

been sterically hindered by the methoxy group (Sundaralingam, 1968). The hydrogen-bond distances and angles are given in Table 7.

The author thanks Professor Per Garegg and Professor Peder Kierkegaard for many stimulating discussions, and is also indebted to Dr Don Koenig for revising the English of this article. This investigation has received financial support from the Swedish Natural Science Research Council.

References

- BERMAN, H. M., CHU, S. C. & JEFFREY, G. A. (1967). *Science*, **157**, 1576–1577.
- BORÉN, H. B., GAREGG, P. I., KENNE, L., MARON, L. & SVENSSON, S. (1972). *Acta Chem. Scand.* **26**, 644–652.
- BORÉN, H. B., GAREGG, P. I., KENNE, L., PILOTTI, Å., SVENSSON, S. & SWAHN, C. -G. (1973). *Acta Chem. Scand.* **27**, 2740–2748.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- GAREGG, P. I., LINDBERG, K. B. & SWAHN, C. -G. (1974). *Acta Chem. Scand.* **B28**, 269–270.
- HAMBLING, P. G. (1953). *Acta Cryst.* **6**, 98.
- HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737–1752.
- JEFFREY, G. A. (1973). *Advanc. Chem. Ser.* **117**, 177–196.
- JEFFREY, G. A., POPLE, J. A. & RADOM, L. (1972). *Carbohydr. Res.* **25**, 117–131.
- LINDBERG, K. B., GAREGG, P. J. & SWAHN, C. -G. (1973). *Acta Chem. Scand.* **27**, 380–381.
- MATHIESON, A. MCL. & TAYLOR, J. C. (1961). *Tetrahedron Lett.* **17**, 590–592.
- NORRESTAM, R. (1972). *Acta Cryst.* **A28**, 303–308.
- PULLMAN, B. & PULLMAN, A. (1974). *Advanc. Protein Chem.* **28**, 347–526.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- SUNDARALINGAM, M. (1968). *Biopolymers*, **6**, 189–213.

Acta Cryst. (1976). **B32**, 645

Methyl 6-*O*-Acetyl- β -D-galactopyranoside

BY K. BÖRJE LINDBERG

Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-104 05 Stockholm, Sweden

(Received 18 September 1975; accepted 20 October 1975)

Abstract. C₉H₁₆O₇, orthorhombic, $P2_12_12_1$, $a=26.230$ (6), $b=9.196$ (2), $c=4.718$ (1) Å, $Z=4$. The C(6)–O(6) bond has the unusual *trans-gauche* conformation.

Introduction. In a series of papers (Borén, Garegg, Kenne, Maron & Svensson, 1972; Borén, Garegg, Kenne, Pilotti, Svensson & Swahn, 1973; Lindberg, Garegg & Swahn, 1973; Garegg, Lindberg & Swahn, 1974) the connexion between the circular dichroism of glycoside monoacetates and molecular geometry is discussed. This article describes the determination by X-ray diffraction of the methyl 6-*O*-acetyl- β -D-galactopyranoside crystal structure.

The cell dimensions were obtained from a powder photograph at 20°C, taken in a Guinier–Hägg focusing camera with highly monochromatized Cu $K\alpha_1$ radiation ($\lambda=1.54051$ Å) and KCl ($a=6.29194$ Å; Hambling, 1953) as an internal standard.

A prismatic crystal (0.05 × 0.05 × 0.2 mm) was mounted on a goniometer head approximately along the c axis. Three-dimensional data were collected on a computer-controlled single-crystal diffractometer (Philips PW 1100) with graphite-monochromatized Cu $K\alpha$ radiation. The 1035 available independent data within $\theta < 60^\circ$ were collected with θ – 2θ scans of 2° scan width and background intensities were measured

on each side. The 796 data with $\sigma(I_{\text{net}})/I_{\text{net}} < 0.25$ were considered observable and used in the subsequent calculations. The calculations of $\sigma(I_{\text{net}})$ were based on conventional statistics. Lorentz and polarization factors were applied, but not absorption corrections.

The crystal structure was solved by a computerized application of direct methods (Norrestam, 1972). Successive cycles of anisotropic full-matrix least-squares refinement gave an R value of 0.044 for the observable data*. The positional parameters of the H atoms were then refined with each H given the approximate isotropic temperature factor of the atom to which it is bonded.

Hughes's (1941) weighting scheme was used ($F_{\text{min}}=5.0$). For C and O, the atomic scattering factors of Cromer & Waber (1965) were used, and for H those of Stewart, Davidson & Simpson (1965). The final structural parameters are listed in Tables 1, 2 and 3. The atomic labels are given in Fig. 1. Intermolecular distances and angles are listed in Table 4.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31480 (2 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 of the non-hydrogen atoms

The estimated standard deviations are given in parentheses.

	x	y	z
C(1)	0.3250 (2)	0.2899 (5)	0.8482 (11)
C(2)	0.2988 (2)	0.1439 (5)	0.8111 (11)
C(3)	0.3277 (2)	0.0244 (5)	0.9639 (10)
C(4)	0.3834 (2)	0.0252 (5)	0.8716 (9)
C(5)	0.4054 (2)	0.1745 (5)	0.9144 (11)
C(6)	0.4602 (2)	0.1917 (6)	0.8155 (14)
C(7)	0.3088 (2)	0.5379 (6)	0.7602 (20)
C(8)	0.5382 (2)	0.1343 (5)	1.0287 (12)
C(9)	0.5665 (3)	0.0381 (9)	1.2194 (18)
O(1)	0.3014 (1)	0.3901 (3)	0.6766 (8)
O(2)	0.2480 (1)	0.1518 (4)	0.9146 (7)
O(3)	0.3057 (1)	-0.1152 (4)	0.9318 (8)
O(4)	0.3867 (1)	-0.0148 (4)	0.5792 (7)
O(5)	0.3773 (1)	0.2808 (3)	0.7592 (7)
O(6)	0.4911 (1)	0.1000 (4)	0.9951 (10)
O(7)	0.5569 (2)	0.2296 (6)	0.8969 (16)

Table 2. Anisotropic thermal parameters of the non-hydrogen atoms

The estimated standard deviations are given in parentheses. The temperature factor expression used is $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$.

	β_{11} ($\times 10^4$)	β_{22} ($\times 10^3$)	β_{33} ($\times 10^3$)	β_{12} ($\times 10^4$)	β_{13} ($\times 10^4$)	β_{23} ($\times 10^3$)
C(1)	11 (1)	10 (1)	36 (3)	9 (3)	-11 (7)	-2 (2)
C(2)	11 (1)	10 (1)	27 (2)	8 (3)	0 (7)	-5 (2)
C(3)	13 (1)	9 (1)	22 (2)	-2 (3)	10 (7)	-2 (2)
C(4)	13 (1)	12 (1)	26 (2)	16 (4)	-5 (7)	4 (2)
C(5)	13 (1)	11 (1)	33 (3)	-2 (3)	-3 (7)	1 (2)
C(6)	12 (1)	13 (1)	62 (4)	0 (4)	12 (9)	16 (3)
C(7)	16 (1)	10 (1)	93 (5)	4 (4)	29 (13)	10 (4)
C(8)	13 (1)	15 (1)	43 (3)	-14 (4)	-6 (8)	3 (3)
C(9)	16 (1)	27 (2)	76 (5)	25 (6)	-10 (12)	3 (5)
O(1)	15 (1)	9 (1)	59 (2)	12 (3)	-48 (6)	3 (2)
O(2)	10 (1)	13 (1)	36 (2)	6 (2)	8 (5)	-2 (2)
O(3)	15 (1)	9 (1)	39 (2)	-11 (3)	0 (5)	1 (2)
O(4)	17 (1)	15 (1)	26 (2)	27 (3)	25 (5)	-2 (2)
O(5)	11 (1)	10 (1)	49 (2)	3 (2)	10 (6)	13 (2)
O(6)	11 (1)	17 (1)	91 (3)	-8 (3)	-33 (7)	34 (2)
O(7)	25 (1)	40 (1)	179 (6)	-94 (5)	-180 (12)	65 (5)

Table 3. Hydrogen fractional atomic coordinates and assigned isotropic temperature factors

The estimated standard deviations are given in parentheses.

	x	y	z	B
H(C1)	0.324 (2)	0.316 (4)	1.063 (11)	3.1
H(C2)	0.299 (2)	0.117 (4)	0.619 (10)	2.6
H(C3)	0.326 (2)	0.053 (4)	1.161 (9)	2.4
H(C4)	0.098 (2)	0.039 (5)	0.482 (10)	3.3
H(C5)	0.406 (2)	0.199 (5)	1.108 (11)	3.4
H(C6a)	0.475 (2)	0.289 (5)	0.831 (10)	3.9
H(C6b)	0.463 (2)	0.143 (5)	0.658 (12)	3.9
H(C7a)	0.342 (2)	0.565 (5)	0.730 (11)	4.2
H(C7b)	0.297 (2)	0.550 (6)	0.959 (12)	4.2
H(C7c)	0.285 (2)	0.597 (5)	0.653 (11)	4.2
H(C9a)	0.590 (2)	-0.012 (6)	1.129 (13)	6.1
H(C9b)	0.546 (2)	-0.016 (6)	1.317 (14)	6.1
H(C9c)	0.594 (2)	0.101 (6)	1.319 (13)	6.1
H(O2)	0.229 (2)	0.177 (5)	0.813 (11)	3.6
H(O3)	0.288 (2)	-0.121 (6)	0.839 (12)	3.5
H(O4)	0.401 (2)	-0.101 (5)	0.567 (11)	4.2

Table 4. Intramolecular non-hydrogen bond distances and angles

Estimated standard deviations are given in parentheses.

C(1)-C(2)	1.519 (6) Å	C(1)-O(2)-C(3)	111.0 (4)°
C(2)-C(3)	1.518 (6)	C(2)-C(3)-C(4)	109.9 (4)
C(3)-C(4)	1.523 (6)	C(3)-C(4)-C(5)	109.6 (4)
C(4)-C(5)	1.504 (7)	C(4)-C(5)-O(5)	110.9 (4)
C(5)-C(6)	1.518 (7)	C(5)-O(5)-C(1)	112.6 (3)
C(8)-C(9)	1.465 (10)	O(5)-C(1)-C(2)	110.3 (4)
C(1)-O(1)	1.374 (6)	C(4)-C(5)-C(6)	114.7 (4)
C(7)-O(1)	1.428 (7)	O(5)-C(5)-C(6)	105.1 (4)
C(2)-O(2)	1.421 (5)	C(5)-C(6)-O(6)	106.8 (5)
C(3)-O(3)	1.416 (5)	C(1)-O(1)-C(7)	114.4 (5)
C(4)-O(4)	1.431 (5)	C(6)-O(6)-C(8)	117.9 (4)
C(1)-O(5)	1.437 (5)	O(6)-C(8)-C(9)	114.5 (5)
C(5)-O(5)	1.427 (6)	O(6)-C(8)-O(7)	121.1 (5)
C(6)-O(6)	1.445 (7)	C(9)-C(8)-O(7)	124.1 (5)
C(8)-O(6)	1.285 (5)	O(1)-C(1)-O(5)	107.3 (4)
C(8)-O(7)	1.181 (8)	O(1)-C(1)-C(2)	108.7 (4)
		C(1)-C(2)-O(2)	109.9 (4)
		C(3)-C(2)-O(2)	110.0 (4)
		C(2)-C(3)-O(3)	113.7 (4)
		C(4)-C(3)-O(3)	111.4 (4)
		C(3)-C(4)-O(4)	109.5 (4)
		C(5)-C(4)-O(4)	109.9 (4)

Table 5. Ring torsion angles (°)

O(5)-C(1)-C(2)-C(3)	+34.7
C(1)-C(2)-C(3)-C(4)	-53.1
C(2)-C(3)-C(4)-C(5)	+54.2
C(3)-C(4)-C(5)-O(5)	-58.1
C(4)-C(5)-O(5)-C(1)	+61.5
C(5)-O(5)-C(1)-C(2)	-59.1

Table 6. Least-squares planes and atom deviations

The planes are defined by $Ax + By + Cz + D = 0$, where x , y and z are in Å relative to the axes a , b and c . An asterisk indicates atoms not used in the plane calculation.

C(2)	0.017 Å	A	-0.197
C(3)	-0.017	B	0.462
C(5)	0.018	C	0.865
O(5)	-0.018	D	-2.360
C(1)*	0.654		
C(4)*	-0.678		
O(6)	-0.009 Å	A	-0.225
C(8)	0.028	B	0.617
C(9)	-0.008	C	0.754
O(7)	-0.011	D	1.215
C(6)*	0.056		

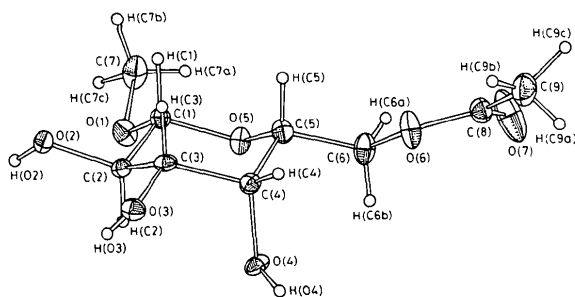


Fig. 1. Perspective view of the molecular structure.

Table 7. *Hydrogen-bond distances and angles*

The estimated standard deviations are given in parentheses.

<i>i</i>	<i>j</i>	<i>k</i>	<i>l</i>	<i>d</i> (<i>jl</i>)	<i>d</i> (<i>kl</i>)	\angle (<i>ijl</i>)	\angle (<i>jkl</i>)
C(2)	O(2)	H(O2)	O(3 <i>a</i>)	2.698 (5) Å	2.09 (5) Å	101.1 (3)°	141 (5)°
C(3)	O(3)	H(O3)	O(2 <i>b</i>)	2.837 (5)	2.24 (6)	113.7 (3)	160 (6)
C(4)	O(4)	H(O4)	O(7 <i>b</i>)	2.779 (6)	1.91 (5)	102.2 (3)	167 (5)

Symmetry operations

	<i>x</i>	<i>y</i>	<i>z</i>
(a)	$\frac{1}{2}-x$	$-y$	$-\frac{1}{2}+z$
(b)	$\frac{1}{2}-x$	$-y$	$\frac{1}{2}+z$
(c)	$1-x$	$-\frac{1}{2}+y$	$\frac{3}{2}-z$

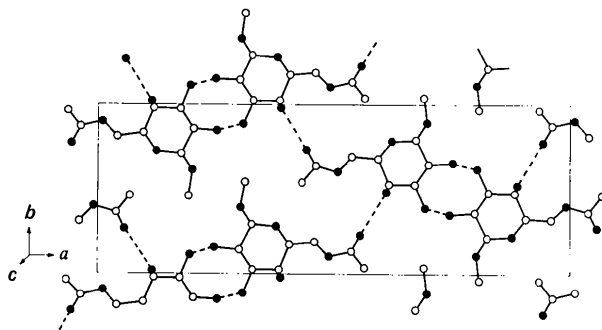


Fig. 2. The projection of the crystal structure along the *c* axis. Filled spheres represent O atoms and unfilled spheres C atoms. Dashed lines indicate hydrogen bonds.

Discussion. As evident in Fig. 1, the pyranose ring has the normal 4C_1 conformation (Table 6). The ring torsion angles, from 53.1° to 61.5° (Table 5), lie within the normal range for strain-free pyranose rings (Jeffrey, 1973). The torsion angle O(5)–C(1)–O(1)–C(7), -82.3° and the short anomeric C(1)–O(1) bond [1.374 (6) Å] are in good agreement with the predictions of Jeffrey, Pople & Radom (1972).

The average C–C and C–O [except the anomeric C(1)–O(1)] bond lengths in the pyranoside moiety are 1.516 and 1.428 Å, respectively, in good agreement with previously reported values for pyranoses (Berman, Chu & Jeffrey, 1967). The torsion angle O(5)–C(5)–C(6)–O(6) is 172.3° (*trans-gauche*). According to Sundaralingam (1968) the three preferred conformations for pyranosides are, in order of increasing energy, *gauche-trans* (60°), *gauche-gauche* (300°) and *trans-gauche* (180°). The *trans-gauche* conformation has been found twice before, *viz.* in 1-kestose (Jeffrey & Park, 1972) and in raffinose pentahydrate (Berman, 1969). C(6) is almost in the plane of the OAc-group (Table 6). The torsion angles C(6)–O(6)–C(8)–C(9) and C(6)–O(6)–C(8)–O(7) are 178.5° and -6.9° , respectively.

The planarity of the ester group was first pointed out by Mathieson & Taylor (1961) and is consonant with theoretical calculations (Pullman & Pullman, 1974). The torsion angle C(5)–C(6)–O(6)–C(8) is -154.2° .

As indicated in Fig. 2, the crystal structure is built up

of non-bonding interactions of the polar O(1)–C(7)H₃ group around the screw axes parallel to the *c* axis (at $\frac{1}{4}$, $\frac{1}{2}$ and $\frac{3}{4}$, 1) and of two hydrogen-bond systems. The first hydrogen-bond system consists of infinite zigzag chains along the *c* axis, involving the O(2) and O(3) atoms serving simultaneously as donors and acceptors. The other system consists of non-catenated hydrogen bonds with O(4) atoms serving as donors and O(7) atoms as acceptors. A list of the hydrogen-bond distances and angles is given in Table 7.

The author thanks Professor Per Garegg and Professor Peder Kierkegaard for many stimulating discussions, and is also indebted to Dr Don Koenig for revising the English of this article. This investigation has received financial support from the Swedish Natural Science Research Council.

References

- BERMAN, H. M. (1969). *Acta Cryst.* B26, 290–299.
 BERMAN, H. M., CHU, S. C. & JEFFREY, G. A. (1967). *Science*, 157, 1576–1577.
 BORÉN, H. B., GAREGG, P. I., KENNE, L., MARON, L. & SVENSSON, S. (1972). *Acta Chem. Scand.* 26, 644–652.
 BORÉN, H. B., GAREGG, P. I., KENNE, L., PILOTTI, Å., SVENSSON, S. & SWAHN, C. -G. (1973). *Acta Chem. Scand.* 27, 2740–2748.
 CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.
 GAREGG, P. I., LINDBERG, K. B. & SWAHN, C. -G. (1974). *Acta Chem. Scand.* B28, 269–270.
 HAMBLING, P. G. (1953). *Acta Cryst.* 6, 98.
 HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* 63, 1737–1752.
 JEFFREY, G. A. (1973). *Advanc. Chem. Ser.* 117, 177–196.
 JEFFREY, G. A. & PARK, Y. J. (1972). *Acta Cryst.* B28, 257–267.
 JEFFREY, G. A., POPLER, J. A. & RADOM, L. (1972). *Carbohydr. Res.* 25, 117–131.
 LINDBERG, K. B., GAREGG, P. J. & SWAHN, C. -G. (1973). *Acta Chem. Scand.* 27, 380–381.
 MATHIESON, A. MCL. & TAYLOR, J. C. (1961). *Tetrahedron Lett.* 17, 590–592.
 NORRESTAM, R. (1972). *Acta Cryst.* A28, 303–308.
 PULLMAN, B. & PULLMAN, A. (1974). *Advanc. Protein Chem.* 28, 347–526.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.
 SUNDARALINGAM, M. (1968). *Biopolymers*, 6, 189–213.