Fig. 3. Projection of the unit cell on (001).

The non-bonded intramolecular approach of 2·94 Å between C(11) and O(24) of the bromoacetate group, causes ring B to be slightly flattened, so that C(16) is only 0·51 Å from the central plane, and angle C(9)–C(10)–C(16) assumes the value 120°. This flattening of ring B together with the constraint imposed by closure of the five-membered ring D, results in a distinct distortion of ring C, as demonstrated by the displacements of C(12) and C(15) from the central plane, 0·58 and 0·86 Å respectively. Ring D itself is forced to adopt the 'envelope' conformation, four atoms coplanar and one distinctly out of plane, and the strain in this ring is evidenced by the fact that all internal angles are significantly less than tetrahedral.

Within error bond lengths are normal, other than C(9)–C(10) which is unusually long. There are no inter-molecular approaches likely to affect the molecular stereochemistry.

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The exo Isomer of Methyl 3,4-O-Ethylidene-β-D-galactopyranoside

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Abstract. $C_9H_{16}O_6$, orthorhombic, $P2_12_12_1$, $a = 16·678$ (1), $b = 12·828$ (2), $c = 4·9165$ (9) Å, $Z = 4$.

Introduction. In studies (Garegg, Lindberg & Swahn, 1974) of the extracellular M-antigen, which can be isolated from the mutant Salmonella typhimurium 395 MRO-M1, it was found that there exists an asymmetric acetal carbon of the ethylidene group linked to the terminal D-galactose residue. To elucidate the configuration about the asymmetric carbon, a crystal structure determination of one of the two isomers of methyl 3,4-O-ethylidene-β-D-galactopyranoside was performed.

The cell dimensions were obtained from a powder photograph taken at 25 °C in a Guiner–Hägg focusing camera with monochromatized Cu $K\alpha_1$ radiation ($\lambda = 1·54051$ Å) and KCl ($a = 6·29300$ Å; Hambling, 1953) as an internal standard. The film was measured with a SAAB film-scanner (Abrahamsson, 1966) connected to an IBM 1800 computer. Data were evaluated by the program PILT (Malmros & Werner, 1973).

A prismatic crystal ($0·02 \times 0·03 \times 0·2$ 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 1185 available independent data within $\theta < 60°$ were collected with θ–2θ scan of 2° scan width; background intensities were measured on each side. The 944 data with $\sigma(I_{net})/I_{net} < 0·5$ were considered observable and used in the subsequent calculations. The calculations of $\sigma(I_{net})$ 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). Successive cycles of anisotropic full-matrix least-squares refinement gave an R value of 0·052 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 ($F_{min}=$ 3·0). 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.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31478 (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)	−0·0136 (3)	0·1142 (4)	0·0894 (10)
C(2)	0·0217 (3)	0·2207 (4)	0·0315 (9)
C(3)	0·0793 (3)	0·2156 (4)	−0·2106 (9)
C(4)	0·1324 (3)	0·1184 (4)	−0·2098 (11)
C(5)	0·0944 (3)	0·0205 (3)	−0·0991 (10)
C(6)	0·1543 (3)	−0·0637 (3)	−0·0343 (11)
C(7)	−0·1066 (3)	0·0281 (5)	0·3706 (16)
C(8)	0·2123 (3)	0·2566 (4)	−0·1214 (13)
C(9)	0·2480 (4)	0·3173 (4)	0·1081 (15)
O(1)	−0·0618 (2)	0·1203 (3)	0·3204 (8)
O(2)	−0·0399 (2)	0·2944 (3)	−0·0303 (7)
O(3)	0·1358 (2)	0·2984 (2)	−0·2018 (8)
O(4)	0·1981 (2)	0·1522 (2)	−0·0409 (7)
O(5)	0·0495 (2)	0·0427 (2)	0·1448 (6)
O(6)	0·1164 (2)	−0·1593 (3)	0·0390 (7)

Table 2. *Anisotropic thermal parameters of the non-hydrogen atoms with estimated standard deviations in parentheses (all × 10^4)*

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)	32 (2)	55 (3)	305 (21)	1 (4)	1 (10)	−17 (13)
C(2)	35 (2)	50 (3)	311 (19)	6 (4)	−12 (10)	−18 (13)
C(3)	37 (2)	53 (3)	249 (17)	7 (4)	−12 (9)	46 (12)
C(4)	33 (2)	57 (3)	297 (20)	2 (4)	28 (10)	5 (13)
C(5)	29 (2)	56 (3)	271 (19)	0 (3)	−9 (10)	−24 (12)
C(6)	31 (2)	46 (3)	461 (25)	−4 (3)	−1 (11)	−1 (14)
C(7)	40 (2)	68 (4)	641 (36)	−9 (5)	100 (16)	23 (20)
C(8)	37 (2)	55 (3)	541 (29)	5 (4)	24 (13)	50 (16)
C(9)	42 (2)	64 (4)	839 (40)	−6 (5)	−49 (18)	0 (21)
O(1)	39 (1)	54 (2)	451 (17)	−2 (3)	78 (9)	−14 (11)
O(2)	44 (1)	62 (2)	346 (15)	30 (3)	16 (8)	41 (10)
O(3)	37 (1)	55 (2)	501 (17)	−1 (3)	−8 (8)	69 (11)
O(4)	31 (1)	49 (2)	518 (18)	2 (2)	−25 (8)	41 (10)
O(5)	30 (1)	50 (2)	322 (13)	10 (2)	9 (7)	11 (9)
O(6)	34 (1)	49 (2)	428 (18)	−6 (3)	0 (8)	−21 (10)

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

The estimated standard deviations are given in parentheses.

	x	y	z	β
H(C1)	−0·040 (3)	0·093 (4)	−0·067 (11)	3·36
H(C2)	0·057 (3)	0·242 (3)	0·217 (10)	3·23
H(C3)	0·045 (3)	0·222 (4)	0·602 (10)	3·34
H(C4)	0·151 (3)	0·112 (3)	0·594 (11)	3·57
H(C5)	0·056 (3)	0·002 (4)	−0·258 (10)	3·31
H(C6a)	0·191 (3)	−0·046 (3)	0·125 (10)	3·70
H(C6b)	0·191 (3)	−0·076 (3)	−0·220 (11)	3·70
H(C7a)	−0·132 (3)	0·009 (4)	0·211 (14)	5·03
H(C7b)	−0·071 (3)	−0·022 (4)	0·411 (13)	5·03
H(C7c)	−0·150 (3)	0·051 (4)	0·485 (13)	5·03
H(C8)	0·253 (3)	0·260 (4)	0·716 (12)	4·45
H(C9a)	0·257 (3)	0·389 (4)	0·044 (11)	5·16
H(C9b)	0·307 (3)	0·289 (4)	0·103 (12)	5·16
H(C9c)	0·196 (3)	0·320 (4)	0·268 (11)	5·16
H(O2)	−0·063 (3)	0·316 (4)	0·124 (11)	3·96
H(O6)	0·095 (3)	−0·180 (4)	−0·090 (11)	3·91

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

Estimated standard deviations are given in parentheses.

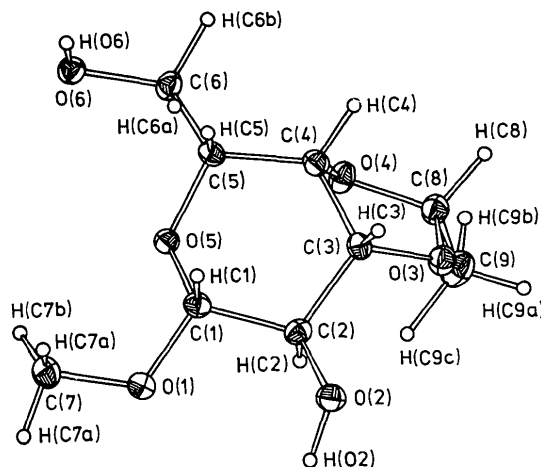|				
C(1)–C(2)	1·515 (6) Å		C(1)–C(2)–C(3)	110·6 (4)°
C(2)–C(3)	1·530 (6)		C(2)–C(3)–C(4)	113·4 (4)
C(3)–C(4)	1·530 (7)		C(3)–C(4)–C(5)	115·8 (4)
C(4)–C(5)	1·510 (7)		C(4)–C(5)–O(5)	110·7 (4)
C(5)–C(6)	1·505 (6)		C(5)–O(5)–C(1)	110·6 (3)
C(8)–C(9)	1·495 (9)		O(5)–C(1)–C(2)	109·3 (4)
C(1)–O(1)	1·393 (6)		C(4)–C(5)–C(6)	113·2 (4)
C(7)–O(1)	1·421 (7)		O(5)–C(5)–C(6)	108·1 (4)
C(2)–O(2)	1·429 (6)		C(5)–C(6)–O(6)	112·1 (4)
C(3)–O(3)	1·420 (5)		C(1)–O(1)–C(7)	113·5 (4)
C(4)–O(4)	1·441 (6)		C(1)–C(2)–O(2)	110·9 (4)
C(1)–O(5)	1·423 (5)		C(3)–C(2)–O(2)	103·3 (4)
C(5)–O(5)	1·442 (6)		C(2)–C(3)–O(3)	111·1 (4)
C(6)–O(6)	1·426 (6)		C(4)–C(3)–O(3)	102·9 (3)
C(8)–O(3)	1·439 (6)		C(2)–C(1)–O(1)	109·1 (4)
C(8)–O(4)	1·416 (6)		O(5)–C(1)–O(1)	107·9 (4)
			C(3)–C(4)–O(4)	101·4 (4)
			C(5)–C(4)–O(4)	111·3 (4)
			C(3)–C(8)–O(4)	106·3 (4)
			O(3)–C(8)–C(9)	111·5 (4)
			O(4)–C(8)–C(9)	110·4 (5)
			C(3)–O(3)–C(8)	108·6 (3)
			C(4)–O(4)–C(8)	104·4 (4)

Fig. 1. Thermal-ellipsoid drawing, with atom labels.

Discussion. As evident in Fig. 1, the pyranose ring has the normal $^4C_1$ conformation (Table 6). The ring torsion angles, ranging from 34·5° to 70·5° (Table 5), lie outside the normal range for strain-free pyranose rings

Table 5. *Torsion angles*

O(5)–C(1)–C(2)–C(3)	58·8°
C(1)–C(2)–C(3)–C(4)	−41·0
C(2)–C(3)–C(4)–C(5)	34·5
C(3)–C(4)–C(5)–O(5)	−43·2
C(4)–C(5)–O(5)–C(1)	61·4
C(5)–O(5)–C(1)–C(2)	−70·5
C(5)–O(5)–C(1)–O(1)	171·1
O(5)–C(1)–O(1)–C(7)	−69·7
O(5)–C(1)–C(2)–O(2)	178·9
C(7)–O(1)–C(1)–C(2)	171·7
O(1)–C(1)–C(2)–C(3)	176·4
O(1)–C(1)–C(2)–O(2)	−63·4
C(1)–C(2)–C(3)–O(3)	−156·3
O(2)–C(2)–C(3)–C(4)	−162·6
O(2)–C(2)–C(3)–O(3)	82·0
C(2)–C(3)–O(3)–C(8)	106·0
C(2)–C(3)–C(4)–O(4)	−86·0
C(3)–O(3)–C(8)–C(9)	−129·4
C(3)–C(4)–C(5)–C(6)	−164·7
C(9)–C(8)–O(4)–C(4)	152·8
C(8)–O(4)–C(4)–C(5)	164·0
O(4)–C(4)–C(5)–C(6)	−49·8
O(4)–C(4)–C(5)–O(5)	71·8
C(4)–C(5)–C(6)–O(6)	−173·0
C(5)–C(4)–C(3)–O(3)	154·6
O(6)–C(6)–C(5)–O(5)	64·0
C(6)–C(5)–O(5)–C(1)	−174·1
C(3)–O(3)–C(8)–O(4)	−9·1
C(3)–C(4)–O(4)–C(8)	−40·3
O(3)–C(8)–O(4)–C(4)	31·8
O(4)–C(4)–C(3)–O(3)	34·1
C(4)–C(3)–O(3)–C(8)	−15·7

(Jeffrey, 1973), indicating a strain caused by the ethylidene group linked to the O(3) and O(4) atoms. The dioxalane ring has a twist conformation (Table 5) with C(4) and O(4) displaced 0·40 and −0·22 Å, respectively, from the plane formed by the atoms C(3), O(3) and C(8) (Table 6).

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 along the axes a, b and c. An asterisk indicates atoms not used in the plane calculation.

C(2)	−0·057 Å	A	0·80
C(3)	0·053	B	0·24
C(5)	−0·057	C	0·54
O(5)	0·061	D	−1·12
C(1)*	−0·709		
C(4)*	0·463		
C(3)	0 Å	A	0·2285
O(3)	0	B	−0·1637
C(8)	0	C	−0·9597
C(4)*	0·403	D	−0·8433
O(4)*	−0·215		

The average C–C and C–O [except the anomeric C(1)–O(1)] bond lengths, 1·514 and 1·427 Å respectively, are in good agreement with previously found values (Berman, Chu & Jeffrey, 1967). The torsion angle O(5)–C(1)–O(1)–C(7), −69·7°, and the short anomeric C(1)–O(1) bond, 1·393 (6) Å, are in good agreement with predictions made by Jeffrey, Pople & Radom (1972, 1974). The torsion angle O(5)–C(5)–C(6)–O(6) is 64·0° (*gauche–trans*). According to Sundaralingam (1968) this is the prefered conformation for pyranoses.

Table 7. *Hydrogen-bond distances and angles*

The estimated standard deviations are given in parentheses.

i j k l	$d\,(jl)$ (Å)	$d\,(kl)$ (Å)	$\angle\,(ijl)$ (°)	$\angle\,(jkl)$ (°)
C(2)–O(2)–H(O2)···O(6a)	2·796 (5)	1·91 (5)	106·6 (3)	172 (5)
C(6)–O(6)–H(O6)···O(2b)	2·870 (5)	2·11 (6)	98·9 (3)	168 (5)

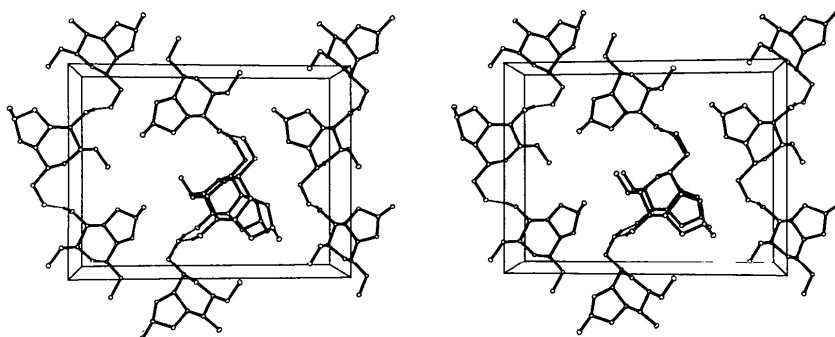| Symmetry operations	x	y	z
None			
(a)	$-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
(b)	$-x$	$-\frac{1}{2}+y$	$-\frac{1}{2}-z$

Fig. 2. Stereoscopic view of the structure down the *c* axis. Hydrogen bonds are drawn as thin lines.

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

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

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

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Abstract. $C_9H_{16}O_7$, monoclinic, $P2_1$, $a = 10·210$ (3), $b = 7·2387$ (8), $c = 7·863$ (2) Å, $\beta = 99·52$ (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 $K\alpha_1$ radia-

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

A prismatic crystal ($0·05 \times 0·2 \times 0·08$ 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 $\sigma(I_{net})/I_{net} < 0·50$ were considered observable and used in the subsequent calculations. The calculations of $\sigma(I_{net})$ 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).