LUXMOORE, A. R. & TRUTER, M. R. (1962). *Acta Cryst.* **15**, 1117–1124.
RIETVELD, H. M. (1966). *Fysica Memo* 153. RCN Petten, The Netherlands.
SANDS, D. E. & DAY, V. W. (1967). *Z. Kristallogr.* **124**, 220–227.
STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1977). B**33**, 3003–3005

The Crystal Structure of Methyl β-D-Galactopyranoside

BY B. SHELDRICK

Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England

(*Received* 17 *February* 1977; *accepted* 18 *March* 1977)

The structure of methyl β-D-galactopyranoside has been determined by direct methods. Refinement was by full-matrix least squares with anisotropic temperature factors for the C and O atoms to produce a final R of 0·040 for 917 measured reflexions. The space group is $P2_12_12_1$ with $Z = 4$, $a = 7{\cdot}7784$ (3), $b = 8{\cdot}5335$ (6), $c = 13{\cdot}1315$ (5) Å. The configuration is 1*e*2*e*3*e*4*a* and hydrogen bonds are formed by each hydroxyl group but the ring O does not act as an acceptor.

Experimental

Cell dimensions and systematic absences ($h00$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$ and $00l$ for $l = 2n + 1$) were established from Weissenberg photographs and the cell dimensions refined by least squares from 2θ measurements of 70 reflexions at room temperature with Cu $K\alpha$ radiation on a diffractometer.

Crystal data

$C_7O_6H_{14}$, FW 194·19, space group $P2_12_12_1$, $a = 7{\cdot}7784$ (3), $b = 8{\cdot}5335$ (6), $c = 13{\cdot}1315$ (5) Å, $V = 871{\cdot}6$ Å^3, $D_x = 1{\cdot}48$ g cm^{-3} for $Z = 4$, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, crystal size: 0·11 × 0·17 × 0·31 mm, crystal shape: prism. Crystallized from 1:10 water:propan-1-ol.

Intensities were collected on an Enraf–Nonius CAD-4 diffractometer in the range $3° < \theta \leq 70°$, scanned in the ω–2θ mode. 917 independent reflexions were measured and 34 more were too weak. The data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) to give the positions of all the non-hydrogen atoms. Refinement with isotropic temperature factors reduced R to 0·166 and with anisotropic factors to 0·084. The positions of the H atoms were established from a difference synthesis and included in the refinement with isotropic temperature factors fixed at the average of the atom to which they are bonded. The final R for the 917 independent measured reflexions is 0·040, and including 34 weak reflexions at one half of the minimum measured intensity R is 0·046. The refinement and

Table 1. *Fractional atom coordinates* (×10^5) *and e.s.d.'s in parentheses*

	x	*y*	*z*
C(1)	16140 (49)	46298 (42)	58422 (25)
C(2)	2010 (47)	34139 (45)	57656 (26)
C(3)	1600 (46)	24583 (45)	67399 (26)
C(4)	19234 (44)	17804 (40)	69986 (24)
C(5)	32494 (47)	31055 (41)	70033 (25)
C(6)	50400 (51)	24992 (46)	72073 (32)
C(7)	27503 (83)	68287 (57)	49717 (41)
O(1)	17819 (40)	54011 (31)	49052 (18)
O(2)	−14296 (34)	41925 (36)	56803 (19)
O(3)	−11048 (38)	12535 (39)	67036 (21)
O(4)	23664 (34)	5979 (28)	62754 (18)
O(5)	32491 (32)	38532 (30)	60268 (17)
O(6)	62233 (35)	37724 (37)	72888 (21)
H(1)	13298 (591)	53486 (526)	64697 (313)
H(2)	3482 (574)	26354 (573)	51222 (360)
H(3)	−787 (663)	32164 (615)	72981 (347)
H(4)	18383 (589)	11967 (566)	76898 (319)
H(5)	28974 (571)	40535 (539)	75472 (308)
H(6)	54114 (582)	17517 (582)	66034 (337)
H(7)	50781 (617)	17743 (562)	78386 (354)
H(8)	−16458 (688)	40476 (593)	51010 (372)
H(9)	−11964 (748)	9444 (678)	61828 (368)
H(10)	28205 (633)	−1020 (608)	65700 (345)
H(11)	66860 (659)	38700 (639)	68049 (349)
H(12)	26211 (761)	73784 (675)	43855 (476)
H(13)	23664 (804)	74361 (691)	54587 (480)
H(14)	40965 (774)	67071 (638)	52938 (435)

Table 2. *Bond lengths* (Å) *and e.s.d.'s in parentheses*

			β-Galactose
C(1)–O(1)		1·400 (4)	1·396 (4)
C(1)–O(5)		1·436 (4)	1·422 (4)
C(2)–O(2)		1·436 (5)	1·432 (4)
C(3)–O(3)		1·424 (5)	1·426 (4)
C(4)–O(4)		1·428 (4)	1·434 (4)
C(5)–O(5)		1·432 (4)	1·440 (4)
C(6)–O(6)		1·428 (5)	1·432 (4)
C(1)–C(2)		1·530 (5)	1·523 (4)
C(2)–C(3)		1·517 (5)	1·524 (4)
C(3)–C(4)		1·527 (5)	1·528 (4)
C(4)–C(5)		1·530 (5)	1·526 (4)
C(5)–C(6)		1·510 (5)	1·512 (4)
O(1)–C(7)		1·435 (6)	
Average bond lengths			
C–O [excluding C(1)–O(1)]		1·430	1·431
C–C		1·523	1·522
C(1)–H(1)	1·055 (43)	O(2)–H(8)	0·789 (49)
C(2)–H(2)	1·081 (48)	O(3)–H(9)	0·736 (50)
C(3)–H(3)	0·995 (49)	O(4)–H(10)	0·794 (50)
C(4)–H(4)	1·037 (44)	O(6)–H(11)	0·735 (47)
C(5)–H(5)	1·113 (44)	C(7)–H(12)	0·907 (61)
C(6)–H(6)	1·058 (46)	C(7)–H(13)	0·876 (62)
C(6)–H(7)	1·035 (47)	C(7)–H(14)	1·134 (60)

Average C–H length 1·029

Fourier syntheses were carried out on the ICL 1906A computer of the Leeds University Centre for Computer Studies with the XRAY 72 program suite (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Scattering factors were taken from *International Tables for X-ray Crystallography* (1962) and unit weights were used. The final coordinates are given in Table 1,* the bond lengths and angles in Tables 2 and 3. A projection down **b** is shown in Fig. 1.

Discussion

The ring configuration is 1*e*2*e*3*e*4*a*, as for β-D-galactose (Sheldrick, 1976), with an average length for the C–C bonds of 1·523 Å and for the C–O bonds [excluding C(1)–O(1)] 1·431 Å. The average standard deviations are 0·005 Å for both values and the C(1)–O(1) value of 1·400 (4) Å is the same as in β-D-galactose, a difference from the average of about 6σ. The system of

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32603 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Bond angles* (°) *and e.s.d.'s in parentheses*

C(1)–O(1)–C(7)	112·76 (31)	C(3)–C(4)–O(4)	109·67 (27)
O(1)–C(1)–O(5)	107·29 (28)	O(4)–C(4)–C(5)	111·23 (27)
O(1)–C(1)–C(2)	108·57 (28)	C(4)–C(5)–O(5)	108·99 (27)
C(2)–C(1)–O(5)	109·62 (28)	C(4)–C(5)–C(6)	111·67 (30)
C(1)–C(2)–C(3)	108·93 (28)	O(5)–C(5)–C(6)	108·16 (29)
C(1)–C(2)–O(2)	109·75 (30)	C(5)–C(6)–O(6)	110·31 (31)
O(2)–C(2)–C(3)	107·20 (28)	C(2)–O(2)–H(8)	101·01 (384)
C(2)–C(3)–C(4)	111·86 (29)	C(3)–O(3)–H(9)	110·89 (446)
C(2)–C(3)–O(3)	111·98 (29)	C(4)–O(4)–H(10)	108·34 (340)
O(3)–C(3)–C(4)	110·77 (30)	C(6)–O(6)–H(11)	109·69 (410)
C(3)–C(4)–C(5)	109·05 (28)		

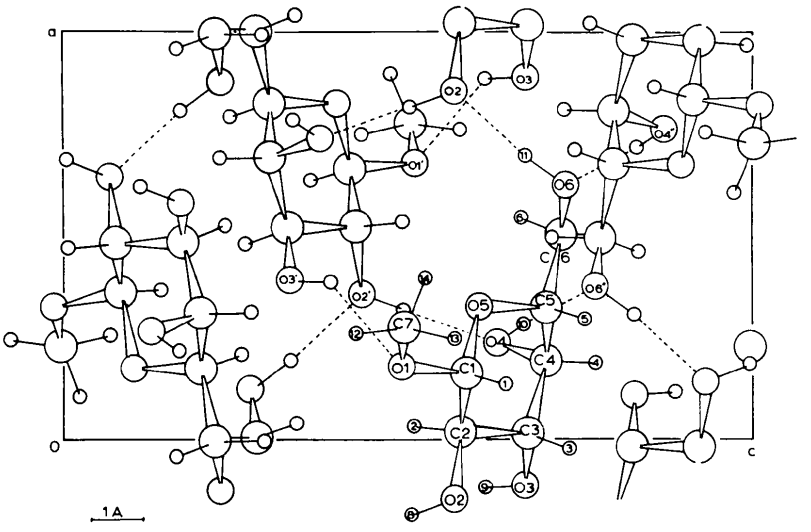

Fig. 1. *b*-axis projection of methyl β-D-galactopyranoside.

Table 4. *Torsion angles* (°)

	Methyl β-D-galacto-pyranoside	β-D-Galactose	Methyl α-D-galacto-pyranoside	α-D-Galactose
C(5)–O(5)–C(1)–O(1)	176·6	177·5	62·1	57·2
C(5)–O(5)–C(1)–C(2)	−65·6	−63·6	−58·0	−61·4
O(5)–C(1)–C(2)–O(2)	174·4	173·4	174·0	−176·4
O(5)–C(1)–C(2)–C(3)	57·3	55·8	52·2	59·1
C(1)–C(2)–C(3)–O(3)	−177·4	−177·2	−173·9	179·5
C(1)–C(2)–C(3)–C(4)	−52·4	−53·0	−52·3	−58·6
C(2)–C(3)–C(4)–O(4)	−69·1	−64·8	−62·8	−68·8
C(2)–C(3)–C(4)–C(5)	52·9	55·9	55·2	55·7
C(3)–C(4)–C(5)–O(5)	−58·0	−60·4	−58·3	−55·8
C(3)–C(4)–C(5)–C(6)	−177·4	−178·0	−178·5	−173·8
C(4)–C(5)–O(5)–C(1)	65·8	65·7	61·5	61·5

hydrogen bonds is shown in Fig. 1, each bond represented by a dashed line. O(2), O(4) and O(6) act as both donors and acceptors but O(1) and O(3) act only as acceptor and donor, respectively, and the ring O(5) forms no hydrogen bond. Three of the hydrogen bonds are between the same atoms as in β-galactose, *i.e.* O(2) → O(4), O(3) → O(1) and O(6) → O(2), but the replacement of the H on O(1) by a methyl group has eliminated the hydrogen bond O(1) → O(6) and the hydrogen bond from O(4) now goes to O(6) in preference to O(3).

Torsion angles of the atoms of the pyranose ring and the non-hydrogen atoms connected to it are given in Table 4, together with the equivalent values for α-D- and β-D-galactose and methyl α-D-galactopyranoside (Gatehouse & Poppleton, 1971). These show some correlation between the difference from the theoretical values of 180° or ±60° and the lengths of the hydrogen bonds to the particular O atoms, *i.e.* the O atoms with the shortest hydrogen bonds tend to have higher disturbances from the theoretical angle, and this perturbation may involve the C of the ring to which the O is bonded. O(4) of methyl β-D-galactopyranoside shares the two shortest H bonds in the structure and the torsion angle is −69·1°, the two torsion angles for C(4) having values of −52·4 and 65·8°. The angle O—H···O appears to have little effect on the torsion angles.

I thank Mr D. Akrigg for technical assistance and the Leeds University Computing Laboratory for the provision of computing facilities.

References

GATEHOUSE, B. M. & POPPLETON, B. J. (1971). *Acta Cryst.* B**27**, 654–660.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

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

SHELDRICK, B. (1976). *Acta Cryst.* B**32**, 1016–1020.

STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.