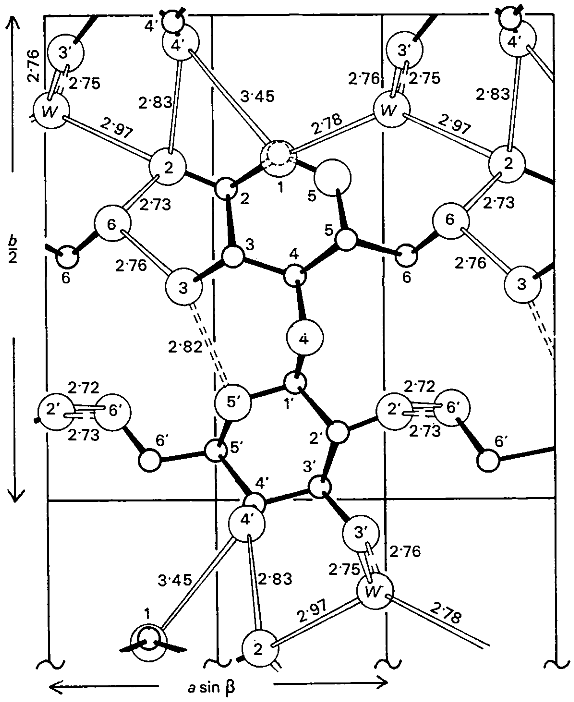

Fig. 2. A projection down the c axis showing one lactose molecule and its surroundings. The bonds within the molecule are shown as black rods, the intramolecular bond is shown dotted, whilst the hydrogen bonds between molecules are shown as white rods. The numbering of the atoms is as in Fig. 1, and the hydrogen bond lengths are given.

Table 2. *Bond lengths Å with their estimated standard deviations*

		e.s.d(× 10^3)			e.s.d(× 10^3)
C(1)–C(2)	1·535	15	C(1′)–C(2′)	1·510	12
C(1)–O(1)	1·399	24	C(2′)–C(3′)	1·550	15
C(2)–C(3)	1·545	8	C(2′)–O(2′)	1·409	16
C(2)–O(2)	1·460	15	C(3′)–C(4′)	1·488	15
C(3)–C(4)	1·531	22	C(3′)–O(3′)	1·423	10
C(3)–O(3)	1·393	17	C(4′)–C(5′)	1·489	9
C(4)–C(5)	1·511	14	C(4′)–O(4′)	1·450	18
C(4)–O(4)	1·448	4	C(5′)–C(6′)	1·543	19
C(5)–C(6)	1·510	23	C(6′)–O(6′)	1·417	10
C(6)–O(6)	1·467	18	C(1′)–O(5′)	1·416	12
C(1)–O(5)	1·406	15	C(5′)–O(5′)	1·417	12
C(5)–O(5)	1·472	7	C(1′)–O(4)	1·415	11

these bonds, but there are a few alternative arrangements possible, and our intensity data are unfortunately not good enough to show the H positions on the Fourier maps. A curious feature of the structure is the distance 2·82 Å from O(3) to O(5′) within the same molecule. Since Fig. 2 is a projection down the short *c* axis the levels of the successive screw axes are lower by 1·13 Å ($\frac{1}{2}a\cos\beta$) as we go to the right. Each molecule is held at $c=4{\cdot}84$ Å from its neighbour by the bonds O(2′)–O(6′), O(3′)–*W*, O(6′)–O(2′), O(3)–O(6), O(6)–O(2). The bonds connecting the molecules in the *a* direction are: O(2′)–O(6′), O(6)–O(2), O(6)–O(3) and O(1)–*W* and O(2)–*W*. In the direction of the *b* axis the bonds holding the molecules together are the bonds to the water molecule (*W*), and also the bonds O(2)–O(4′) and O(1)–O(4′). There are in fact no less than sixteen hydrogen bonds fixing one molecule in place, and these are no doubt sufficient to account for the considerable hardness of the lactose crystal. The water molecule also plays a considerable part in this since it links together oxygens from four different lactose molecules. The bond angles at the water molecule are however rather variable, being from 82 to 126°.

References

BEEVERS, C. A. & EHRLICH, H. (1959). *Z. Kristallogr.* **112**, 414.
BHUIYA, A. K. & STANLEY, E. (1963). *Acta Cryst.* **16**, 981.
GROTH, P. (1910). *Chemische Krystallographie*, Vol. 3, p. 450.
HASSEL, O. & OTTAR, B. (1947). *Acta Chem. Scand.* **1**, 929.

Acta Cryst. (1971). B**27**, 1325

The Crystal and Molecular Structure of 2,3,4,4a,9,9a-Hexahydro-2-methyl-9-phenyl-1*H*-indeno[2,1-*c*]pyridine hydrobromide, $C_{19}H_{22}NBr$

BY F. H. ALLEN, D. ROGERS AND P. G. H. TROUGHTON
Chemical Crystallography Laboratory, Imperial College, London S.W.7, *England*

(*Received* 22 *August* 1968 *and in revised form* 28 *May* 1970)

A full three-dimensional X-ray study of 2,3,4,4a,9,9a-hexahydro-2-methyl-9-phenyl-1*H*-indeno[2,1-*c*]pyridine has been carried out using the hydrobromide, $C_{19}H_{22}NBr$. The crystals are monoclinic, space-group $P2_1/c$, with $a=9{\cdot}853$, $b=15{\cdot}099$, $c=11{\cdot}682$ Å, $\beta=105{\cdot}81°$, and four formula units per cell. The intensities of the 2873 independent reflexions with $\theta\leq 65°$ were measured using a Siemens automatic four-circle single-crystal diffractometer, a coupled ω:2θ scan, and a five-value measuring technique. Bromine was used as a phase-determining heavy atom, and structure solution, using the 2396 reflexions with intensities significantly greater than background, was straightforward. An absorption correction was applied, and block-diagonal least-squares refinement reduced *R* to 0·035. The accuracy of the diffractometer experiment is discussed in some detail. The suggested all-*cis* stereochemistry for the compound has been confirmed, and the geometry of the fused ring system is discussed. A notable feature is the shortness of the N—H---Br bond at 3·124 Å.

Introduction

Catalytic hydrogenation of the antihistamine phenindamine (tetrahydroindenopyridine, I) (Plati & Wenner, 1950), and of the dihydro-1*H*-indeno[2,1-*c*]pyridine (II) (Plati & Wenner, 1955), gave (III) which can be readily isomerized to (IV) using mild alkali (Leeming & Ham, 1968). Correlation of nuclear magnetic reso-