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The Crystal Structure of 1,6-Anhydro-3,4-*O*-isopropylidene-β-D-talopyranose

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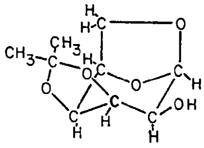
The crystal structure of 1,6-anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose, $C_9H_{14}O_5$, [$P2_12_12_1$, $a=20{\cdot}191$ (5), $b=8{\cdot}220$ (2), $c=5{\cdot}717$ (2) Å, $Z=4$], has been determined from 1017 X-ray reflections measured with Cu $K\alpha$ radiation on an automatic diffractometer and refined to an $R=0{\cdot}045$. The anhydro and isopropylidene rings are on the same side of the pyranose ring, so that the molecule has an overall 'tub' conformation. The pyranose ring is a distorted $^3C_0$ chair. Small differences are observed in the C–O bond lengths of the anhydride ring, similar to those observed in other anhydro sugars. The molecules are packed by van der Waals interactions with one weak intermolecular hydrogen bond with a H···O distance of 2·3 Å.

Introduction

This work was undertaken as part of a structural study of the fused ring sugars. Related compounds previously studied by crystal structure analysis are: 2,7-anhydro-D-altro-heptulose monohydrate (sedoheptulosan, Brown & Thiessen, 1969); 1,6-anhydro-*β*-D-glucopyranose (levoglucosan, Park, Kim & Jeffrey, 1971); 1,6:2,3-dianhydro-*β*-D-gulopyranose (Berking & Seeman, 1971); 1,6-anhydro-*β*-D-mannofuranose (Lechat & Jeffrey, 1972); 2,6-anhydro-*β*-D-fructopyranose (Dreissig & Lugar, 1972); and 1,2:4,5-di-*O*-isopropylidene-*β*-D-fructopyranose (Takagi, Shiono & Rosenstein, 1973).

Single crystals of 1,6-anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose, (I), $C_9H_{14}O_5$, were provided by Professor H. Paulsen of the Institute of Organic Chemistry and Biochemistry, University of Hamburg.



(I)

Experimental

The unit-cell constants, given in Table 1, and the three-dimensional intensity data consisting of 1171 reflections were measured with a Nonius CAD-4 automatic diffractometer from a crystal with dimensions 0·3 × 0·3 × 0·3 mm, using graphite-monochromated Cu $K\alpha$ radiation and the θ–2θ scan mode to $2\theta=150°$. The monitored reflections 040, 223, and 801 showed a loss of intensity which was linear with the time that the crystal was exposed. The net loss of intensity amounted to 6%. The intensities were therefore corrected for decay according to the intensity decrease of the monitored reflections using a four-point interpolation method (Shiono, 1969). The intensities were corrected for Lorentz and polarization effects. No absorption correction was applied. 146 reflections with $I_{\text{meas}} \le 2\sigma(I_{\text{meas}})$ were given zero weight in the refinement.

Table 1. *Crystal data*

1,6-Anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose ($C_9O_5H_{14}$)
M.W. 202·22
Space group $P2_12_12_1$, from systematic absences $h00$, $h=2n+1$; $0k0$, $k=2n+1$; $00l$, $l=2n+1$
$a=20{\cdot}191$ (5), $b=8{\cdot}220$ (2), $c=5{\cdot}717$ (2) Å
$Z=4$, $V=949$ (1) Å^3
$D_m=1{\cdot}431$, $D_x=1{\cdot}416$ g cm^{-3}
$\mu(\text{Cu } K\alpha)=9{\cdot}9$ cm^{-1}

Structure determination

The structure was solved using the multiple-solution symbolic-addition programs of Germain, Main & Woolfson (1970) from 356 reflections with E greater than 1·1. The E map, calculated from the solution with the highest figure of merit, showed 12 of the heavy atoms within the highest peaks, while the two remaining atoms could be identified amongst the lower peaks. This model was refined with isotropic temperature factors, using only the observed reflections. When the R value $(=\sum|F_o-|F_c||/\sum F_o)$ was 0·06, the hydrogen-atom positions were determined from a difference Fourier map and the model was further refined with anisotropic temperature factors using a full-matrix least-squares program. The hydrogen atoms were given the thermal parameters of the atoms to which they are attached and only their coordinates were refined. In the final cycles of refinement, the reflections 400, 110, 310, 120, 310, 420, 211 and 010 appeared to show extinction and were given zero weight. The other reflections were weighted with the function $w=1/(1{\cdot}07+0{\cdot}02F_o+0{\cdot}05F_o^2)$. The final unweighted and weighted agreement indices were $R=0{\cdot}032$, $R_w=(\sum w|F_o-|F_c||^2/\sum wF_o^2)^{1/2}=0{\cdot}045$. Scattering factors used for carbon and oxygen atoms were those of *International Tables for X-ray Crystallography* (1968), while those used for

Table 2. *Atomic positional and thermal coordinates*

The temperature factor expression used was $\exp\,[-10^{-4}(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$. The estimated standard deviations given in parentheses refer to the least significant digit in parameter values.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·3920 (1)	0·3158 (3)	0·3231 (5)	28·8 (7)	114 (3)	269 (8)	9 (1)	8 (2)	8 (4)
C(2)	0·3296 (1)	0·3788 (3)	0·4395 (5)	20·2 (5)	104 (3)	251 (7)	−6 (1)	−5 (2)	−14 (4)
C(3)	0·3241 (1)	0·5644 (3)	0·4318 (4)	14·9 (4)	109 (3)	219 (6)	−1 (1)	−6 (2)	7 (4)
C(4)	0·3617 (1)	0·6459 (3)	0·2315 (4)	22·4 (5)	116 (3)	177 (6)	−1 (1)	−9 (2)	25 (4)
C(5)	0·4218 (1)	0·5460 (3)	0·1539 (4)	25·8 (6)	150 (4)	217 (7)	0 (1)	14 (2)	31 (5)
C(6)	0·4726 (1)	0·5090 (4)	0·3421 (5)	19·6 (5)	174 (4)	349 (9)	10 (1)	14 (2)	49 (6)
C(7)	0·3625 (1)	0·8054 (3)	0·5664 (4)	25·2 (6)	97 (3)	198 (6)	−7 (1)	5 (2)	17 (4)
C(8)	0·4176 (2)	0·8814 (4)	0·7068 (5)	38·1 (9)	138 (4)	288 (9)	−23 (2)	−20 (3)	22 (6)
C(9)	0·2979 (2)	0·8991 (3)	0·5906 (7)	31·2 (8)	100 (3)	495 (13)	4 (1)	27 (3)	10 (6)
O(1)	0·44912 (9)	0·3606 (2)	0·4471 (4)	20·2 (4)	187 (3)	348 (6)	9·3 (9)	0 (1)	85 (4)
O(2)	0·32139 (9)	0·3154 (2)	0·6680 (4)	25·7 (5)	104 (2)	298 (6)	−10·4 (9)	12 (1)	19 (3)
O(3)	0·35395 (8)	0·6386 (2)	0·6323 (3)	22·7 (4)	85 (2)	173 (4)	−4·4 (7)	0 (1)	16 (2)
O(4)	0·38171 (8)	0·7993 (2)	0·3245 (3)	24·8 (4)	114 (2)	201 (5)	−10·3 (8)	0 (1)	40 (3)
O(5)	0·3978 (1)	0·3855 (2)	0·0973 (3)	38·7 (6)	148 (3)	204 (5)	7 (1)	13 (2)	−20 (3)

Table 2 (*cont.*)

	x	y	z
H(O2)	0·346 (2)	0·356 (4)	0·737 (6)
H(1)	0·390 (1)	0·199 (4)	0·313 (5)
H(2)	0·288 (1)	0·344 (3)	0·343 (5)
H(3)	0·277 (1)	0·592 (3)	0·418 (5)
H(4)	0·333 (1)	0·665 (3)	0·091 (5)
H(5)	0·446 (1)	0·590 (4)	0·006 (5)
H(6,1)	0·473 (1)	0·596 (4)	0·458 (6)
H(6,2)	0·518 (1)	0·471 (4)	0·258 (6)
H(8,1)	0·425 (2)	0·996 (4)	0·653 (5)
H(8,2)	0·459 (2)	0·817 (4)	0·676 (6)
H(8,3)	0·406 (2)	0·881 (4)	0·876 (6)
H(9,1)	0·303 (1)	1·015 (4)	0·539 (6)
H(9,2)	0·261 (2)	0·847 (4)	0·489 (7)
H(9,3)	0·283 (2)	0·895 (4)	0·759 (7)

hydrogen atoms are given by Stewart, Davidson & Simpson (1965). The final atomic positional and temperature parameters are given in Table 2, the structure factors are listed in Table 3,* and the interatomic distances and angles in Table 4.

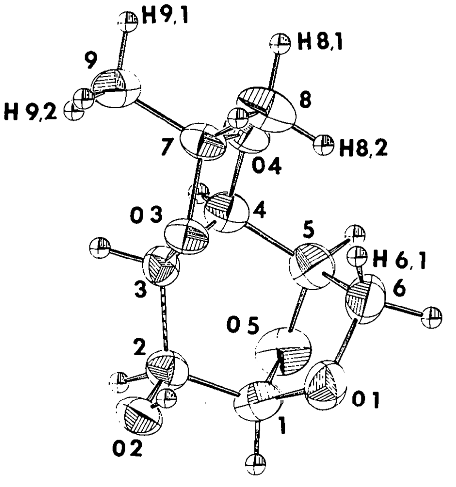


Fig. 1. 1,6-Anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose, showing thermal ellipsoids at 50% probability, except for the hydrogen atoms which are drawn with an arbitrary isotropic temperature factor.

Discussion

The molecule, shown in Fig. 1, has three fused rings; the pyranose ring, C(1)C(2)C(3)C(4)C(5)O(5), the anhydride ring, C(1)O(1)C(6)C(5)O(5), and the isopropylidene ring, C(3)C(4)O(4)C(7)O(3). The last two rings are fused to the pyranose ring and face each other so that the whole molecule has the overall shape of a 'tub', with the anhydro and isopropylidene rings at dihedral angles of about 120° on the same side of the pyranose ring, as shown in Fig. 2(*a*).

The conformation of the rings and their orientation, relative to each other and the crystal lattice, is described by the data for the least-squares planes, given in Table 5. The best four-atom plane for the pyranose ring is *P*1 in which three adjacent C(2)C(3)C(4) and one non-

Table 4. *Interatomic distances and bond angles*

Bond lengths

C(1)–C(2)	1·517 (4) Å	C(1)–H(1)	0·96 (3) Å
C(2)–C(3)	1·531 (3)	C(2)–H(2)	1·04 (3)
C(3)–C(4)	1·529 (3)	C(3)–H(3)	0·98 (3)
C(4)–C(5)	1·532 (3)	C(4)–H(4)	1·00 (3)
C(5)–C(6)	1·518 (4)	C(5)–H(5)	1·04 (3)
C(7)–C(8)	1·507 (4)	C(6)–H(6,1)	0·98 (3)
C(7)–C(9)	1·521 (4)	C(6)–H(6,2)	1·08 (3)
C(1)–O(1)	1·403 (3)	C(8)–H(8,1)	1·00 (3)
C(1)–O(5)	1·417 (3)	C(8)–H(8,2)	1·01 (3)
C(2)–O(2)	1·416 (3)	C(8)–H(8,3)	1·00 (3)
C(3)–O(3)	1·432 (3)	C(9)–H(9,1)	1·00 (3)
C(4)–O(4)	1·427 (3)	C(9)–H(9,2)	1·04 (3)
C(5)–O(5)	1·442 (3)	C(9)–H(9,3)	1·01 (3)
C(6)–O(1)	1·440 (3)	O(2)–H(O2)	0·71 (3)
C(7)–O(3)	1·432 (3)	Mean C–C	1·522
C(7)–O(4)	1·437 (3)	Mean C–O	1·429

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* Table 3 has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30356 (7pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4 (*cont.*)

Non-bonded intramolecular distances

H(6,1)···H(8,1)	3·15 (5) Å	H(6,1)···C(7)	2·88 (4) Å
H(6,1)···H(8,2)	2·22 (5)	H(6,1)···C(8)	2·96 (4)
H(6,1)···H(8,3)	3·17 (5)	O(3)·····O(1)	3·168 (3)
H(6,1)···O(3)	2·62 (3)	O(3)·····O(2)	2·744 (2)
H(6,1)···O(4)	2·59 (3)	O(2)·····O(1)	2·896 (3)

Bond angles

C(1)–C(2)–C(3)	112·8 (2)°	C(4)–C(5)–C(6)	116·0 (2)°
C(1)–C(2)–O(2)	112·1 (2)	C(4)–C(5)–O(5)	106·8 (2)
C(1)–O(1)–C(6)	106·4 (2)	C(4)–O(4)–C(7)	108·3 (2)
C(1)–O(5)–C(5)	101·2 (2)	C(5)–C(6)–O(1)	104·0 (2)
C(2)–C(3)–C(4)	115·0 (2)	C(5)–C(4)–O(4)	110·9 (2)
C(2)–C(3)–O(3)	111·8 (2)	O(3)–C(7)–O(4)	104·6 (2)
C(2)–C(1)–O(5)	109·3 (2)	O(3)–C(7)–C(8)	110·3 (2)
C(2)–C(1)–O(1)	111·9 (2)	O(3)–C(7)–C(9)	110·9 (2)
C(3)–C(2)–O(2)	112·6 (2)	O(4)–C(7)–C(8)	109·2 (2)
C(3)–C(4)–C(5)	112·1 (2)	O(4)–C(7)–C(9)	109·7 (2)
C(3)–C(4)–O(4)	104·4 (2)	C(8)–C(7)–C(9)	112·0 (2)
C(3)–O(3)–C(7)	104·4 (2)	C(2)–O(2)–H(O2)	105·1 (2)

adjacent atom O(5) form an exact plane within 0·003 Å; this differs from the strainless skew form, however, in that the two excluded atoms, C(1) and C(5), are displaced almost equally on the same side of the plane. If these displacements were exactly equal, the pyranose ring would be a strained form with *m* (C_s) symmetry. The conventional four-atom plane for ${}^1C_4$*, *i.e.* C(2), C(3), C(5), O(5), is not planar as shown by *P*2, and the ring is best described by reference to *P*3, which corresponds to a ${}^3C_0$ chair in which C(3) is flattened by 0·5 Å. The five-membered anhydride and isopropylidene rings have envelope conformations (${}^0E$) with the O(5) and O(3) atoms 0·62 and 0·56 Å above the least-squares planes of the other four atoms, as shown by planes *P*4 and *P*5 in Table 5. The torsion angles associated with the pyranose ring are given in Fig. 3, and the ring torsion angles for the five-membered rings in Table 6.

The distortion of the pyranose ring is compared in Table 5 with that in levoglucosan and sedoheptulosan, see Fig. 2. All three molecules have 1,6-anhydro rings, and the displacement of C(3) from the ideal chair is progressively smaller, as shown by comparing planes *P*3, *P*6 and *P*8. In sedoheptulosan, the closest approach to H(6,1) is O(2) at 4·2 Å, there is no steric repulsion and the difference between the displacements of O(5) and C(3) are a consequence of the strain induced by the 1,6-anhydro ring formation. In levoglucosan, because of the axial O(3), this ring conformation would have resulted in a H(6,1) to O(3) separation of 2·3 Å, which is increased to 2·54 Å by a further distortion of the ring by bringing C(3) 0·1 Å closer to the four-atom plane, *P*6.

In the isopropylidene talopyranose molecule, the flattening at C(3) increases the H(6,1) to O(3) separation but decreases that to O(4). The conformation observed is that in which these two non-bonding separations are equal at 2·60 Å. Compared with isopropyli-

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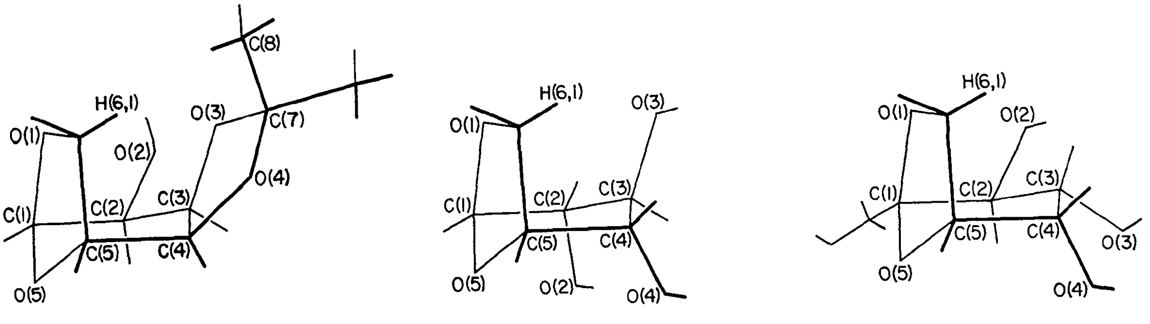
* For conformation nomenclature, see Schwartz (1973).



(*a*) (*b*) (*c*)

Fig. 2. Comparison of the structure of (*a*) 1,6-anhydro-3,4-*O*-isopropylidene-β-D-talopyranose with that of (*b*) 1,6-anhydro-β-D-glucopyranose (levoglucosan; Park, Kim & Jeffrey, 1971) and with that of (*c*) 2,7-anhydro-sedoheptulose monohydrate (sedoheptulosan; Brown & Thiessen, 1969). The numbering system for sedoheptulosan is not the standard one, and is different from that used in the above reference.

dene-fructopyranose (Takagi, Shiono & Rosenstein, 1973), the dihedral angle between the plane of the isopropylidene ring and the pyranose ring $P3/P5$ is greater by 9° as a consequence of the flattening at C(3).

The bond lengths and angles are given in Table 4. The C–C bonds are within 3σ of the mean value of 1·522 Å, the C–O bond lengths show a wider variation with differences from the mean which appear to be systematic when compared with those observed in other anhydro rings, as in Table 7. These observations may be related to those associated with the anomeric effect as discussed by Jeffrey, Pople & Radom (1972).

There is only one hydrogen atom available for hydrogen bonding, H(O2), which forms a weak intermolecular bond to the ring oxygen O(5) of the adjacent molecule displaced by the c axis translation. The H···O distance is 2·3 Å and the O–H···O angle 148°. Two intramolecular approaches of H(O2) to O(1) and O(3) at 2·9 and 2·7 Å (Table 8), with O–H···O angles of 102 and 110° respectively, are not regarded as hydrogen-bond interactions because of the long distances and acute angles. The molecular packing is shown in Fig. 4. The H(O2)–O(5) hydrogen bond links the molecules in rows, parallel to the c axis, which are packed by van der Waals interactions.

From a n.m.r. study in solution of 1,6-anhydro-3,4-*O*-isopropylidene-*β*-D-galactopyranose, which differs from this molecule in that the positions of O(2)–H(O2) hydroxyl and H(2) hydrogen are interchanged (see Fig. 2), Heyns, Koll & Paulsen (1971) have concluded that the pyranose ring approaches the boat conformation. This would increase the O(3)–H(6,1) distance, but it would also decrease the O(4)–H(6,1) distance to about 2·3 Å, and, from this work, appears to be a less favorable conformation than the partially flattened $^3C_0$ chair.

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Table 5. *Least-squares planes in* 1,6-*anhydro*-3,4-*O-isopropylidene-β*-D-*talopyranose and related compounds*

Equation of the planes: $Ax+By+Cz=D$, with A,B,C,D in Å. Distances to the plane are given in Å. Asterisks indicate atoms not included in the calculation of the plane.

1,6-Anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose

	Pyranose ring			Anhydride ring		Isopropylidene ring	
	*P*1	*P*2	*P*3		*P*4		*P*5
C(1)	0·607*	0·700*	−0·020	C(1)	0·008	C(3)	−0·014
C(2)	0·002	0·176	0·017	O(5)	0·621*	C(4)	0·023
C(3)	−0·003	−0·166	0·342*	C(5)	−0·007	O(4)	−0·024
C(4)	0·002	−0·510*	−0·017	C(6)	0·011	C(7)	0·015
C(5)	0·675*	0·177	0·020	O(1)	−0·012	O(3)	0·565*
O(5)	−0·001	−0·188	−0·853*				
A	16·426	14·396	10·352		11·120		17·294
B	0·593	−1·254	2·250		−4·256		−3·544
C	3·299	3·913	4·652		−3·743		1·622
D	7·086	5·813	6·292		1·798		4·319

Interplanar angles

*P*1,*P*2	15·4°	*P*1,*P*3	25·0°	*P*1,*P*5	34·0°	*P*3,*P*5	56·5°
*P*2,*P*3	28·3	*P*1,*P*4	88·1	*P*3,*P*4	113·1	*P*4,*P*5	59·4

1,6-Anhydro-*β*-D-glucopyranose (levoglucosan; Park, Kim & Jeffrey, 1971) — 2,7-Anhydro-sedoheptulose monohydrate (sedoheptulosan; Brown & Thiessen, 1969)

Pyranose ring		Anhydride ring		Pyranose ring		Anhydride ring	
	*P*6		*P*7		*P*8		*P*9
C(1)	0·001	C(1)	0·012	C(1)	−0·002	C(1)	0·013
C(2)	−0·001	O(5)	0·623*	C(2)	0·002	O(5)	0·615*
C(3)	0·460*	C(5)	−0·011	C(3)	0·568*	C(5)	−0·012
C(4)	0·001	C(6)	0·017	C(4)	−0·002	C(6)	0·018
C(5)	−0·001	O(1)	−0·018	C(5)	0·002	O(1)	−0·019
O(5)	−0·820*			O(5)	−0·828*		

Interplanar angles

*P*6,*P*7	110·6°	*P*8,*P*9	111·6°

Table 6. *Torsion angles of the anhydride and isopropylidene rings*

Anhydride ring		Isopropylidene ring	
O(1) C(1) O(5) C(5)	−43·2°	C(3) O(3) C(7) O(5)	−38·4°
C(1) O(5) C(5) C(6)	42·3	O(3) C(7) O(4) C(4)	20·4
O(5) C(5) C(6) O(1)	−27·6	C(7) O(4) C(4) C(3)	4·2
C(5) C(6) O(1) C(1)	2·0	O(4) C(4) C(3) O(3)	−27·0
C(6) O(1) C(1) O(5)	25·4	C(4) C(3) O(3) C(7)	39·9
Pyranose ring			
C(1) C(2) C(3) C(4)	25·2°	C(4) C(5) O(5) C(1)	−79·7°
C(2) C(3) C(4) C(5)	−27·9	C(5) O(5) C(1) C(2)	77·8
C(3) C(4) C(5) O(5)	55·0	O(5) C(1) C(2) C(3)	−50·7

Table 7. C–O *bond lengths in anhydride rings* (Å)

		Mean	Differences from the mean C–O									Torsion angles (°)	
		C–O		θ	Φ							θ	Φ
I	1,6-Anhydro-3,4-*O*-isopropylidene-		C(6)		O(1)		C(1)		O(5)		C(5)		
	β-D-talopyranose (this work)	1·425		+14		−18		−3		+17		25	−43
II	1,6-Anhydro-*β*-D-glucopyranose		C(6)		O(1)		C(1)		O(5)		C(5)		
	(Park, Kim & Jeffrey, 1971)	1·428		+16		−1		−29		+15		25	−44
III	Sedoheptulosan		C(7)		O(4)		C(2)		O(6)		C(6)		
	(Brown & Thiessen, 1969)	1·434		+13		−7		−9		+2		23	−42
IV	1,6:2,3-Dianhydro-*β*-D-gulopyranose		C(6)		O(1)		C(1)		O(5)		C(5)		
	(Berking & Seeman, 1971)	1·421		+16		−9		−16		+10		34	−46
		1·420		+10		−17		−11		+19		33	−46
V	2,6-Anhydro-*β*-D-fructofuranose		C(6)		O(6)		C(2)		O(2)		C(5)		
	(Dreissig & Luger, 1972)	1·444		+6		−5		−18		+18			

Table 8. *Environment of the* H(O2) *atom*

i	j	k	d_{ij}	d_{ik}	d_{jk}	$\angle_{ijk}$
O(2)	H(O2)	O(1)	0·71 (3) Å	2·896 (3) Å	2·67 (3) Å	102 (1)°
O(2)	H(O2)	O(3)		2·744 (2)	2·41 (3)	111 (2)
O(2)	H(O2)	O(5)*		2·956 (3)	2·33 (3)	148 (2)

* Translated by c.

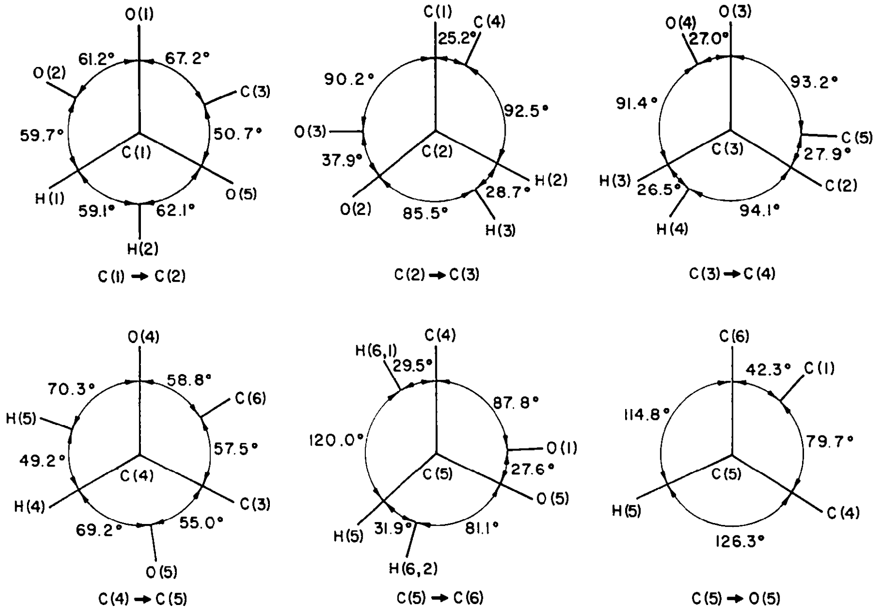

Fig. 3. Torsion angles for the pyranose ring of 1,6-anhydro-3,4-*O*-isopropylidene-*β*-D-talopyranose.

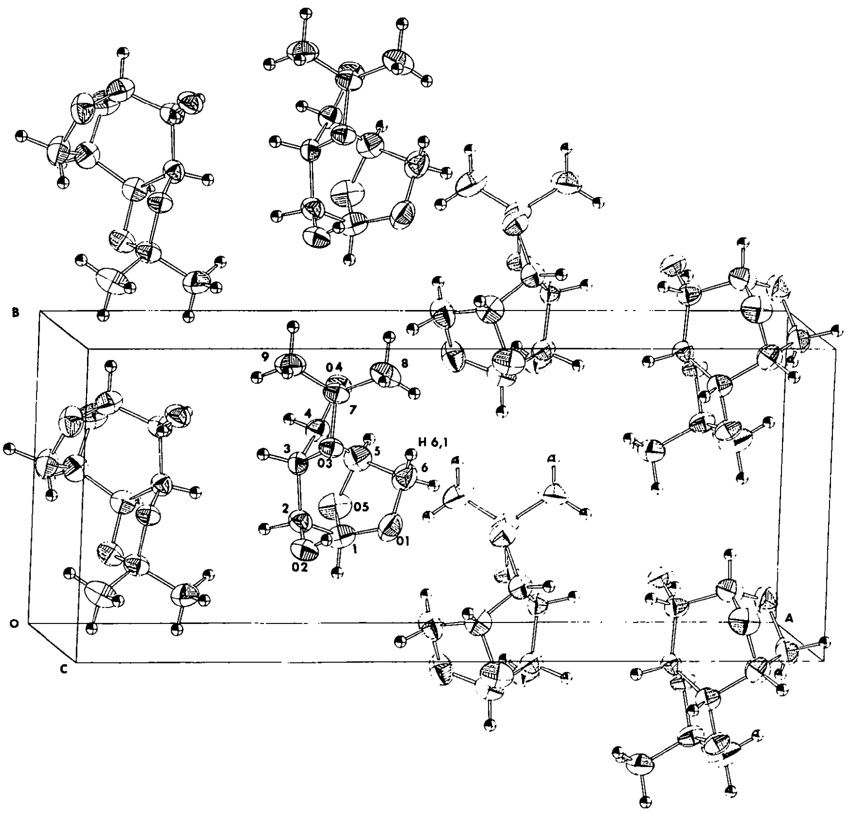

Fig. 4. Molecular packing, [001] projection, in 1,6-anhydro-3,4-*O*-isopropylidene-β-D-talopyranose.

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