

## X-Ray investigation of benzyl 3,4-anhydro-2-*O*-mesyl-6-*O*-trityl- $\alpha$ -D-altropyranoside: ring conformations in the 3,4-anhydropyranose system

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### ABSTRACT

Single crystals of benzyl 3,4-anhydro-2-*O*-mesyl-6-*O*-trityl- $\alpha$ -D-altropyranoside (**1**) were tetragonal, space group  $P4_1$ , with cell dimensions  $a = 16.393(3)$ ,  $b = 16.393(2)$ ,  $c = 11.952(2)$  Å,  $Z = 4$ . The 3,4-anhydropyranose ring adopts a half-chair ( $^1H_6$ ) conformation. The conformations of 3,4-anhydro- $\alpha$ -D-hexopyranosides with the *allo*, *altro*, *galacto*, and *talo* configurations are discussed.

### INTRODUCTION

Carbohydrate oxiranes undergo a variety of useful reactions and, in general, the stereochemistry of the cleavage of 2,3- and 3,4-anhydropyranosides can be predicted<sup>1</sup>. However, some sugar epoxides, especially 3,4-anhydropyranosides, undergo “abnormal” reactions<sup>2</sup> because of conformational factors. For this reason, the conformational analysis of sugar epoxides has received considerable attention. N.m.r. studies of aldopyranose epoxides<sup>3–14</sup> have indicated half-chair conformations, but X-ray investigations<sup>15</sup> of 2,3-anhydro-4-deoxyhexopyranosides revealed a slightly distorted sofa conformation for the  $\alpha$ -*ribo* epoxide and a half-chair conformation for the *lyxo* isomer.

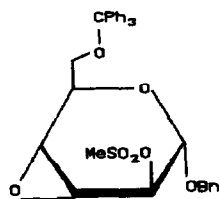
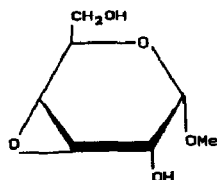
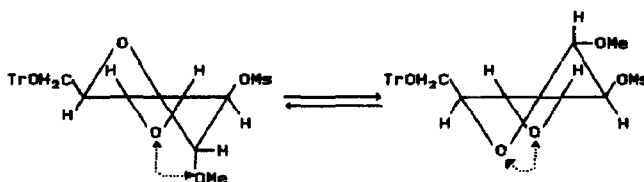
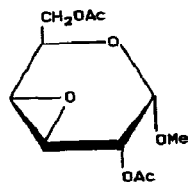
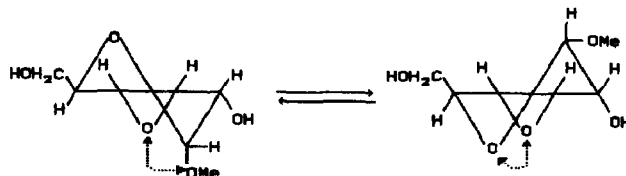
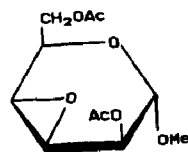
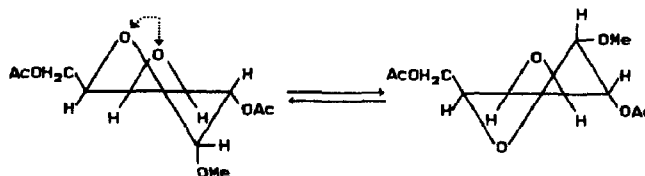
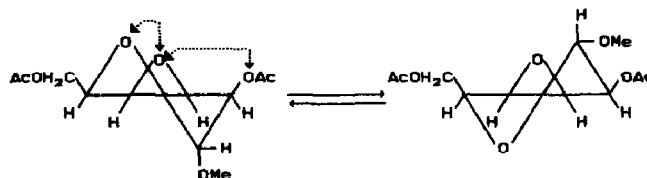
In the 3,4-anhydro- $\alpha$ -D-hexopyranoside series, distorted sofa (*E*) conformations were assigned to the *allo* (**2**)<sup>16</sup> and *talo* (**4**)<sup>17</sup> isomers, whereas the *galacto* (**3**) isomer adopts an almost ideal half-chair (*H*) conformation<sup>18</sup>.

We now report X-ray data for benzyl 3,4-anhydro-2-*O*-mesyl-6-*O*-trityl- $\alpha$ -D-altropyranoside (**1**). The conformational equilibria of **1–4** have also been interpreted in terms of steric and polar interactions of the oxirane ring and the other substituents on the pyranoid ring.

### EXPERIMENTAL

*General methods.* — T.l.c. was performed on Silica Gel G (Merck) and column chromatography on silica gel (40–63  $\mu$ m, Merck). <sup>1</sup>H-n.m.r. spectra were recorded with a Bruker AM-500 (500 MHz) spectrometer for solutions in CDCl<sub>3</sub> and C<sub>6</sub>D<sub>6</sub> (internal

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1  $\alpha$ -D-altro2  $\alpha$ -D-allo3  $\alpha$ -D-galacto4  $\alpha$ -D-talo

Me<sub>4</sub>Si). The optical rotation was measured with a Perkin–Elmer 141 automatic polarimeter.

Compound 1, prepared as described<sup>19</sup>, had m.p. 83–84° (from ether–acetone),  $[\alpha]_D^{22} + 38^\circ$  (*c* 0.5, chloroform);  $\nu_{\max}^{\text{KBr}}$  1365 and 1176  $\text{cm}^{-1}$  (OMs). <sup>1</sup>H-N.m.r. data (CDCl<sub>3</sub>):  $\delta$  7.25–7.45 (m, 20 H, 4 Ph), 5.99 (d, 1 H,  $J_{1,2}$  4.44 Hz, H-2), 4.74 (d, 1 H,  $J$  11.93 Hz, PhCH), 4.67 (d, 1 H, H-1), 4.42 (d, 1 H, PhCH), 4.26 (t, 1 H,  $J_{5,6a}$  4.56 Hz, H-5), 3.53 (d, 1 H,  $J_{2,3}$  0.0,  $J_{3,4}$  3.75 Hz, H-3), 3.47 (dd, 1 H, H-6a), 3.46 (dd, 1 H,  $J_{5,6b}$  4.76,  $J_{6a,6b}$  12.78 Hz, H-6b), 3.26 (d, 1 H,  $J_{4,5}$  0.0 Hz, H-4), 2.96 (s, 3 H, Ms); (C<sub>6</sub>D<sub>6</sub>)  $\delta$ : 7.07–7.47 (m, 20 H, 4 Ph), 5.12 (d, 1 H,  $J_{1,2}$  4.50,  $J_{2,3}$  0.0 Hz, H-2), 4.77 (d, 1 H, H-1), 4.61 (d,  $J$  11.96 Hz, PhCH), 4.23 (d,  $J$  11.95 Hz, PhCH), 4.03 (t,  $J_{5,6a}$  4.26 Hz, H-5), 3.35 (d, 1 H,  $J_{3,4}$  3.88 Hz,

H-3), 3.26 (dd, 1 H,  $J_{5,6b}$  6.98 Hz, H-6a), 3.21 (dd, 1 H,  $J_{6a,6b}$  10.31 Hz, H-6b), 2.81 (d,  $J_{4,5}$  0.0 Hz, H-4), 2.15 (s, 3 H, Ms).

*Anal.* Calc. for  $C_{33}H_{32}O_7S$ : C, 69.22; H, 5.63; S, 5.60. Found: C, 69.13; H, 5.97; S, 5.29.

*X-Ray diffractometry.* — A colourless crystal of **1** ( $0.42 \times 0.21 \times 0.19$  mm) was obtained from ethyl ether–acetone. The reflection intensities were collected on an Enraf–Nonius CAD-4 diffractometer, using graphite-monochromated Cu- $K_\alpha$  radiation (1.54178 Å). The cell constants were obtained from a least-squares refinement on the setting angles of 25 reflections. The data were collected with the  $\omega/2\theta$  scan technique up to  $2\theta_{\max} = 153^\circ$ .

Crystal data:  $C_{33}H_{32}O_7S$ .  $M_r = 572.65$ , tetragonal, space group  $P4_1$ ,  $a = 16.393$  (3),  $b = 16.393$  (2),  $c = 11.952$  (2) Å,  $V = 3211.9$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $F(000) = 572$ ,  $D_x = 1.19$  g.cm<sup>-3</sup>,  $\mu(\text{Cu-}K_\alpha) = 1.65$  mm<sup>-1</sup>.

A total of 3854 reflections were collected, of which 2825 were unique; 2670 reflections were found to be  $I > 2\sigma(I)$ . Lorentz and polarisation corrections were applied to the data. No absorption correction was applied at the measurement stage.

The phase problem was solved by direct methods (program SHELXS-86<sup>20</sup>) in the  $P4_1$  space group. A total of 41 atoms were found in an  $E$ -map giving the expected D enantiomorph. Initially, the positional parameters and individual isotropic temperature factors of all non-hydrogen atoms were refined to  $R = 0.127$  (program SHELX-76<sup>21</sup>). At this stage, an empirical surface-absorption correction (program DIFABS<sup>22</sup>) was performed. For 2670 reflections, the minimum, maximum, and average corrections were 0.587, 1.470, and 0.979, respectively.

The positions of the hydrogen atoms bonded to carbon atoms were generated from assumed geometries, and added with isotropic temperature factors to the set of atomic parameters. The refinement of the atomic positional and thermal anisotropic parameters (isotropic for H) was performed by the least-squares, full-matrix procedure using SHELX-76. The final  $R$  was 0.0594, with  $R_w = 0.0547$ ,  $w = 1.58/\sigma^2(F)$ . The highest peak in the final difference map was 0.22 e/Å<sup>3</sup>. A relatively high thermal parameter for some atoms (sulfone oxygens, O-7 and O-8, and aromatic carbons C-31 and C-32) may be due to some vibrations of remote atomic groups at the ends of side chains in the molecule.

The refined positional parameters for the non-H atoms of **1**, together with their  $B_{eq}$  values, are given in Table I\*.

## RESULTS AND DISCUSSION

Fig. 1 presents the stereo-view of **1** with the crystallographic numbering of the atoms, and Table II contains the calculated bond lengths and valence angles.

\* Lists of structure factors, hydrogen co-ordinates, and anisotropic thermal parameters have been deposited with, and can be obtained from, Elsevier Science Publishers B.V., BBA Data Deposition, P. O. Box 1527, Amsterdam, The Netherlands. Reference should be made to No. BBA/DD/484/*Carbohydr. Res.*, 225 (1992) 1–9.

TABLE I

Fractional co-ordinates ( $\times 10^4$ ) and  $B_{eq}$  values<sup>a</sup> for non-hydrogen atoms<sup>b</sup> for **1**

Atom	x/a	y/b	z/c	$B_{eq}$	Atom	x/a	y/b	z/c	$B_{eq}$
C-1	9677(3)	6519(3)	-1463(5)	4.2(2)	C-14	10978(3)	7348(3)	-4326(5)	4.6(2)
C-2	8921(4)	6856(3)	-943(5)	4.3(2)	C-15	9244(3)	6459(3)	-5996(5)	3.7(2)
C-3	8173(3)	6606(4)	-1573(6)	5.0(2)	C-16	9670(4)	6407(3)	-6992(5)	4.5(2)
C-4	8185(4)	5939(4)	-2376(6)	5.1(2)	C-17	9314(5)	6079(4)	-7944(5)	5.2(2)
C-5	8985(3)	5498(3)	-2567(5)	4.3(2)	C-18	8515(5)	5838(4)	-7913(7)	6.0(3)
O-5	9537(2)	5672(2)	-1666(4)	4.3(1)	C-19	8075(4)	5894(4)	-6952(7)	6.0(2)
O-1	10325(2)	6564(2)	-723(4)	5.0(1)	C-20	8429(4)	6216(3)	-5989(6)	5.2(2)
O-2	9010(2)	7738(2)	-984(3)	5.2(1)	C-21	9350(3)	7753(3)	-4908(5)	3.3(1)
S-2	8601(1)	8270(1)	0 <sup>c</sup>	6.5(1)	C-22	8866(3)	8060(3)	-4079(5)	4.2(2)
O-7	7879(4)	7878(6)	336(6)	11.1(4)	C-23	8672(3)	8902(3)	-4049(6)	4.8(2)
O-8	8568(6)	9053(4)	-487(5)	10.1(4)	C-24	8968(3)	9406(3)	-4851(6)	4.3(2)
C-7	9264(6)	8238(5)	1081(7)	7.8(3)	C-25	9445(3)	9096(3)	-5714(6)	4.4(2)
O-3	7833(2)	5836(3)	-1280(4)	6.0(1)	C-26	9628(3)	8267(3)	-5736(5)	3.9(2)
C-6	9340(4)	5660(3)	-3723(5)	4.3(2)	C-27	11077(4)	6384(4)	-1268(7)	6.2(2)
O-6	9222(2)	6508(2)	-3945(3)	3.7(1)	C-28	11749(5)	6277(4)	-446(8)	5.9(3)
C-8	9607(3)	6845(3)	-4935(5)	3.4(1)	C-29	11677(5)	6350(4)	663(8)	6.5(3)
C-9	10523(3)	6747(3)	-4836(5)	3.5(1)	C-30	12282(7)	6220(6)	1392(11)	8.6(5)
C-10	10934(3)	6049(3)	-5155(5)	4.2(2)	C-31	12952(12)	6000(9)	1071(18)	11.9(9)
C-11	11763(4)	5968(3)	-5020(6)	5.2(2)	C-32	13154(8)	5937(9)	-119(21)	12.1(7)
C-12	12221(4)	6592(4)	-4521(6)	5.8(2)	C-33	12506(6)	6081(6)	-879(10)	9.7(4)
C-13	11820(4)	7277(4)	-4165(6)	5.6(2)					

<sup>a</sup> Calculated from anisotropic thermal parameters as  $B_{eq} = 8 \pi^2 D_u^{1/3}$  where  $D_u$  is the determinant of the  $U$  matrix. <sup>b</sup> In this and subsequent Tables, the values in parentheses are estimated standard deviations. <sup>c</sup> Fixed co-ordinate.

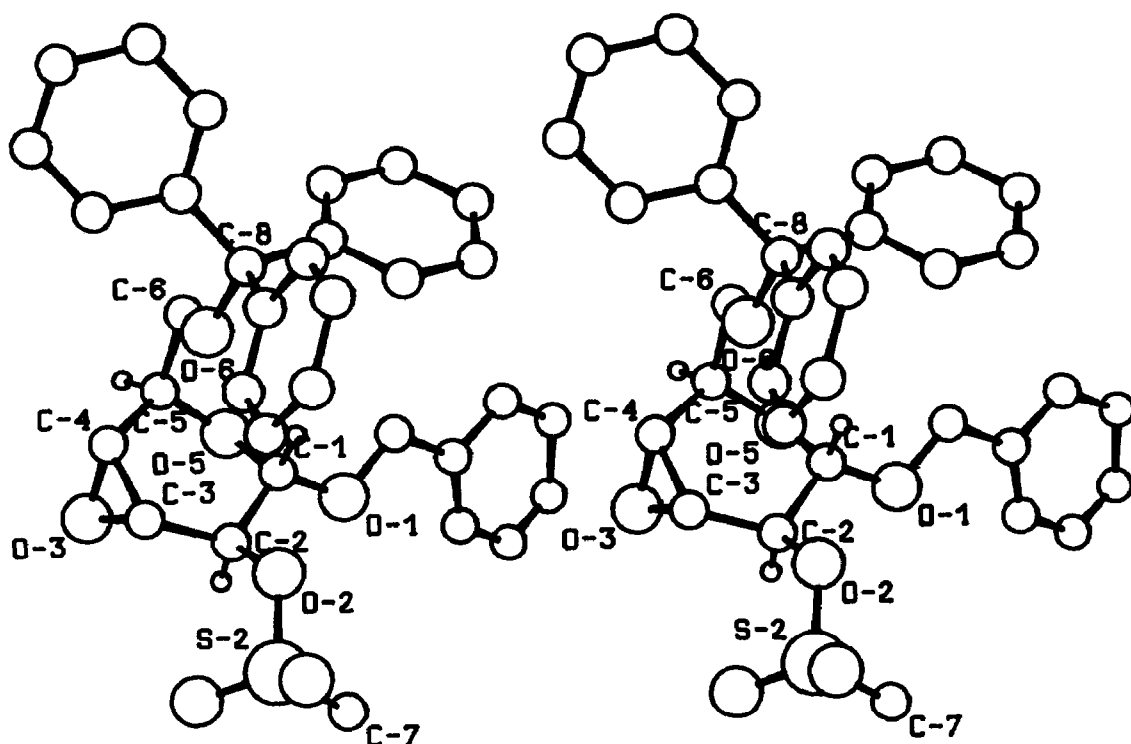


Fig. 1. A stereo-view of 1 with crystallographic numbering of atoms.

*Conformations of 3,4-anhydropyranose rings.* — Conformational analysis of the 3,4-anhydropyranose ring in 1, based on calculation of puckering<sup>23</sup> and asymmetry<sup>24</sup> parameters (see Table III) and torsion angles (Table IV), suggest a “pure” half-chair  $^1H_0$  conformation with BnO-1 equatorial and CH<sub>2</sub>OTr-5 pseudo-axial (*pax*).

This conformation strongly resembles that of methyl 3,4-anhydro- $\alpha$ -D-arabinopyranoside, assigned by Buchanan *et al.*<sup>3</sup> from  $^1H$ -n.m.r. data ( $J_{1,2}$  6.8 Hz) as  $^5H_0$ . Indeed, the chemical shifts and the vicinal coupling constants (in relation to the oxirane ring), calculated on the basis of the modified Karplus equation, are similar for 3,4-epoxides with *arabino* and *altro* configurations, indicating the same conformation<sup>3</sup>. However, it must be emphasised that the calculated coupling constants of the 3,4-epoxide ring protons with the neighbouring protons, *i.e.* H-2 and H-5 which depend on the dihedral angle, have the same values ( $\sim 0$  Hz) for both half-chair conformers ( $^0H_1$  and  $^1H_0$ ) of 1. Therefore, the preponderant conformation of 1 may be deduced only from the  $J_{1,2}$  value which would be expected to be low for the  $^1H_0$  conformer ( $J_{1eq,2peq}$ ) and much larger for the  $^0H_1$  conformer ( $J_{1ax,2pax}$ ). These calculated coupling constants, based on a purely theoretical model, may have only a qualitative value. In contrast, single-crystal X-ray analysis indicates the exact location of all of the non-hydrogen atoms and the precise conformation of the ring.

TABLE II

Non-hydrogen bond lengths (Å) and angles (°)

<i>Bond</i>	<i>Distance</i>	<i>Bond</i>	<i>Distance</i>
C-2-C-1	1.492(8)	C-27-O-1	1.425(8)
O-5-C-1	1.428(6)	O-7-S-2	1.405(8)
O-1-C-1	1.384(7)	O-8-S-2	1.410(7)
C-3-C-2	1.496(8)	C-7-S-2	1.689(9)
O-2-C-2	1.454(6)	O-6-C-6	1.428(6)
C-4-C-3	1.455(10)	C-8-O-6	1.450(7)
O-3-C-3	1.424(8)	C-9-C-8	1.515(7)
C-5-C-4	1.515(8)	C-15-C-8	1.537(8)
O-3-C-4	1.441(8)	C-21-C-8	1.547(7)
O-5-C-5	1.435(7)	C-28-C-27	1.486(11)
C-6-C-5	1.523(8)		
Averaged bond lengths in phenyl rings are 1.383(9) Å.			
<i>Bonds</i>	<i>Angle</i>	<i>Bonds</i>	<i>Angle</i>
C-1-C-2-C-3	111.7(5)	O-7-C-27-C-28	111.3(7)
C-1-C-2-O-2	105.7(4)	O-7-S-2-O-8	120.1(5)
C-2-C-1-O-5	107.3(4)	O-7-S-2-C-7	108.0(4)
C-2-C-1-O-1	110.6(5)	O-2-S-2-C-7	111.6(4)
C-1-O-5-C-5	115.0(4)	C-6-O-6-C-8	117.6(4)
O-5-C-1-O-1	106.5(4)	O-6-C-8-C-9	109.1(4)
C-1-O-1-C-27	111.1(5)	O-6-C-8-C-15	110.4(4)
C-2-C-3-C-4	121.8(5)	O-6-C-8-C-21	103.4(4)
C-2-C-3-O-3	116.1(5)	C-8-C-9-C-10	123.3(5)
C-3-C-2-O-2	109.7(5)	C-8-C-9-C-14	119.7(4)
C-3-C-4-C-5	118.0(5)	C-9-C-8-C-15	113.9(4)
C-3-C-4-O-3	58.9(4)	C-9-C-8-C-21	111.7(4)
C-4-C-3-O-3	60.1(4)	C-8-C-15-C-16	122.7(5)
C-3-O-3-C-4	61.0(4)	C-8-C-15-C-20	118.9(5)
C-4-C-5-O-5	109.8(5)	C-15-C-8-C-21	107.9(4)
C-4-C-5-C-6	112.6(5)	C-8-C-21-C-22	121.9(5)
C-5-C-4-O-3	115.3(5)	C-8-C-21-C-26	118.9(5)
O-5-C-5-C-6	113.9(4)	C-27-C-28-C-29	125.4(8)
C-5-C-6-O-6	106.7(4)	C-27-C-28-C-33	116.4(9)
Averaged bond angles in phenyl rings are 120.1(8)°			

The observed relatively low  $J_{1,2}$  value (4.46 Hz) for **1** suggests significant contributions of both  $^oH_1$  and  $^1H_o$  conformers, whereas the X-ray data (Table III) established a "pure"  $^1H_o$  form. Although different physical states are involved, the molecule usually adopts the same conformation in the crystal and in solution.

The preference for the  $^1H_o$  conformer of **1** is probably due to the steric and electrostatic repulsions of the axial MeO-1 and the oxirane oxygen, which destabilise the  $^oH_1$  conformer. Moreover, the pseudo-axial (*pax*) polar MsO-2 group may disfavour the  $^oH_1$  conformation<sup>25</sup>. These factors outweigh the anomeric effect and force **1** to adopt the  $^1H_o$  conformation. On the other hand, the Reeves  $\Delta^2$  effect of the pseudo-equatorial (*peq*) MsO-2 and equatorial BnO-1 in close proximity (Table V), as well as the dipolar

TABLE III

Conformational parameters of 3,4-anhydropyranose rings for 1-4

	1	2	3	4
<i>Puckering parameters</i>				
Q(Å)	0.513	0.450	0.501	0.461
$\varphi$ (°)	329.5	346.7	336.8	316.9
$\theta$ (°)	50.4	48.7	50.4	50.7
$q_z$ (Å)	0.395	0.338	0.386	0.357
<i>Endocyclic torsion angles (degrees)</i>				
C-1-C-2	-48.0	-50.0	-51.0	-39.0
C-2-C-3	16.1	23.3	19.0	9.0
C-3-C-4	-0.2	-3.4	-0.6	-0.9
C-4-C-5	17.0	10.1	13.6	21.9
C-5-O-5	-54.7	-41.2	-49.1	-54.2
O-5-C-1	72.3	61.5	69.5	64.2
<i>Deviations from least-squares planes (Å)</i>				
	4-atom	5-atom	4-atom	5-atom
O-5	-0.392	—	-0.304	-0.602
C-1	0.381	0.644	0.449	—
O-1	-0.029	-2.035	-1.819	-1.428
O-2	1.002	-0.760	-0.902	1.361
O-3	-1.197	-1.140	1.208	1.147
<i>Asymmetry parameters (degrees)</i>				
$\Delta C_2$	4.8	11.2	4.1	14.0
$\Delta C_5$	—	12.9	—	12.4
<i>Conformation</i>	${}^1H_0$	${}^1E + {}^1H_0$	${}^1H_0$	$E_0 + {}^1H_0$

TABLE IV

Selected torsion angles (°) for 1

Bonds	Angle	Bonds	Angle
C-3-C-2-C-1-O-5	-48.0(6)	O-5-C-5-C-4-C-3	17.0(7)
O-2-C-2-C-1-O-5	-167.3(4)	C-6-C-5-C-4-C-3	-111.0(6)
C-3-C-2-C-1-O-1	-163.8(5)	O-5-C-5-C-4-O-3	-49.7(6)
O-2-C-2-C-1-O-1	76.9(5)	C-6-C-5-C-4-O-3	-177.7(5)
C-5-O-5-C-1-C-2	72.3(6)	C-3-O-3-C-4-C-5	108.7(6)
C-5-O-5-C-1-O-1	-169.3(4)	C-1-O-5-C-5-C-4	-54.7(6)
C-27-O-1-C-1-C-2	-169.5(4)	C-1-O-5-C-5-C-6	72.6(5)
C-27-O-1-C-1-O-5	74.3(5)	O-6-C-6-C-5-C-4	41.4(6)
C-4-C-3-C-2-C-1	16.1(8)	O-6-C-6-C-5-O-5	-84.4(5)
O-3-C-3-C-2-C-1	85.7(6)	C-28-C-27-O-1-C-1	-169.2(5)
C-4-C-3-C-2-O-2	133.0(6)	C-8-O-6-C-6-C-5	171.8(4)
O-3-C-3-C-2-O-2	-157.5(5)	C-9-C-8-O-6-C-6	-61.2(6)
C-5-C-4-C-3-C-2	-0.2(7)	C-15-C-8-O-6-C-6	64.6(5)
O-3-C-4-C-3-C-2	103.9(7)	C-21-C-8-O-6-C-6	179.8(4)
C-5-C-4-C-3-O-3	-104.2(6)	C-10-C-9-C-8-O-6	85.8(7)
C-4-O-3-C-3-C-2	-113.3(6)	C-14-C-9-C-8-O-6	-88.9(6)

TABLE V

Selected non-bonding distances (Å) between oxygen atoms in 1–4

Molecule	O-1-O-2	O-1-O-3	O-2-O-3	O-2-O-5	O-3-O-5
1	2.91	4.31	3.68	3.59	2.84
2	2.90	2.90	3.16	3.64	3.19
3	4.02	4.02	3.67	3.62	2.92
4	4.08	4.08	2.82	3.02	2.86

interactions of the epoxide and pyranoid oxygens and the lack of the anomeric effect in the  $^1H_o$  conformer, could influence the stability of 1, which is reflected in its reactions with nucleophiles<sup>1,2,26,27</sup>.

That 1,3-stereoelectronic interactions are the major factor that govern the conformations of the 3,4-anhydropyranosides is supported by the data for the *allo* isomer 2 where the  $^oH_1$  conformation is disfavoured and a slightly distorted sofa (*E*) conformation was found<sup>16</sup> (Table III). This “new” hybrid conformation may be explained by the destabilising polar interactions of three oxygen atoms (oxirane, pyranoid, and HO-2*pax*) in the other  $^1H_o$  conformation (see formulae).

An *E* conformation is strongly preferred over the  $^oH_1$  conformation in the  $\alpha$ -*talo* isomer 4 (Table III) despite the stabilising anomeric effect and the lack of 1,3-diaxial interactions. Evidently, steric and electrostatic interactions between the dipoles associated with the oxirane and pyranoid ring oxygen atoms, supported by AcO-2*pax* in the  $^oH_1$  conformation, force the molecule into an *E* conformation.

The most stable “pure”  $^oH_1$  conformation in the series of 3,4-anhydro- $\alpha$ -D-hexopyranosides is ascribed<sup>18</sup> to the *galacto* isomer 3. This situation may be attributed to the favourable anomeric effect and the spatial arrangement of AcO-2*peq* and CH<sub>2</sub>OAc-5*peq*, which results in the relative long, non-bonded distances between the oxygen atoms (Table V) and minimises their interactions.

The conclusion to be drawn from the X-ray data is that, in each of the isomeric epoxides 1–4, the stereoelectronic 1,3-diaxial interactions are the major factor that govern the conformation. Other polar interactions associated with the oxygen atoms, *i.e.* between the oxirane and pyranose oxygens, also cause marked destabilisation. The vicinal RO-2*pax* substituent as well as the Reeves effect seem to be more important in the equilibrium of conformers than the effect produced by CH<sub>2</sub>OR-5*pax*.

The additive dipole and pseudo-axial AcO-2 interactions predominate over the anomeric effect (see 4). In contrast, the anomeric effect plays a dominant stabilising role only when one dipole interaction is present in the molecule (see 3).

These conclusions are in reasonable accord with those of Williams<sup>2</sup> on the conformational analysis of 3,4-anhydropyranosides.



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