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AN X-RAY CRYSTALLOGRAPHIC STUDY OF 5-*O*-ACETYL-1,2:3,4-DI-*O*-ISOPROPYLIDENE-α-D-GALACTOSEPTANOSE*

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ABSTRACT

Three-dimensional, single-crystal X-ray diffraction methods were used to determine the solid-state structure of 5-*O*-acetyl-1,2:3,4-di-*O*-isopropylidene-α-D-galactoseptanose. The crystals are tetragonal with cell dimensions: $a = b = 9.571(5)$, $c = 16.944(5)$ Å, $\alpha = \beta = \gamma = 90°$, and $z = 4$. The space group is $P4_1$. The structure was solved by direct methods and refined by least-squares techniques to give a conventional discrepancy-factor, *R*, of 0.040. The seven-membered ring is close to a boat conformation, $^{1,2,5}B$.

INTRODUCTION

5-*O*-Acetyl-1,2:3,4-di-*O*-isopropylidene-α-D-galactoseptanose (**1**) has been the subject of a ^{1}H-n.m.r. spectroscopic study[1,2] which indicated that its solution-state conformation is most likely a twist boat,[1,2] $TB_{3,4}$. As both X-ray and ^{1}H-n.m.r. studies on 1,2:3,4-di-*O*-isopropylidene-α-D-glucoseptanose 5-chloroacetate[3] (**2**) and 5-acetate[4] (**3**) showed that molecules of these compounds adopt the $^{4,5}TC_{6,O}$ conformation in the solid state and probably also in solution, it was of interest to determine the solid-state conformation of **1**.

EXPERIMENTAL

The title compound **1** has been prepared[1] from 5-*O*-benzyl-1,2-*O*-isopropylidene-4-*O*-*p*-tolylsulfonyl-α-D-glucoseptanose by displacement of the *p*-tolylsulfonyloxy group by using sodium benzoate followed by conventional deprotection of O-4 and O-5, acetonation, and acetylation. The details of these transformations will be presented elsewhere[2]. Needle crystals of **1**, m.p. 103–104°, were obtained from a solution in light petroleum (b.p. 60–80°).

Crystal data. — Oscillation and Weissenberg photographs showed the crystals to be tetragonal. Systematic absences among the reflections indicate the space group

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*Dedicated to Professor Stephen J. Angyal on the occasion of his retirement.

to be $P4_1$. The crystal used for data collection was prismatic with sides of 0.225, 0.2, and 0.375 mm in the 100, 010, and 001 directions, respectively. Single-crystal diffractometry yielded the following lattice constants: $a = b = 9.571(5)$ and $c = 16.944(5)$ Å. There are four formula units per unit cell.

Intensity data. — Intensity measurements were carried out on a computer-controlled Siemens A.E.D. diffractometer. Integrated intensities for 1713 independent reflections (θ up to 70°) were measured by using CuKα radiation and a $\theta/2\theta$ scan technique[5]. These intensities were corrected for background and absorption[6], and for Lorentz and polarisation factors. The reflections were assigned standard deviations of $\sigma(I)$, where $\sigma(I) = [I_{\text{scan}} + I_{\text{bkg}}]^{1/2}$. In the absorption correction, a linear absorption-coefficient of 7.387 cm^{-1} was used; this value was obtained from the calculated density (1.06 g cm^{-3}) and the mass absorption-coefficients for CuKα radiation[7].

Structure determination and refinement. — All non-hydrogen atoms were located by direct methods, using the program MULTAN of Germain, Main, and Woolfson (1971). Eventually all hydrogen atoms were located from a Fourier difference-synthesis and the structure was refined by full-matrix, least-squares procedures[8]. The hydrogen-atom positional parameters were included in the refinement, but the

TABLE I

PARAMETERS FOR NON-HYDROGEN ATOMS AND THEIR STANDARD DEVIATIONS

Atom	*x/a*	*y/b*	*z/c*	$\beta_{11}{}^a$	$\beta_{22}{}^a$	$\beta_{33}{}^a$	$\beta_{12}{}^a$	$\beta_{13}{}^a$	$\beta_{23}{}^a$
O-1	0.7607(3)	0.7474(4)	0.4083	153(4)	475(8)	36(1)	123(5)	9(2)	15(2)
O-2	0.5421(3)	0.8228(3)	0.3792(2)	155(3)	311(6)	30(1)	90(4)	−5(1)	−1(2)
O-3	0.3453(2)	0.9437(3)	0.4867(2)	121(3)	195(4)	37(1)	33(3)	−12(1)	−20(1)
O-4	0.3640(2)	0.9354(2)	0.6176(2)	127(3)	145(3)	37(1)	15(2)	1(1)	6(1)
O-5	0.5863(2)	0.8525(2)	0.7140(2)	172(3)	127(3)	30(1)	−15(2)	−10(1)	7(1)
O-6	0.7634(2)	0.8703(3)	0.5243(2)	113(3)	208(4)	40(1)	−15(3)	−6(1)	29(2)
O-7	0.4893(3)	0.6421(3)	0.7289(2)	230(4)	158(3)	47(1)	−44(3)	21(2)	5(2)
C-1	0.6966(4)	0.7642(4)	0.4805(2)	127(4)	203(6)	34(1)	38(4)	1(2)	18(2)
C-2	0.5440(3)	0.8020(4)	0.4619(2)	113(4)	143(4)	32(1)	10(3)	−1(2)	7(2)
C-3	0.4929(3)	0.9350(3)	0.4997(2)	107(4)	118(4)	31(1)	2(3)	−3(2)	8(2)
C-4	0.5043(3)	0.9461(3)	0.5908(2)	128(4)	95(3)	33(1)	−6(3)	−6(2)	1(2)
C-5	0.5930(3)	0.8344(4)	0.6286(2)	132(4)	122(4)	28(1)	−3(3)	−9(2)	6(2)
C-6	0.7463(4)	0.8516(5)	0.6071(2)	125(4)	183(5)	34(1)	−17(4)	−16(2)	20(2)
C-7	0.6699(3)	0.7835(3)	0.3456(2)	119(4)	143(4)	31(1)	16(3)	1(2)	−7(2)
C-8	0.7338(16)	0.9036(11)	0.3061(12)	238(14)	332(21)	167(10)	−23(14)	13(9)	145(14)
C-9	0.6480(7)	0.6541(8)	0.2989(7)	191(7)	409(13)	297(8)	96(8)	−61(7)	−258(9)
C-10	0.2801(3)	0.9915(3)	0.5557(2)	126(4)	119(4)	37(2)	20(3)	−2(2)	−9(2)
C-11	0.2752(5)	1.1493(4)	0.5575(3)	162(5)	133(4)	60(2)	18(4)	−13(3)	−5(3)
C-12	0.1361(4)	0.9293(5)	0.5610(4)	137(5)	136(5)	77(2)	19(4)	4(3)	−15(3)
C-13	0.5319(3)	0.7483(4)	0.7568(2)	98(4)	137(4)	38(1)	19(3)	6(2)	10(2)
C-14	0.5350(5)	0.7798(6)	0.8413(3)	149(5)	244(7)	34(1)	1(5)	−2(2)	10(3)

[a] The values have been multiplied by 10^4. The temperature factor is in the form: $T = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

TABLE II

HYDROGEN-ATOM POSITIONAL PARAMETERS

Atom	*x/a*	*y/b*	*z/c*
H-C-1	0.712(3)	0.680(4)	0.511(2)
H-C-2	0.479(3)	0.713(3)	0.475(2)
H-C-3	0.537(3)	1.013(3)	0.473(2)
H-C-4	0.540(3)	1.036(3)	0.605(2)
H-C-5	0.561(3)	0.737(3)	0.613(2)
H^{I}-C-6	0.800(3)	0.764(4)	0.623(2)
H^{II}-C-6	0.795(3)	0.934(3)	0.631(2)
H^{I}-C-8	0.733(14)	1.021(12)	0.330(10)
H^{II}-C-8	0.828(6)	0.891(7)	0.297(5)
H^{III}-C-8	0.679(6)	0.897(7)	0.270(5)
H^{I}-C-9	0.736(5)	0.651(6)	0.267(4)
H^{II}-C-9	0.639(6)	0.570(7)	0.334(6)
H^{III}-C-9	0.564(5)	0.662(6)	0.264(5)
H^{I}-C-11	0.374(4)	1.179(3)	0.558(2)
H^{II}-C-11	0.242(4)	1.179(4)	0.601(2)
H^{III}-C-11	0.227(4)	1.181(4)	0.512(2)
H^{I}-C-12	0.142(4)	0.847(4)	0.565(3)
H^{II}-C-12	0.081(4)	0.970(4)	0.521(3)
H^{III}-C-12	0.090(4)	0.971(4)	0.608(3)
H^{I}-C-14	0.584(4)	0.717(5)	0.870(2)
H^{II}-C-14	0.596(4)	0.860(5)	0.860(2)
H^{III}-C-14	0.459(4)	0.777(4)	0.864(2)

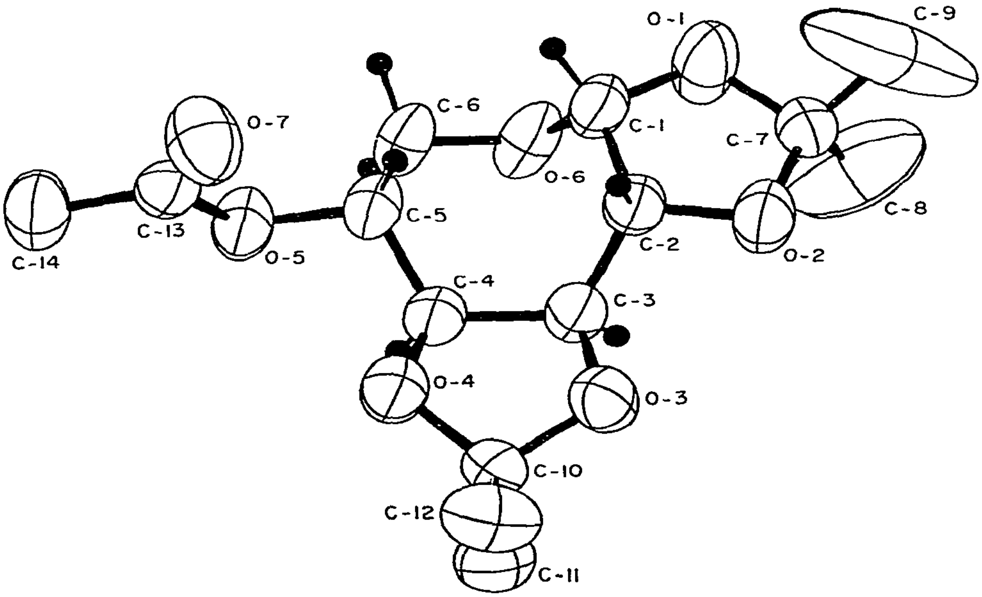

Fig. 1. ORTEP plot of the thermal ellipsoids, showing the atom-numbering system for **1**.

thermal parameters for these atoms were kept fixed at the values of the atoms with which they were most closely associated. An isotropic extinction-parameter[9] was introduced in the final stages of the refinement. Individual weights w_{hkl}, equal to the reciprocal of the square of the estimated standard-deviation of the measurement, $\sigma(I)$, were used throughout the refinement, and the function that was minimised was $\sum w(|F_O| - |F_C|)^2$. A final, conventional discrepancy-factor of 0.040 was obtained. The scattering factors used were those of Doyle and Turner[10], and the anomalous scattering-factors for oxygen atoms were taken from the data of Cromer[11]. The final positional and thermal parameters are given in Tables I and II and the molecular structure and atom-numbering system are shown in Fig. 1.

RESULTS AND DISCUSSION

Bond distances and bond angles. — Bond distances and bond angles involving non-hydrogen atoms are given in Tables III and IV respectively. The mean standard-deviation in the bond lengths is 0.005 Å and in the bond angles 0.3°. An interesting feature of the ring C–C bond-distances is that C-1–C-2 and C-3–C-4 are significantly greater than the remainder. A similar feature has been found for 5-*O*-acetyl-1,2:3,4-di-*O*-isopropylidene-α-D-glucoseptanose (3), in which the seven-membered-ring C–C bonds that are also involved in the dioxolane rings are greater than the other C–C bonds[4].

Of the various C–O bonds, C-1–O-1 is significantly shorter than other C–O single bonds. A similar effect was noted[3] for the structure of 5-*O*-chloroacetyl-1,2:3,4-di-*O*-isopropylidene-α-D-glucoseptanose, and is in keeping with the generally short C-1–O bonds in aldopyranosides, for which the C-5–O-5–C-1–O-1 torsional angle is approximately 180°. The phenomenon has been accounted for by molecular-orbital calculations on dimethoxymethane[12,13]. It is of interest to compare this region of the structure with the corresponding region in 4,5-di-*O*-acetyl-3-*O*-methyl-α-D-guloseptanose[14] (4). The conformation of 4 is such that the O-1–C-1–O-6–C-6

TABLE III

INTERATOMIC DISTANCES AND STANDARD DEVIATIONS (Å)

Bond	*Distance*	*Bond*	*Distance*	*Bond*	*Distance*
C-1–C-2	1.537(5)	C-2–O-2	1.415(4)	O-4–C-10	1.426(4)
C-2–C-3	1.507(5)	O-1–C-7	1.415(4)	C-10–C-11	1.511(5)
C-3–C-4	1.551(5)	O-2–C-7	1.401(4)	C-10–C-12	1.504(6)
C-4–C-5	1.508(5)	C-7–C-8	1.464(9)	C-5–O-5	1.459(4)
C-5–C-6	1.521(6)	C-7–C-9	1.485(10)	O-5–C-13	1.339(4)
C-6–O-6	1.423(4)	C-3–O-3	1.432(4)	C-13–O-7	1.193(5)
O-6–C-1	1.411(5)	C-4–O-4	1.421(4)	C-13–C-14	1.464(6)
C-1–O-1	1.378(5)	O-3–C-10	1.402(5)		

TABLE IV

INTERATOMIC ANGLES AND STANDARD DEVIATIONS (DEGREES)

Bond	*Angle (°)*	*Bond*	*Angle (°)*
O-6–C-1–C-2	111.7(3)	O-3–C-10–C-12	109.2(3)
O-6–C-1–O-1	110.4(4)	O-3–C-10–O-4	103.9(2)
C-2–C-1–O-1	105.6(4)	C-11–C-10–C-12	111.5(5)
C-1–O-1–C-7	111.4(3)	C-11–C-10–O-4	112.3(3)
O-1–C-7–C-8	106.2(3)	C-12–C-10–O-4	108.9(4)
O-1–C-7–C-9	106.5(6)	C-10–O-4–C-4	105.6(3)
O-1–C-7–O-2	107.3(3)	O-4–C-4–C-3	104.3(3)
C-8–C-7–C-9	118.1(3)	O-4–C-4–C-5	110.1(3)
C-9–C-7–O-2	108.5(5)	C-3–C-4–C-5	114.5(3)
C-8–C-7–O-2	109.9(5)	C-4–C-5–O-5	108.2(3)
C-7–O-2–C-2	110.7(3)	C-4–C-5–C-6	111.4(3)
C-1–C-2–O-2	104.4(3)	O-5–C-5–C-6	105.5(3)
C-1–C-2–C-3	114.8(3)	C-5–O-5–C-13	117.8(3)
O-2–C-2–C-3	107.3(3)	C-14–C-13–O-5	111.6(4)
C-2–C-3–C-4	117.3(3)	O-7–C-13–O-5	123.6(3)
C-2–C-3–O-3	107.7(3)	C-14–C-13–O-7	124.8(3)
C-4–C-3–O-3	102.6(3)	C-5–C-6–O-6	111.1(3)
C-3–O-3–C-10	109.3(3)	C-6–O-6–C-1	112.1(3)
O-3–C-10–C-11	110.9(4)		

TABLE V

TORSIONAL ANGLES AND STANDARD DEVIATIONS (DEGREES)

Bond	*Angle (°)*	*Bond*	*Angle (°)*
O-6–C-1–C-2–C-3	−5.4(5)	O-3–C-3–C-4–O-4	9.6(4)
C-1–C-2–C-3–C-4	−56.3(4)	C-3–C-4–O-4–C-10	−29.5(3)
C-2–C-3–C-4–C-5	12.3(5)	C-4–O-4–C-10–O-3	38.8(3)
C-3–C-4–C-5–C-6	68.3(4)	O-4–C-10–O-3–C-3	−32.9(3)
C-4–C-5–C-6–O-6	−48.6(5)	C-10–O-3–C-3–C-4	14.2(4)
C-5–C-6–O-6–C-1	−52.6(5)	O-2–C-2–C-3–O-3	73.2(4)
C-6–O-6–C-1–C-2	88.9(4)	C-7–O-1–C-1–O-6	−115.1(6)
O-1–C-1–C-2–O-2	−8.3(5)	O-1–C-1–O-6–C-6	−153.9(4)
C-1–C-2–O-2–C-7	8.0(5)	O-7–C-13–O-5–C-5	0.1(5)
C-2–O-2–C-7–O-1	−4.9(4)	C-13–O-5–C-5–C-6	−122.7(3)
O-2–C-7–O-1–C-1	−0.9(8)	C-13–O-5–C-5–C-4	118.0(3)
C-7–O-1–C-1–C-2	5.7(6)		

torsional angle is 63°, that is, the acetal geometry is similar to that in an α-D-aldopyranoside. The C-1–O-1 bond-length in **4** is 1.401 Å, which is close to the mean value found for α-D-aldopyranosides[12].

Bond angles within the septanose ring are all greater than 109.5°. Such large angles have also been found for all of the septanose compounds that have been studied thus far: **2** (ref. 3), **3** (ref. 4), **4** (ref. 14), methyl α-D-glucoseptanoside tetraacetate[15], ethyl 2,3:4,5-di-*O*-isopropylidene-1-thio-β-D-glucoseptanoside[16], 3-*O*-acetyl-1,2:4,5-di-*O*-isopropylidene-α-D-glucoseptanose[17], methyl β-D-glucoseptanoside tetraacetate[18], methyl 2,3:4,5-di-*O*-isopropylidene-α-D-alloseptanoside[19], methyl 2,3:4,5-di-*O*-isopropylidene-α-D-glucoseptanoside[20], and methyl α-D-galactoseptanoside tetraacetate[21].

Energy calculations on cycloheptane showed[22] that, for all of the four conformations corresponding to minima in the energy–geometry profile, bond angles within the ring range from 113 to 118°. In a study that is rather more pertinent to the present work, Bocian and Strauss[23] determined the geometries of the low-energy conformations of oxepan. With the exception of one angle in a twist-boat form, the angles within the rings are equal to or greater than the tetrahedral angle.

MOLECULAR CONFORMATION

A selection of torsional angles is given in Table V.

The septanose ring. — In contrast to the structurally related compound **2** in which the seven-membered ring has a twist-chair conformation in the solid state[3], the conformation of the septanose ring in **1** may be described as a *boat*, $^{1,2,5}B$ (see ref. 24 for nomenclature) in which the pseudoplane of symmetry passes through C-5. Table VI gives the torsional angles of the septanose ring for **1**, together with the torsional angles for the boat-2 conformation[25,26] of oxepan. A comparison of these values shows that the signs of the torsional angles for **1** are correct for a boat conformation, and the magnitudes of the torsional angles in **1** in the region of the molecule C-5, C-6, O-6, C-1, and C-2 agree well with the oxepan values. The torsional angle C-2–C-3–C-4–C-5 is 22° less than the oxepan value, which shows that the septanose ring is flattened in this region. Inspection of molecular models shows that a decrease of the C-2–C-3–C-4–C-5 torsional angle results in a decrease of the C-1–C-2–C-3–C-4 torsional angle and an increase in the C-3–C-4–C-5–C-6 torsional angle. The observed values are consistent with these changes. The effect of the ring-flattening in the C-3–C-4 region is to move the septanose ring-conformation towards the twist-boat, $^{1,2}TB_{3,4}$. This conformation corresponds to the oxepan twist-boat A of Bocian and Strauss[25]; however, the torsional-angle values for that conformation are not available for comparison. We suggest that the septanose ring-conformation can best be described as a boat, $^{1,2,5}B$, flattened in the region of C-3 and C-4. Table VII gives values for displacements of atoms from selected planes. It is of interest to compare the torsional angles of the septanose ring and the displacements of C-1, C-2, and C-5 from the least-squares plane through the other four ring-atoms of **1** with the corresponding values (included in Tables VI and VII) for another seven-membered-ring sugar derivative, namely, methyl 2,3:4,5-di-*O*-isopropylidene-α-D-alloseptanoside (**5**), in which the septanoside ring has been described[19] as having a boat conformation, $_{2,3,6}B$. These values show that the seven-membered rings in **1** and **5** have very similar conformations. This result is noteworthy because, whereas

TABLE VI

COMPARISON OF OBSERVED RING-TORSIONAL ANGLES (DEGREES) FOR **1**, AND THOSE COMPUTED FOR THE BOAT-2 CONFORMATION OF OXEPAN AND THE OBSERVED VALUES FOR **5**

Bond	*Observed for* **1** *(°)*	*Computed for oxepan, boat-2 (°)*	*Observed for* **5** *(°)*
C-1–C-2	−5	−2	−5 (C-2–C-3)
C-2–C-3	−56	−74	60 (C-3–C-4)
C-3–C-4	12	34	−16 (C-4–C-5)
C-4–C-5	68	56	−65 (C-5–C-6)
C-5–C-6	−49	−53	49 (C-6–O-6)
C-6–O-6	−53	−47	40 (O-6–C-1)
O-6–C-1	89	86	−69 (C-1–C-2)

TABLE VII

DISTANCES OF ATOMS FROM LEAST-SQUARES PLANES (Å)

Atom		*Septanose*		
		1		**5**
C-1	1.086	−0.003[a]		−0.071[a]
O-1		0.005[a]		
C-7		−0.005[a]		
O-2		0.003[a]		
C-2	1.173	0.129		1.034
C-3	0.083[a]		0.000[a]	1.159
O-3			0.000[a]	
C-10			0.326	
O-4			−0.229	
C-4	−0.089[a]		0.000[a]	0.066[a]
C-5	0.671			−0.078[a]
C-6	0.095[a]			0.600
O-6	−0.089[a]			0.083[a]

[a] Atoms included in calculation of the plane.

the 1,2-*O*-isopropylidene ring in **1** occupies the favourable "equatorial" disposition on the septanose ring, the 2,3-*O*-isopropylidene ring in **5** occupies the "axial" disposition on the septanoside ring (that is, it is *endo* to the seven-membered ring).

The dioxolane rings. —The dioxolane ring involving O-1 is unusual in that none of the torsional angles is greater than 8.3°, which implies that the ring is close to being planar. In a survey[27] of the geometry of more than forty 2,2-dimethyl-1,3-dioxolane rings, it was found that only the 1,2-dioxolane ring in **1** appeared to be nearly planar. Examination of the thermal parameters for C-8 and C-9 (see also Fig. 1) shows that these methyl groups librate to a much greater extent than the methyl groups on C-10. Similarly, O-1 and, to a lesser extent, O-2 show abnormally large thermal parameters. It would appear, thus, that the flatness of this dioxolane ring is more apparent than real, the final shape representing the mean of several morepuckered rings. The large thermal parameters of C-8 and C-9 also account for the shortness of the C-7–C-8 and C-7–C-9 bonds.

The other dioxolane ring, involving C-3 and C-4, exists as a twist form in which C-10 and O-4 are displaced on opposite sides of the three-atom plane O-3–C-3–C-4 (see Table VII).

The acetoxyl group. — The torsional angles for the acetoxyl group show that this is planar, with the carbonyl group eclipsing C-5–H-5, an arrangement that is commonly observed for *O*-acetyl groups[21].

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