Table 8. *Non-bonded intermolecular contacts less than 3·8 Å involving non-hydrogen atoms*

O(1)···O(6*a*)	3·050 (5) Å	C(7) ··C(9*c*)	3·589 (9) Å
C(6)···O(2*b*)	3·397 (6)	C(4)···O(5*f*)	3·595 (6)
C(7)···O(3*c*)	3·402 (7)	C(6)···O(4*e*)	3·639 (5)
C(2)···O(6*a*)	3·484 (6)	C(6)···O(4*d*)	3·682 (5)
C(8)···O(6*d*)	3·537 (7)	C(9)···O(6*e*)	3·703 (7)
C(5)···O(2*b*)	3·543 (5)	O(2)···O(5*a*)	3·710 (5)
C(3)···O(1*f*)	3·513 (6)	O(4)···O(6*d*)	3·721 (5)

Symmetry operations

–	x,	y,	z
(*a*)	$-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$
(*b*)	$-x$,	$-\frac{1}{2}+y$,	$-\frac{1}{2}-z$
(*c*)	$-x$,	$-\frac{1}{2}+y$,	$\frac{1}{2}-z$
(*d*)	$\frac{1}{2}-x$,	$-y$,	$-\frac{1}{2}+z$
(*e*)	$\frac{1}{2}-x$,	$-y$,	$\frac{1}{2}+z$
(*f*)	x,	y,	$-1+z$

As indicated in Fig. 2, the hydrogen-bond system involving O(2) and O(6) produces a layer structure in the *bc* plane. The layers are held together in the **a** direction by non-bonding interactions. The hydrogen-bonding geometry is given in Table 7 and Table 8 shows the non-bonded contacts.

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

Abrahamsson, S. (1966). *J. Sci. Instrum.* **43**, 931–933.
Berman, H. M., Chu, S. C. & Jeffrey, G. A. (1967). *Science*, **157**, 1576–1577.
Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104–109.
Garegg, P. J., Lindberg, K. B. & Swahn, C. -G. (1974). *Acta Chem. Scand.* B**28**, 381–384.
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.
Jeffrey, G. A., Pople, J. A. & Radom, L. (1974). *Carbohydr. Res.* **38**, 81–95.
Malmros, G. & Werner, P. -E. (1973). *Acta Chem. Scand.* **27**, 493–502.
Norrestam, R. (1972). *Acta Cryst.* A**28**, 303–308.
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). B**32**, 642

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

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. $C_9H_{16}O_7$, monoclinic, $P2_1$, $a=10{\cdot}210$ (3), $b=7{\cdot}2387$ (8), $c=7{\cdot}863$ (2) Å, $\beta=99{\cdot}52$ (2)°, $Z=2$. The ring oxygen serves as acceptor in an axially directed hydrogen bond.

Introduction. In a series of papers (Borén, Garegg, Kenne, Maron & Svensson, 1972; Borén, Garegg, Kenne, Pilotti, Svensson & Swahl, 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-glucopyranoside crystal structure.

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 ($\lambda=1{\cdot}54051$ Å) and KCl ($a=6{\cdot}29194$; Hambling, 1953) as an internal standard.

A prismatic crystal (0·05 × 0·2 × 0·08 mm) was mounted on a goniometer head approximately along the *b* axis. Three-dimensional data were collected on a computer-controlled single-crystal diffractometer (Philips PW 1100) with graphite-monochromatized Cu $K\alpha$ radiation. The 1266 available independent data within $|\theta| < 70°$ were collected with θ–2θ scans of 2° scan width and background intensities were measured on each side. The 1028 data with $\sigma(I_{\rm net})/I_{\rm net} < 0{\cdot}50$ were considered observable and used in the subsequent calculations. The calculations of $\sigma(I_{\rm net})$ were based on conventional counter 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).

Table 1. *Fractional atomic coordinates of the non-hydrogen atoms*

The estimated standard deviations are given in parentheses.

	x	*y*	*z*
C(1)	0·1569 (3)	0·8107 (6)	0·4735 (4)
C(2)	0·0440 (3)	0·8055 (5)	0·3204 (4)
C(3)	0·0914 (3)	0·8996 (5)	0·1685 (4)
C(4)	0·1496 (3)	1·0890 (5)	0·2144 (4)
C(5)	0·2475 (3)	1·0863 (5)	0·3850 (4)
C(6)	0·2840 (5)	1·2820 (6)	0·4464 (5)
C(7)	0·2201 (5)	0·7140 (8)	0·7588 (5)
C(8)	0·4588 (4)	1·2279 (7)	0·6666 (6)
C(9)	0·5060 (7)	1·2365 (15)	0·8626 (8)
O(1)	0·1148 (2)	0·7319 (5)	0·6155 (2)
O(2)	0·0083 (3)	0·6202 (4)	0·2718 (3)
O(3)	−0·0193 (3)	0·9136 (5)	0·0326 (3)
O(4)	0·2186 (2)	0·1546 (5)	0·0835 (3)
O(5)	0·1861 (2)	1·0000*	0·5151 (2)
O(6)	0·3393 (2)	1·2917 (5)	0·6246 (4)
O(7)	0·5267 (3)	1·1693 (7)	0·5706 (5)

* Fixed value.

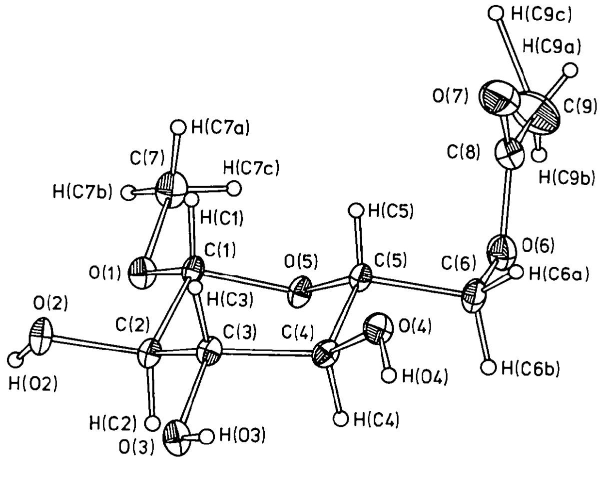

Fig. 1. Perspective view of the molecular structure.

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)	0·244 (3)	0·753 (5)	0·445 (5)	2·56
H(C2)	−0·039 (4)	0·865 (5)	0·342 (4)	2·72
H(C3)	0·162 (3)	0·827 (5)	0·139 (4)	2·47
H(C4)	0·080 (3)	1·163 (5)	0·234 (4)	2·36
H(C5)	0·327 (4)	1·027 (6)	0·382 (5)	2·86
H(C6*a*)	0·342 (4)	1·330 (7)	0·383 (5)	3·93
H(C6*b*)	0·208 (4)	1·360 (6)	0·439 (5)	3·93
H(C7*a*)	0·301 (4)	0·677 (7)	0·729 (5)	3·93
H(C7*b*)	0·188 (4)	0·648 (7)	0·825 (5)	3·93
H(C7*c*)	0·253 (4)	0·812 (7)	0·797 (5)	3·93
H(C9*a*)	0·572 (7)	1·260 (11)	0·903 (9)	7·43
H(C9*b*)	0·456 (7)	1·275 (11)	0·900 (8)	7·43
H(C9*c*)	0·602 (6)	1·097 (10)	0·871 (7)	7·43
H(O2)	−0·038 (4)	0·599 (6)	0·321 (5)	3·06
H(O3)	0·004 (4)	0·962 (6)	−0·031 (6)	3·12
H(O4)	0·174 (4)	1·207 (7)	0·025 (5)	3·31

Table 4. *Intramolecular non-hydrogen bond distances and angles*

Estimated standard deviations are given in parentheses.

C(1)–C(2)	1·522 (4) Å	C(1)–C(2)–C(3)	108·2 (3)°
C(2)–C(3)	1·522 (5)	C(2)–C(3)–C(4)	112·4 (3)
C(3)–C(4)	1·514 (5)	C(3)–C(4)–C(5)	111·7 (3)
C(4)–C(5)	1·534 (4)	C(4)–C(5)–O(5)	109·4 (3)
C(5)–C(6)	1·522 (6)	C(5)–O(5)–C(1)	110·8 (2)
C(8)–C(9)	1·538 (8)	O(5)–C(1)–C(2)	107·9 (3)
C(1)–O(1)	1·384 (4)	C(4)–C(5)–C(6)	110·8 (3)
C(7)–O(1)	1·428 (5)	O(5)–C(5)–C(6)	107·0 (3)
C(2)–O(2)	1·425 (5)	C(5)–C(6)–O(6)	113·2 (3)
C(3)–O(3)	1·423 (4)	C(1)–O(1)–C(7)	112·4 (3)
C(4)–O(4)	1·421 (4)	C(6)–O(6)–C(8)	115·9 (4)
C(1)–O(5)	1·429 (4)	O(6)–C(8)–C(9)	111·5 (4)
C(5)–O(5)	1·429 (4)	O(6)–C(8)–O(7)	126·2 (5)
C(6)–O(6)	1·423 (5)	C(9)–C(8)–O(7)	122·3 (5)
C(8)–O(6)	1·295 (5)	O(1)–C(1)–O(5)	106·9 (3)
C(8)–O(7)	1·184 (6)	O(1)–C(1)–C(2)	109·4 (3)
		C(1)–C(2)–O(2)	111·2 (3)
		C(3)–C(2)–O(2)	108·1 (3)
		C(2)–C(3)–O(3)	107·8 (3)
		C(4)–C(3)–O(3)	110·2 (3)
		C(3)–C(4)–O(4)	110·6 (3)
		C(5)–C(4)–O(4)	108·0 (3)

Table 2. *Anisotropic thermal parameters of the non-hydrogen atoms* ($\times 10^4$)

The estimated standard deviations are given in parentheses. The temperature factor expression used is:

$$\exp\left[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+hl\beta_{13}+kl\beta_{23})\right].$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	89 (3)	92 (6)	83 (4)	−17 (7)	44 (6)	2 (8)
C(2)	87 (3)	99 (6)	91 (5)	−17 (8)	39 (7)	−3 (8)
C(3)	83 (3)	112 (6)	88 (5)	−3 (8)	44 (6)	−15 (9)
C(4)	79 (3)	115 (6)	93 (5)	13 (8)	48 (6)	35 (9)
C(5)	79 (3)	89 (6)	122 (5)	−16 (7)	31 (7)	5 (9)
C(6)	123 (5)	108 (7)	166 (7)	−39 (9)	3 (9)	1 (11)
C(7)	13 (5)	183 (9)	102 (6)	−29 (11)	−7 (8)	39 (12)
C(8)	89 (4)	176 (8)	299 (10)	−46 (10)	45 (11)	−2 (16)
C(9)	144 (7)	523 (25)	227 (11)	−79 (24)	−87 (14)	−73 (27)
O(1)	103 (2)	147 (5)	86 (3)	−38 (6)	31 (4)	50 (6)
O(2)	138 (3)	110 (5)	96 (4)	−78 (6)	76 (5)	−26 (7)
O(3)	117 (3)	170 (5)	77 (4)	−75 (7)	0 (5)	65 (7)
O(4)	93 (3)	171 (6)	130 (4)	−18 (6)	56 (5)	117 (8)
O(5)	102 (2)	98 (4)	83 (3)	−12 (5)	17 (4)	−3 (6)
O(6)	114 (3)	175 (6)	195 (5)	−46 (7)	31 (6)	−69 (9)
O(7)	116 (4)	330 (10)	341 (9)	−2 (11)	6 (9)	−92 (16)

Successive cycles of anisotropic full-matrix least-squares refinement gave an R value of 0·046 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 ($F_{\min} = 2{\cdot}0$). 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. Intramolecular distances and angles are listed in Table 4.

Discussion. As evident in Fig. 1, the pyranose ring has the normal $^4C_1$ conformation (Table 6). The ring torsional angles, from 46·8° to 70·3° (Table 5), lie outside the normal range for strain-free pyranose rings (Jeffrey, 1973), indicating a strain caused by the ester group. This was not observed for the galactose analogue (Lindberg *et al.*, 1973). The torsional angle O(5)–C(1)–O(1)–C(7), −69·8°, and the short anomeric C(1)–O(1) bond [1·384 (4) 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 1·523 and 1·426 Å, respectively, in good agreement with previously found values for pyranoses (Berman, Chu & Jeffrey, 1967).

The torsion angle O(5)–C(5)–C(6)–O(6) is 42·2° (*gauche–trans*). According to Sundaralingam (1968) this is the preferred conformation for pyranosides. 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 −177·2° and 2·8°, 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 torsional angle C(5)–C(6)–O(6)–C(8) is 72·2°.

As indicated in Fig. 2, the noncatenated hydrogen-bond system O(4)–H(O4)···O(3)–H(O3)···O(2)–H(O2)···O(5) maintains the structure in the *bc* plane and the non-bonding interactions of the polar ester group hold the structure together in the **a** direction. The ring oxygen serves as a hydrogen-bond acceptor. The hydrogen bond is directed axially to the ring oxygen. An equatorial direction would probably have

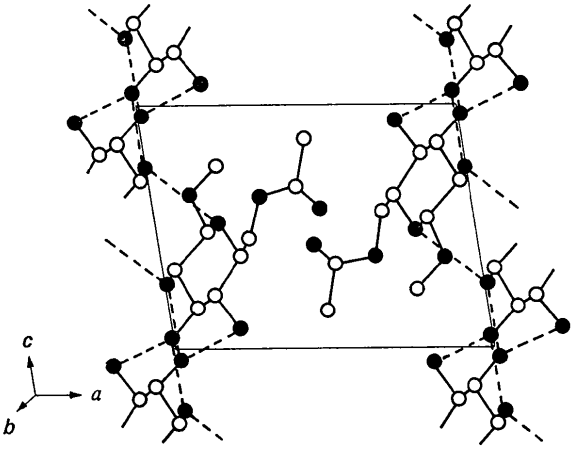

Fig. 2. The projection of the crystal structure along the *b* axis. Filled spheres represent O atoms and unfilled spheres C atoms. Dashed lines indicate hydrogen bonds.

Table 5. *Ring torsion angles* (°)

O(5)–C(1)–C(2)–C(3)	61·9
C(1)–C(2)–C(3)–C(4)	−51·6
C(2)–C(3)–C(4)–C(5)	46·8
C(3)–C(4)–C(5)–C(5)	−51·2
C(4)–C(5)–O(5)–C(1)	64·0
C(5)–O(5)–C(1)–C(2)	−70·3

Table 6. *Least-squares planes and atomic 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 included in the plane calculation.

C(2)	−0·032 Å	*A*	−0·706
C(3)	0·030	*B*	0·706
C(5)	−0·033	*C*	0·064
O(5)	0·034	*D*	−3·990
C(1)*	−0·743		
C(4)*	0·601		
O(6)	−0·0002 Å	*A*	0·3681
C(8)	0·0001	*B*	0·9162
C(9)	0·0001	*C*	−0·1583
O(7)	0·0001	*D*	−9·1361
C(6)*	−0·0618		

Table 7. *Hydrogen-bond distances and angles*

The estimated standard deviations are given in parentheses.

i *j* *k* *l*	*d* (*jl*)	*d* (*kl*)	∠ (*ijl*)	∠ (*jkl*)
C(2)–O(2)–H(O2)···O(5*a*)	2·932 (3) Å	2·25 (4) Å	107·0 (2)°	175 (5)°
C(3)–O(3)–H(O3)···O(2*b*)	2·840 (4)	2·20 (4)	122·2 (2)	157 (4)
C(4)–O(4)–H(O4)···O(3*a*)	2·806 (4)	2·17 (5)	92·3 (2)	151 (4)

Symmetry operations

–	x, y, z
(*a*)	$x, y-\frac{1}{2}, -z$
(*b*)	$-x, y+\frac{1}{2}, -z$

---

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31479 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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.* B**28**, 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.* A**28**, 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). B**32**, 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. $C_9H_{16}O_7$, orthorhombic, $P2_12_12_1$, $a=26\cdot230$ (6), $b=9\cdot196$ (2), $c=4\cdot718$ (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 ($\lambda=1\cdot54051$ Å) and KCl ($a=6\cdot29194$ Å; Hambling, 1953) as an internal standard.

A prismatic crystal ($0\cdot05\times0\cdot05\times0\cdot2$ 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 $\theta<60°$ were collected with θ–2θ scans of 2° scan width and background intensities were measured on each side. The 796 data with $\sigma(I_{\rm net})/I_{\rm net}<0\cdot25$ were considered observable and used in the subsequent calculations. The calculations of $\sigma(I_{\rm net})$ 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 ($F_{\rm min}=5\cdot0$). 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.