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The Crystal and Molecular Structure of β-Lyxose

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The crystals of β-lyxose belong to the space group $P2_12_12_1$, with unit cell dimensions: $a = 9.58$ Å, $b = 10.35$ Å, and $c = 6.515$ Å. There are four formula units per unit cell.

The structure has been solved by direct sign determination, and the atomic parameters refined by least squares methods. The refinement comprises 683 observed $hk0-hk5$ and $0kl$ reflections.

β-Lyxose occurs in the conversion form $1e2a3e4e$ with C–C bond lengths in the range $1.509-1.538 \pm 0.007$ Å, $C_1-O_1 = 1.364 \pm 0.006$ Å, and the other C–O bond lengths varying from 1.399 to 1.435 ± 0.006 Å. Nine of the fourteen bond angles in which only carbon and oxygen are involved, deviate significantly from the tetrahedral value. No carbon atom seems to be "ideally" sp^3 hybridized, and the observed differences in C–C and C–O bond lengths which probably are real, may be explained in relation to hybridization effects.

In the crystal there is a complete set of hydrogen bonds: $O_1\cdots O_3' = 2.808$, $O_2\cdots O_1' = 2.958$, $O_3\cdots O_4' = 2.762$, and $O_4\cdots O_5' = 2.697 \pm 0.005$ Å. There is further an intramolecular contact of 2.35 ± 0.05 Å between H_7, bonded to O_1, and O_2.

Reeves [1] in 1950 proposed a scheme from which the normal conformation of various pyranose sugars may be predicted. The scheme is based on three stability factors, assumed to be of different importance and therefore given different weights. Thus, an axial group (other than hydrogen) is assumed to represent one instability unit, a CH_2OH group in 5 position with an axial hydroxyl group on the same side of the ring (the Hassel and Ottar effect [2]) 0.5 instability unit, and hydroxyl groups in $1e2a$ configuration (the $\Delta 2$ condition [1]) 2.5 instability units. According to Reeves: "Conformational instability is anticipated (but not necessarily realized under all experimental conditions) when the two forms differ by not more than one instability unit; or when the most stable ring form contains as many as 2.5 instability units."

The conformations of α-glucose,[3,4] β-arabinose,[5,6] α-rhamnose,[7] α-xylose,[8] and β-glucose,[9] found through X-ray structure studies, agree with Reeves' predictions. For 2-deoxy-β-ribose [10] the conversion form $1a3e4a$, which according to the above is less stable than the alternative $1e3a4e$, has been found to crystallize. Since the difference in stability between the two forms is small,

only one unit, it is assumed that the conformation may be determined by crystal forces.

The conversion forms for β-lyxose are 1*e*2*a*3*e*4*e* and 1*a*2*e*3*a*4*a*, and the sum of instability units for the two forms are 2.5 and 3, respectively. Although thus Reeves' criterion favours the former to a small degree, stability considerations based on overlap of non-bonded atoms [11] seem to favour the latter, and a structure investigation of β-lyxose was therefore thought of interest.

EXPERIMENTAL

β-Lyxose crystallized from acetonitrile as orthorhombic needles elongated along the *c* axis. The crystals belong to the space group $P2_12_12_1$ with $Z = 4$, and with the unit cell dimensions, $a = 9.58$ Å, $b = 10.35$ Å, and $c = 6.515$ Å. The experimental error is believed to be within 0.2 %.

The intensities of the *hk*0—*hk*5 and 0*kl* reflections were estimated visually from Weissenberg photographs taken with Cu$K\alpha$ radiation, of crystals with cross-section 0.05 × 0.05 mm. By using exposure times of 70—80 h, 683 of the 728 reflections obtainable with Cu$K\alpha$ could be measured, with a range of intensities of 500 000 to 2. The intensities were corrected in the usual way to give sets of relative structure factors. Common reflections in *hk*0—*hk*5 and 0*kl* were used to put all the reflections on the same scale.

The calculated structure factors in Table 7 are based on the scattering curves for oxygen, carbon and hydrogen given in the *International Tables*, [12] the first set of the listed scattering factors for carbon being used.

STRUCTURE DETERMINATION

The structure was solved in the *c* axis projection by means of direct methods, and a brief account of this has been reported earlier.[13] A more detailed description of the procedure, which mainly follows a method by Woolfson,[14] is given here. Observed *hk*0 structure factors were put on an absolute scale by means of Wilson's method,[15] and unitary structure factors down to $U = 0.10$ were calculated.

The 23 reflections with unitary structure factors > 0.22 were arranged in four groups denoted *a*, *b*, *c*, and *d*. Within the respective groups the sign and indices are represented by a_i, b_i, c_i and d_i, and corresponding unitary structure factors are given in brackets.

a *h even k even*

$s(4,0,0) = a_1(0.45)$
$s(8,0,0) = a_2(0.31)$
$s(0,8,0) = a_3(0.32)$
$s(4,2,0) = a_4(0.26)$
$s(6,8,0) = a_5(0.30)$
$s(6,10,0) = a_6(0.30)$
$s(8,4,0) = a_7(0.33)$
$s(8,8,0) = a_8(0.23)$

b *h even k odd*

$s(2,1,0) = b_1(0.41)$
$s(2,3,0) = b_2(0.26)$
$s(2,11,0) = b_3(0.26)$
$s(4,9,0) = b_4(0.25)$
$s(6,1,0) = b_5(0.22)$
$s(6,5,0) = b_6(0.24)$
$s(6,9,0) = b_7(0.25)$
$s(8,9,0) = b_8(0.22)$
$s(10,5,0) = b_9(0.26)$

c *h odd k odd*

$s(1,13,0) = c_1(0.22)$
$s(7,9,0) = c_2(0.26)$
$s(11,5,0) = c_3(0.23)$

d *h odd k even*

$s(1,12,0) = d_1(0.26)$
$s(9,8,0) = d_2(0.26)$

By comparing the Patterson c-projection of β-lyxose with the Patterson a-projection of β-arabinose [5] it became obvious that the "plane" of the β-lyxose molecule had to be nearly parallel to the crystallographic bc plane. From this, and a consideration of the Bragg-Lipson charts for the two strongest reflections, 4,0,0 and 2,1,0, it could be concluded that the structure invariant 4,0,0 most probably had to be negative. The same information is given by the relationships:

$$s(2,1,0)\cdot s(2,\bar{1},0)(U = 0.41) \approx s(4,0,0)(U = 0.45) \approx -$$
$$s(2,3,0)\cdot s(2,\bar{3},0)(U = 0.26) \approx s(4,0,0) \approx -$$
$$s(2,11,0)\cdot s(2,\bar{11},0)(U = 0.26) \approx s(4,0,0) \approx -$$

From the above, a_1 was taken to be negative, and the following products were found to interrelate reflections of group a:

$$a_2a_3a_8 \approx +$$
$$a_4a_4a_2 \approx a_2 \approx +$$
$$a_7a_7a_3 \approx a_3 \approx +$$
$$a_4a_4a_7 \approx a_7 \approx +$$

No relationship include a_5 and a_6, and for the remaining five reflections of group a there are 2^5 possible sign sets of which two satisfy the four relationships. For the subsequent derivation of b signs, it was desirable to include a_6 and a_5, and by this the number of possible sign sets for group a increased to eight. These were arranged in two groups, A and B.

		a_1	a_2	a_3	a_4	a_5	a_6	a_7	a_8
A	Sign sets 1—4	−	+	+	+	±	±	+	+
B	Sign sets 5—8	−	+	+	−	±	±	+	+

The sign relationships which related one member of group a with two members of group b were then sought, and these are given in Table 1.

b_1 was chosen to be positive, and then two sign sets from group A and two from group B gave, by means of Table 1, well developed sign sets for group b. The other four sign sets of group A and B gave poor interrelation of group a and b, and were therefore rejected. b_2 could not be derived on the basis of group B.

Then c_1 was chosen to be negative and it followed from the relationship $c_1c_2 = a_7 \approx +$ that c_2 was probably also negative. The probable sign for d_2 was found from the relationships $c_1b_9 \approx d_2 \approx +$ and $c_2b_1 \approx d_2 \approx +$

Thus finally two sets of signs were derived for 20 reflections on the basis of group A, and two sets of signs for 19 reflections were derived on the basis of group B. The two sign sets based on group A had 15 signs in common, and the two sign sets based on group B had the same. The Fourier maps corresponding to the "common signs" were calculated and examined. The signs based on group A could then be rejected, while the signs based on group B gave a Fourier map which showed a rough picture of the molecule.

It was now desirable to obtain more detailed Fourier maps in order to find out which one of the two group B sign sets was right. Therefore signs for reflections with unitary factors down to 0.10 were derived from the equation

Table 1. Sign relationships between one member of group a and two of group b.

	b_1	b_2	b_3	b_4	b_5	b_6	b_7	b_8	b_9
b_1		$-a_4$		a_6 $-a_5$	$-a_2$	$-a_7$	$-a_8$	a_5 $-a_6$	a_7
b_2	$-a_4$		a_3		a_7 a_4	a_8 a_4			$-a_8$
b_3		a_3					a_4		
b_4	a_6 $-a_5$							—	
b_5	$-a_2$	a_7 a_4					a_3		
b_6	$-a_7$	a_8 a_4							—
b_7	$-a_8$		a_4		a_3				
b_8	a_5 $-a_6$			—					
b_9	a_7	$-a_8$				—			

of Cochran and Woolfson,[16] $s(\mathbf{h}) \approx s(\sum_{\mathbf{h}'} U_{\mathbf{h}'} U_{\mathbf{h}+\mathbf{h}'})$. The corresponding Fourier maps were calculated and are shown in Fig. 1*a* and *b*. They both gave a picture of the molecule and could both be interpreted. The interpretations turned out to be almost the same, but turned 180° relatively to each

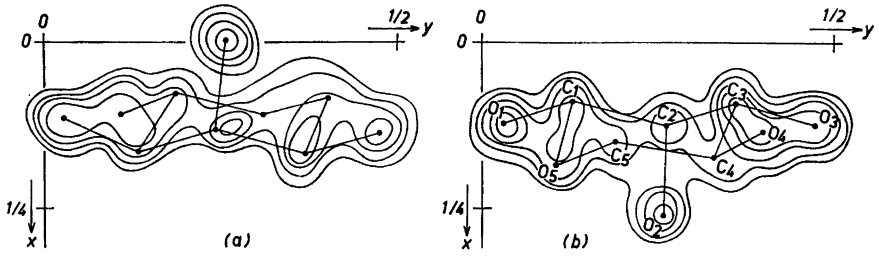

Fig. 1. (a) and (b). Electron density projection on the (001) plane corresponding to the two most probable sign sets. (b) represents the correct c projection of the β-lyxose molecule.

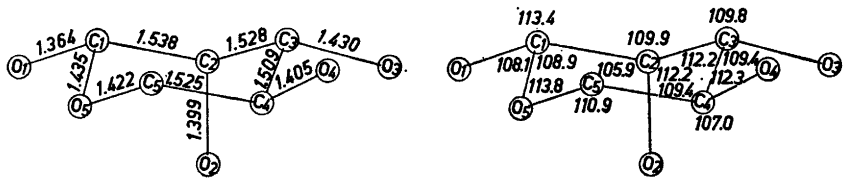

Fig. 2. Bond angles (°, right) and bond lengths (Å, left) in the β-lyxose molecule.

other about $x/8$, $y/4$ in the projection plane. They were both refined by Fourier and difference technique, and while the refinement of the interpretation on Fig. 2*a* stopped at $R = 0.26$, the interpretation on Fig. 2*b* could easily be refined down to $R = 0.15$, a satisfactory value. The contributions from the hydrogen atoms were so far not included in the structure factors.

Approximate z-coordinates for the carbon and oxygen atoms were easily estimated from a consideration of probable hydrogen bonds in the crystal structure, and thereafter the atomic parameters were refined by least squares methods on an IBM 1620[II] computor, using a program designed by Mair.[17] Weighting scheme No. 3, recommended by Mair, was used with $a = 12$ and $b = 7.5$. The refinement comprised the 683 $hk0$—$hk5$ and $0kl$ reflections. Anisotropic temperature factors were applied to carbon and oxygen and isotropic temperature factors to hydrogen.

Final coordinates and temperature parameters are given in Tables 2 and 3, respectively. The observed and calculated structure factors are listed in Table 7. The conventional agreement factor R for all reflections as listed in Table 7, did not improve beyond 0.074.

Table 2. Atomic coordinates in fractions of corresponding cell edges.

Atom	x	y	z
C_1	0.3479	0.1194	−0.0735
C_2	0.3705	0.2569	−0.1565
C_3	0.3419	0.3556	0.0127
C_4	0.4242	0.3277	0.2051
C_5	0.4007	0.1875	0.2689
O_1	0.3812	0.0252	−0.2115
O_2	0.5089	0.2612	−0.2245
O_3	0.3749	0.4825	−0.0593
O_4	0.3823	0.4054	0.3707
O_5	0.4333	0.1017	0.1053
H_1	0.248	0.103	−0.035
H_2	0.311	0.273	−0.262
H_3	0.236	0.357	0.057
H_4	0.532	0.339	0.141
H_5	0.465	0.163	0.371
H_6	0.304	0.169	0.319
H_7	0.471	0.041	−0.269
H_8	0.520	0.314	−0.298
H_9	0.314	0.519	−0.106
H_{10}	0.434	0.456	0.383

Table 3. Temperature parameters, β_{ij} for carbon and oxygen, and B for hydrogen. The expressions used are $\exp-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + kl\beta_{23} + hl\beta_{13})$ for carbon and oxygen, and $\exp-[B(\sin^2\theta/\lambda^2)]$ for hydrogen.

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
C_1	0.0141	0.0103	0.0199	0.0004	−0.0055	0.0014
C_2	0.0135	0.0106	0.0267	0.0002	−0.0046	0.0015
C_3	0.0125	0.0093	0.0214	0.0048	−0.0003	0.0007
C_4	0.0133	0.0100	0.0231	−0.0010	0.0001	0.0003
C_5	0.0181	0.0106	0.0185	0.0009	−0.0018	0.0016
O_1	0.0181	0.0109	0.0308	−0.0084	0.0000	−0.0005
O_2	0.0169	0.0112	0.0281	0.0009	0.0101	0.0010
O_3	0.0143	0.0100	0.0378	0.0094	0.0020	0.0023
O_4	0.0167	0.0108	0.0282	−0.0073	0.0054	−0.0036
O_5	0.0168	0.0098	0.0236	0.0012	−0.0026	0.0041

	H_1	H_2	H_3	H_4	H_5	H_6	H_7	H_8	H_9	H_{10}
B	0.7	7.7	2.1	1.8	2.3	2.6	8.4	4.9	4.2	5.3

Table 4. Bond lengths l and standard deviation in bond length $\sigma(l)$ in the β-lyxose molecule.

Bond	l (Å)	$\sigma(l)$ (Å)
C_1-C_2	1.538	0.007
C_2-C_3	1.528	0.007
C_3-C_4	1.509	0.007
C_4-C_5	1.525	0.006
C_1-O_1	1.364	0.006
C_2-O_2	1.399	0.006
C_3-O_3	1.430	0.005
C_4-O_4	1.405	0.006
C_5-O_5	1.422	0.006
C_1-O_5	1.435	0.006
C_1-H_1	1.00	0.04
C_2-H_2	0.91	0.05
C_3-H_3	1.05	0.04
C_4-H_4	1.12	0.04
C_5-H_5	0.94	0.04
C_5-H_6	1.00	0.04
O_1-H_7	0.96	0.05
O_2-H_8	0.73	0.05
O_3-H_9	0.76	0.05
O_4-H_{10}	0.73	0.05

DISCUSSION

It has been found, *cf.* Fig. 2, that β-lyxose in the crystalline state has the conversion form 1*e*2*a*3*e*4*e*, which from Reeves' stability scheme possesses 2.5 instability units against 3 for the alternative form 1*a*2*e*3*a*4*a*.

Bond lengths and angles for the β-lyxose molecule as derived from the coordinates in Table 2, are listed in Table 4 and 5 and, apart from those involving

Table 5. Bond angles in the β-lyxose molecule. The standard deviations for C—C—C, C—C—O, and C—O—C angles are 0.4°, for angles including one hydrogen 4°, and for the $H_5—C_5—H_6$ angle 6°.

	Angle (°)		Angle (°)
$C_1—C_2—C_3$	109.9	$O_5—C_1—H_1$	109
$C_2—C_3—C_4$	112.2	$O_1—C_1—H_1$	106
$C_3—C_4—C_5$	109.4	$C_2—C_1—H_1$	112
$C_4—C_5—O_5$	110.9	$C_1—C_2—H_2$	110
$C_5—O_5—C_1$	113.8	$C_3—C_2—H_2$	108
$O_5—C_1—C_2$	108.9	$O_2—C_2—H_2$	111
$O_5—C_1—O_1$	108.1	$C_2—C_3—H_3$	112
$C_2—C_1—O_1$	113.4	$C_4—C_3—H_3$	106
$C_1—C_2—O_2$	105.9	$O_3—C_3—H_3$	107
$C_3—C_2—O_2$	112.2	$C_3—C_4—H_4$	99
$C_2—C_3—O_3$	109.8	$C_5—C_4—H_4$	110
$C_4—C_3—O_3$	109.4	$O_4—C_4—H_4$	120
$C_3—C_4—O_4$	112.3	$C_4—C_5—H_5$	111
$C_5—C_4—O_4$	107.0	$O_5—C_5—H_5$	102
		$C_4—C_5—H_6$	114
$C_1—O_1—H_7$	110	$O_5—C_5—H_6$	109
$C_2—O_2—H_8$	111	$H_5—C_5—H_6$	109
$C_3—O_3—H_9$	114		
$C_4—O_4—H_{10}$	107		

Table 6. Atomic distances and angles of interest for the Δ2 condition and for hydrogen bonding. The standard deviations are 0.005 Å in the distances and 0.4° in the angles.

		Distance (Å)		Angle (°)
Δ2 condition	$O_1\cdots O_5$	2.267	$O_1—C_1—C_2$	113.4
	$O_1\cdots O_2$	2.733	$C_1—C_2—O_2$	105.9
	$O_2\cdots O_5$	2.805	$O_5—C_1—C_2$	108.9
			$O_1—C_1—O_5$	108.1
Hydrogen bonds	$O_1\cdots O_3'$	2.808	$C_1—O_2\cdots O_3'$	131.1
	$O_2\cdots O_1'$	2.958	$C_2—O_2\cdots O_1'$	114.3
	$O_3\cdots O_4'$	2.762	$C_3—O_3\cdots O_4'$	104.0
	$O_4\cdots O_5'$	2.697	$C_4—O_4\cdots O_5'$	106.8

hydrogen, shown in Fig. 2. The carbon-carbon bond lengths are in the range 1.509—1.538 ± 0.007 Å, the $C_1—O_1$ bond is 1.364 ± 0.006 Å, and the lengths of the other carbon-oxygen bonds vary from 1.399—1.435 ± 0.006 Å.

The author believes that the observed differences in carbon-carbon and carbon-oxygen bond lengths are real and should be seen in relation to hybridization effects. At least this seems to be indicated from the first fourteen entries of bond angles in Table 5. If three times the standard deviation is taken as a significant figure, nine of these bond angles deviate significantly from the tetrahedral value, 109.5°, and none of the five carbon atoms seems to be "ideally" sp^3 hybridized. It is therefore likely that the various carbon bonding-orbitals possess different degrees of *s* and *p* character, diverging slightly from ideal sp^3 hybridization, and reflected in different bond lengths.

The β-lyxose molecule as it occurs in the crystal, represents a compromise between energy factors including interaction with environment as well as atomic hybridization. One can therefore hardly *a priori* deduce which of the carbon bonding orbitals has the greatest *s*-character. However, from the

Table 7. Observed and calculated structure factors for β-lyxose.

H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c
2			59.24	51.53	6	7		0.62	0.73	1	8	1	18.86	17.59	8	6	1	2.40	2.53
4			70.99	66.88	6	8		4.96	6.65	1	9	1	11.48	11.20	8	7	1	2.38	2.52
6			1.89	1.00	6	9		4.28	5.02	1	10	1	7.47	7.17	8	8	1	2.46	2.52
8			15.00	12.85	6	10		4.65	4.98	1	11	1	2.99	2.80	8	9	1	2.60	3.17
10			0.75	0.90	6	11		1.31	1.32	1	12	1	2.49	2.53					
12			0.26	0.00						1	13	1	1.48	1.94	9	1	1	4.40	4.16
					7	1		3.45	4.33						9	2	1	< 0.66	0.60
	2		4.90	4.74	7	2		6.19	5.99	2	1	1	24.18	24.54	9	3	1	2.35	2.35
	4		3.17	3.25	7	3		0.68	0.61	2	2	1	16.38	16.45	9	4	1	4.64	4.29
	6		1.74	2.03	7	4		3.38	3.14	2	3	1	10.58	10.40	9	5	1	0.86	0.76
	8		16.38	17.08	7	5		4.27	4.24	2	4	1	6.39	5.60	9	6	1	0.80	0.75
	10		2.78	2.98	7	6	<	0.50	0.41	2	5	1	7.89	7.65	9	7	1	1.63	1.41
	12		3.97	3.76	7	7		0.93	0.98	2	6	1	10.75	10.79	9	8	1	1.60	1.99
					7	8		2.85	3.19	2	7	1	4.62	4.12					
1	1		7.48	7.76	7	9		3.16	3.58	2	8	1	13.65	13.53	10	1	1	3.98	3.63
1	2		1.44	1.20	7	10		1.11	1.44	2	9	1	2.76	2.30	10	2	1	1.36	1.41
1	3		1.86	1.40						2	10	1	3.26	3.17	10	3	1	3.43	3.00
1	4		15.07	14.87	8	1		2.00	1.56	2	11	1	< 0.58	0.33	10	4	1	4.44	4.68
1	5		4.32	3.70	8	2		2.58	2.38	2	12	1	0.67	0.45	10	5	1	2.30	2.63
1	6		5.99	6.17	8	3		3.01	3.12						10	6	1	1.19	1.16
1	7		3.86	3.60	8	4		10.18	10.61	3	1	1	29.66	28.76	10	7	1	1.39	1.61
1	8		0.90	1.00	8	5		2.32	2.84	3	2	1	30.22	29.83					
1	9		1.19	1.65	8	6		1.91	2.28	3	3	1	9.63	9.54	11	1	1	< 0.50	0.44
1	10	<	0.50	0.32	8	7		2.43	2.41	3	4	1	10.55	11.59	11	2	1	< 0.49	0.39
1	11	<	0.46	0.41	8	8		3.72	3.97	3	5	1	17.28	17.93	11	3	1	1.34	1.11
1	12		3.73	4.00	8	9		3.11	3.07	3	6	1	13.62	13.79	11	4	1	0.99	1.08
1	13		2.67	3.28						3	7	1	7.08	7.02	11	5	1	0.65	0.77
					9	1		1.70	1.66	3	8	1	9.09	8.46					
2	1		112.95	105.07	9	2		1.95	1.80	3	9	1	11.16	10.53	12	1	1	0.97	0.85
2	2		27.41	23.11	9	3		2.13	2.13	3	10	1	0.90	0.49					
2	3		56.79	47.90	9	4		3.73	3.82	3	11	1	1.49	1.37	1		2	37.77	42.81
2	4		11.99	11.37	9	5		0.88	0.54	3	12	1	1.67	1.77	2		2	37.36	39.35
2	5		5.47	5.42	9	6		1.67	1.58						3		2	16.17	16.92
2	6		8.47	8.91	9	7		0.62	0.73	4	1	1	8.87	8.58	4		2	7.27	7.26
2	7		1.36	0.85	9	8		4.28	4.11	4	2	1	13.28	13.93	5		2	34.91	31.77
2	8		0.77	1.11						4	3	1	2.21	2.28	6		2	6.25	6.47
2	9		5.45	5.66	10	1		1.78	1.40	4	4	1	8.37	8.98	7		2	10.48	10.75
2	10		1.06	1.26	10	2		0.93	0.85	4	5	1	8.44	8.69	8		2	3.70	3.06
2	11		4.53	4.84	10	3	<	0.65	0.17	4	6	1	3.93	3.54	9		2	7.14	6.83
2	12		2.05	2.27	10	4		3.58	3.92	4	7	1	4.54	4.71	10		2	5.21	5.29
2	13		3.51	3.15	10	5		3.49	4.06	4	8	1	2.92	2.64	11		2	3.05	3.03
					10	6	<	0.35	0.10	4	9	1	7.74	8.21					
3	1		3.14	3.45						4	10	1	4.10	3.82		1	2	22.55	24.94
3	2		7.70	7.63	11	1		0.85	1.00	4	11	1	1.46	1.27		2	2	34.95	38.12
3	3		3.46	3.19	11	2	<	0.38	0.32	4	12	1	1.14	1.05		3	2	15.74	15.97
3	4		10.49	10.02	11	3		1.52	1.80							4	2	31.37	33.23
3	5		1.55	1.74	11	4		0.52	0.65	5	1	1	17.19	17.96		5	2	17.23	17.64
3	6		7.06	6.92	11	5		2.67	2.65	5	2	1	22.16	20.17		6	2	4.21	3.89
3	7		1.95	2.30						5	3	1	5.97	6.26		7	2	10.01	10.45
3	8	<	0.50	0.54	12	1		1.59	1.77	5	4	1	7.54	7.96		8	2	8.00	8.12
3	9		1.61	1.77	12	2		1.94	1.74	5	5	1	10.17	10.87		9	2	7.78	7.97
3	10		1.16	1.23						5	6	1	5.59	6.28		10	2	2.65	2.12
3	11		0.62	0.62	1		1	6.53	6.73	5	7	1	2.72	2.71		11	2	2.46	2.34
3	12		0.42	0.32	2		1	63.09	64.48	5	8	1	3.65	3.71		12	2	2.23	2.25
					3		1	5.70	4.64	5	9	1	3.70	3.79					
4	1		8.47	8.66	4		1	5.63	4.95	5	10	1	2.32	2.54	1	1	2	23.95	28.19
4	2		31.92	33.41	5		1	19.02	18.61	5	11	1	0.99	1.04	1	2	2	11.05	12.52
4	3		11.93	11.94	6		1	10.70	9.65						1	3	2	10.31	11.29
4	4		0.61	1.09	7		1	20.93	20.43	6	1	1	11.66	11.41	1	4	2	11.95	12.33
4	5	<	0.42	0.62	8		1	6.23	5.65	6	2	1	2.37	2.48	1	5	2	21.91	22.56
4	6		8.29	8.91	9		1	4.68	4.84	6	3	1	3.39	3.35	1	6	2	7.19	6.49
4	7		2.53	2.51	10		1	1.64	1.08	6	4	1	10.75	11.37	1	7	2	8.01	7.51
4	8		3.45	3.84	11		1	2.28	1.78	6	5	1	8.18	8.19	1	8	2	10.63	10.67
4	9		5.90	6.51	12		1	2.33	2.34	6	6	1	5.85	6.97	1	9	2	5.72	5.24
4	10		3.16	3.62						6	7	1	1.58	1.58	1	10	2	2.45	1.98
4	11		1.41	1.63		1	1	50.25	54.01	6	8	1	3.92	4.39	1	11	2	1.42	1.11
4	12		0.57	0.66		2	1	12.92	12.98	6	9	1	3.62	3.66	1	12	2	2.10	2.03
						3	1	17.39	16.85	6	10	1	1.78	2.00					
5	1		15.04	14.86		4	1	23.17	24.17	6	11	1	0.53	0.57	2	1	2	13.76	14.84
5	2		1.14	0.94		5	1	4.03	3.36						2	2	2	23.36	25.34
5	3		1.60	1.56		6	1	5.84	5.42	7	1	1	5.01	4.92	2	3	2	13.59	13.43
5	4		4.61	4.54		7	1	3.28	2.91	7	2	1	6.97	6.75	2	4	2	20.78	20.99
5	5		5.51	5.17		8	1	10.05	9.86	7	3	1	3.22	3.24	2	5	2	14.58	14.26
5	6		3.63	4.67		9	1	11.18	11.45	7	4	1	4.83	4.79	2	6	2	1.61	1.38
5	7		1.51	1.30		10	1	1.24	1.39	7	5	1	5.25	5.56	2	7	2	10.01	10.53
5	8	<	0.50	0.33		11	1	2.98	2.52	7	6	1	2.36	2.19	2	8	2	9.33	9.07
5	9		3.17	3.58		12	1	1.81	1.50	7	7	1	1.22	1.50	2	9	2	6.36	6.55
5	10		1.04	1.22		13	1	1.20	1.35	7	8	1	2.31	2.45	2	10	2	3.13	2.66
5	11	<	0.37	0.14						7	9	1	3.71	4.39	2	11	2	0.97	0.73
					1	1	1	15.04	16.01	7	10	1	1.31	1.40	2	12	2	1.68	1.69
6	1		16.69	16.45	1	2	1	11.92	11.13										
6	2		8.89	8.41	1	3	1	11.16	10.97	8	1	1	< 0.66	0.53	3	1	2	24.37	25.08
6	3		2.89	3.37	1	4	1	59.82	57.28	8	2	1	2.98	2.53	3	2	2	15.25	15.92
6	4		4.53	5.41	1	5	1	34.50	34.21	8	3	1	4.63	4.34	3	3	2	19.68	21.95
6	5		9.57	10.46	1	6	1	10.93	11.19	8	4	1	8.39	7.93	3	4	2	13.40	13.19
6	6		3.37	3.50	1	7	1	7.38	7.72	8	5	1	3.46	3.14	3	5	2	12.68	12.78

H	K	L	F_o	F_c
3	6	2	7.30	7.00
3	7	2	4.74	4.42
3	8	2	6.71	6.34
3	9	2	2.52	2.52
3	10	2	2.73	2.63
3	11	2	2.65	2.46
3	12	2	< 0.55	0.40
4	1	2	15.58	15.26
4	2	2	14.18	15.31
4	3	2	5.14	4.42
4	4	2	10.30	9.53
4	5	2	9.60	9.91
4	6	2	9.09	8.66
4	7	2	7.05	6.77
4	8	2	9.28	9.19
4	9	2	2.03	1.46
4	10	2	2.64	2.38
4	11	2	1.92	1.76
4	12	2	0.94	1.36
5	1	2	14.22	12.93
5	2	2	4.48	4.67
5	3	2	4.82	4.46
5	4	2	2.02	1.89
5	5	2	6.90	6.83
5	6	2	9.78	9.30
5	7	2	5.19	5.35
5	8	2	5.00	4.98
5	9	2	2.37	2.45
5	10	2	3.50	3.56
5	11	2	1.40	1.40
6	1	2	7.29	7.13
6	2	2	1.84	1.68
6	3	2	3.74	3.59
6	4	2	9.41	8.73
6	5	2	9.96	10.06
6	6	2	9.10	9.57
6	7	2	< 0.95	0.37
6	8	2	2.61	2.49
6	9	2	3.10	3.23
6	10	2	< 0.67	0.47
6	11	2	< 0.44	0.40
7	1	2	13.53	12.18
7	2	2	4.70	4.52
7	3	2	7.80	6.81
7	4	2	2.70	2.58
7	5	2	5.19	4.52
7	6	2	2.51	2.23
7	7	2	3.78	3.57
7	8	2	4.11	4.80
7	9	2	1.22	1.25
7	10	2	0.97	1.07
8	1	2	8.61	8.00
8	2	2	4.01	4.17
8	3	2	< 0.96	0.68
8	4	2	3.57	3.27
8	5	2	4.08	3.84
8	6	2	4.28	4.69
8	7	2	1.51	1.43
8	8	2	2.31	1.95
8	9	2	1.04	1.20
9	1	2	4.81	4.68
9	2	2	1.61	1.41
9	3	2	2.25	1.91
9	4	2	1.08	1.27
9	5	2	2.84	2.23
9	6	2	1.83	1.51
9	7	2	1.23	1.49
9	8	2	0.95	0.96
10	1	2	< 0.84	0.77
10	2	2	2.03	2.07
10	3	2	1.54	1.42
10	4	2	0.99	1.00
10	5	2	1.17	1.07
10	6	2	0.70	0.68
11	1	2	2.31	2.19
11	2	2	1.02	0.77
11	3	2	< 0.59	0.55
11	4	2	< 0.52	0.32
1		3	18.93	17.93
2		3	5.06	4.66
3		3	24.08	22.42
4		3	10.21	9.51
5		3	7.51	6.30
6		3	1.16	0.98
7		3	< 0.87	0.46
8		3	5.30	4.62
9		3	1.77	1.31
10		3	1.12	0.84
11		3	0.73	0.85
	1	3	5.54	5.57
	2	3	6.62	6.48
	3	3	19.95	20.64
	4	3	15.40	15.60
	5	3	13.86	13.46
	6	3	8.43	7.79
	7	3	1.20	1.19
	8	3	7.92	8.25
	9	3	3.70	3.45
	10	3	1.27	0.65
	11	3	1.45	0.89
	12	3	2.44	2.29
1	1	3	17.74	20.21
1	2	3	9.98	11.24
1	3	3	16.58	16.18
1	4	3	10.24	9.71
1	5	3	9.35	9.31
1	6	3	17.64	17.83
1	7	3	6.00	5.59
1	8	3	6.84	5.84
1	9	3	3.59	3.51
1	10	3	2.21	1.71
1	11	3	1.31	1.27
1	12	3	0.79	0.53
2	1	3	20.74	20.29
2	2	3	12.84	12.50
2	3	3	22.94	22.64
2	4	3	15.19	14.45
2	5	3	11.49	11.33
2	6	3	7.20	7.31
2	7	3	4.02	3.50
2	8	3	< 0.89	0.75
2	9	3	1.61	1.92
2	10	3	2.27	1.58
2	11	3	0.79	0.71
2	12	3	1.58	1.49
3	1	3	15.95	15.46
3	2	3	11.58	11.47
3	3	3	19.53	17.62
3	4	3	15.20	14.70
3	5	3	4.70	4.91
3	6	3	4.01	3.88
3	7	3	10.14	9.93
3	8	3	6.83	5.87
3	9	3	1.03	0.76
3	10	3	2.78	2.34
3	11	3	1.28	1.21
4	1	3	11.31	11.33
4	2	3	6.15	5.86
4	3	3	9.63	9.10
4	4	3	9.26	9.12
4	5	3	8.11	7.72
4	6	3	10.70	11.03
4	7	3	2.75	2.45
4	8	3	4.95	4.68
4	9	3	3.08	2.81
4	10	3	3.21	2.72
4	11	3	0.75	0.39
5	1	3	10.25	9.75
5	2	3	15.06	14.57
5	3	3	10.45	10.11
5	4	3	10.11	8.71
5	5	3	2.62	2.63
5	6	3	4.20	4.40
5	7	3	4.32	4.11
5	8	3	4.03	3.90
5	9	3	3.54	3.34
5	10	3	1.88	1.62
5	11	3	1.73	2.23
6	1	3	3.61	3.26
6	2	3	9.13	8.51
6	3	3	12.10	13.21
6	4	3	8.82	8.07
6	5	3	9.82	9.34
6	6	3	3.16	3.07
6	7	3	3.39	2.67
6	8	3	2.25	1.86
6	9	3	1.16	1.43
6	10	3	2.54	2.60
7	1	3	5.24	4.52
7	2	3	7.24	6.31
7	3	3	3.05	2.67
7	4	3	6.53	5.95
7	5	3	3.20	2.66
7	6	3	1.78	1.27
7	7	3	3.65	3.53
7	8	3	2.29	2.17
7	9	3	1.63	1.70
8	1	3	1.87	1.90
8	2	3	5.42	4.72
8	3	3	5.45	4.70
8	4	3	2.84	2.57
8	5	3	3.57	3.51
8	6	3	3.29	3.40
8	7	3	1.70	1.69
8	8	3	3.15	3.19
9	1	3	1.94	1.61
9	2	3	2.57	2.44
9	3	3	3.55	2.86
9	4	3	1.40	1.25
9	5	3	1.47	1.44
9	6	3	1.66	1.68
9	7	3	2.08	2.00
10	1	3	0.86	0.80
10	2	3	0.84	0.71
10	3	3	2.76	2.30
10	4	3	1.26	1.17
10	5	3	1.90	2.00
10	6	3	0.77	1.06
11	1	3	0.88	0.88
11	2	3	< 0.47	0.00
11	3	3	0.82	0.62
1		4	0.97	0.58
2		4	1.16	1.39
3		4	1.46	1.39
4		4	5.75	5.80
5		4	< 1.04	0.45
6		4	4.40	4.76
7		4	1.94	2.65
8		4	< 1.07	0.84
9		4	< 0.95	0.44
10		4	1.19	1.27
	1	4	16.92	16.44
	2	4	11.49	11.59
	3	4	10.32	9.70
	4	4	14.71	13.46
	5	4	14.97	13.34
	6	4	15.93	14.42
	7	4	4.76	3.89
	8	4	2.29	2.48
	9	4	2.21	2.28
	10	4	4.69	4.27
	11	4	5.09	4.23
1	1	4	8.52	7.83
1	2	4	4.61	4.31
1	3	4	8.10	7.39
1	4	4	5.12	4.60
1	5	4	10.16	8.97
1	6	4	6.83	6.57
1	7	4	7.88	7.25
1	8	4	1.92	1.55
1	9	4	1.27	0.48
1	10	4	1.91	1.49
1	11	4	1.46	1.12
2	1	4	8.16	7.73
2	2	4	10.75	9.67
2	3	4	10.04	9.93
2	4	4	12.07	11.51
2	5	4	7.41	7.30
2	6	4	9.50	9.06
2	7	4	13.11	13.37
2	8	4	5.29	5.05
2	9	4	2.86	2.31
2	10	4	1.51	1.38
2	11	4	1.28	1.34
3	1	4	3.33	3.02
3	2	4	4.26	3.42
3	3	4	2.53	2.84
3	4	4	3.46	3.72
3	5	4	4.38	4.15
3	6	4	7.19	6.89
3	7	4	4.57	4.70
3	8	4	4.16	3.98
3	9	4	< 0.98	1.26
3	10	4	< 0.82	0.89
3	11	4	1.82	1.72
4	1	4	7.50	8.79
4	2	4	11.30	11.65
4	3	4	10.75	10.81
4	4	4	2.27	2.23
4	5	4	8.43	9.85
4	6	4	12.18	12.59
4	7	4	5.23	5.06
4	8	4	3.52	3.41
4	9	4	0.91	0.95
4	10	4	< 0.73	0.65
5	1	4	3.77	4.08
5	2	4	7.12	7.85
5	3	4	2.03	2.21
5	4	4	4.55	4.46
5	5	4	2.63	2.63
5	6	4	2.35	3.16
5	7	4	3.29	3.36
5	8	4	1.80	1.57
5	9	4	1.16	1.16
5	10	4	2.09	2.20
6	1	4	4.89	6.04
6	2	4	2.08	2.54
6	3	4	4.62	5.14
6	4	4	2.96	2.98
6	5	4	1.35	1.71
6	6	4	4.61	5.03
6	7	4	3.06	2.97
6	8	4	3.70	4.33
6	9	4	0.96	0.95
7	1	4	4.83	5.55
7	2	4	2.49	2.72
7	3	4	4.95	5.50
7	4	4	3.67	3.66
7	5	4	1.46	1.23
7	6	4	1.36	1.77
7	7	4	0.85	0.87
7	8	4	1.10	1.24
8	1	4	1.51	1.26
8	2	4	3.46	4.06
8	3	4	4.41	5.24
8	4	4	1.56	1.57
8	5	4	2.04	2.53
8	6	4	0.99	1.43
8	7	4	1.33	1.76
9	1	4	1.83	2.05
9	2	4	3.69	4.45
9	3	4	1.07	1.37
9	4	4	< 0.82	0.20
9	5	4	0.72	0.77
9	6	4	0.80	1.12
10	1	4	1.73	2.26
10	2	4	0.99	1.22
10	3	4	1.01	1.12
10	4	4	1.11	1.82
1		5	7.62	9.04
2		5	3.67	3.83
3		5	0.77	0.30
4		5	1.70	2.45
5		5	1.26	1.79
6		5	3.81	4.28
7		5	2.66	3.36
8		5	1.24	1.26
9		5	1.00	1.09
	1	5	6.36	5.90
	2	5	< 0.67	0.44
	3	5	5.05	5.65
	4	5	< 0.83	0.62
	5	5	1.39	1.27
	6	5	5.36	5.31
	7	5	3.14	2.89

H	K	L	F_o	F_c
	8	5	1.33	1.41
	9	5	4.37	3.91
	10	5	2.10	2.16
1	1	5	8.40	8.14
1	2	5	13.24	13.39
1	3	5	19.77	17.64
1	4	5	4.02	4.03
1	5	5	5.77	5.52
1	6	5	7.14	6.57
1	7	5	2.28	1.28
1	8	5	3.98	3.26
1	9	5	1.72	1.69
1	10	5	1.60	1.28
2	1	5	4.21	4.04
2	2	5	3.48	3.72
2	3	5	7.27	6.93
2	4	5	2.79	2.45
2	5	5	4.63	4.23
2	6	5	4.98	5.07
2	7	5	1.40	1.57
2	8	5	1.00	0.90
2	9	5	1.11	1.05
2	10	5	1.75	2.12
3	1	5	2.65	2.76
3	2	5	13.94	15.85
3	3	5	9.59	10.70
3	4	5	5.21	5.16
3	5	5	4.31	4.07
3	6	5	4.68	4.77
3	7	5	2.20	2.48
3	8	5	3.54	3.21
3	9	5	0.80	0.91
3	10	5	2.74	3.17
4	1	5	1.60	2.01
4	2	5	1.23	1.12
4	3	5	4.41	4.28
4	4	5	2.12	2.19
4	5	5	1.28	1.81
4	6	5	3.23	3.00
4	7	5	1.30	1.51
4	8	5	1.26	1.35
4	9	5	0.82	1.04
5	1	5	< 0.90	1.51
5	2	5	7.54	8.96
5	3	5	5.66	6.88
5	4	5	3.07	2.95
5	5	5	2.34	2.35
5	6	5	2.94	2.75
5	7	5	2.01	1.72
5	8	5	< 0.64	0.24
6	1	5	2.84	3.42
6	2	5	2.39	2.80
6	3	5	< 0.89	0.82
6	4	5	1.23	1.15
6	5	5	1.17	0.95
6	6	5	2.22	2.36
6	7	5	1.40	1.47
6	8	5	1.51	1.92
7	1	5	1.94	2.06
7	2	5	1.21	1.22
7	3	5	2.28	2.82
7	4	5	< 0.79	1.26
7	5	5	1.16	1.33
7	6	5	< 0.64	0.64
8	1	5	1.23	1.83
8	2	5	1.31	1.47
8	3	5	1.69	2.35
8	4	5	< 0.66	0.94
8	5	5	1.53	1.93
9	1	5	1.31	1.92
9	2	5	1.03	1.18
9	3	5	1.14	1.52
	4		2.75	3.23
	6		6.88	6.57
	8		2.10	1.46
1	6		9.96	11.20
2	6		2.55	1.96
3	6		6.83	6.94
4	6		1.03	1.49
5	6		3.54	3.35
6	6		1.02	1.18
7	6		< 0.49	0.26
1	7		2.38	2.01
2	7		4.41	4.17
3	7		5.13	4.26
4	7		2.37	1.81
5	7		2.58	2.22
6	7		0.86	0.55
1	8		1.77	1.13
2	8		2.15	1.37
3	8		< 0.30	0.35

experimental results it seems relevant to think that when the angle between two bonding carbon orbitals is significantly greater than the tetrahedral angle, one or both of these orbitals exceed sp^3 in s character, and may therefore contribute to "short" bonds. The other bonding orbitals of the carbon in question then probably exceed sp^3 in p-character and contribute to "long" bonds. The molecular dimensions of β-lyxose show no inconsistency with this view.

The shortest carbon-oxygen bond in β-lyxose, $C_1-O_1 = 1.364 \pm 0.006$ Å, probably has somewhat different bonding conditions than the other carbon-oxygen bonds in the molecule, because C_1 also is engaged in the cyclic C_1-O_5 bond. The length of the C_1-O_1 bond in pyranose structures is generally found to be smaller than the accepted value, 1.43 Å, for a carbon-oxygen single bond. In β-arabinose [6] for instance, it was found to be 1.382 ± 0.012 Å, and in β-glucose,[9] 1.404 ± 0.010 Å. The difference between the latter value and that found in β-lyxose may be related to hybridization effects. Thus in β-lyxose the $O_5-C_1-O_1$ and $C_2-C_1-O_1$ angles are 108.1 and $113.4 \pm 0.4°$, respectively, and in β-glucose they are both only $107.3 \pm 0.6°$. According to the above, this may explain why C_1-O_1 is found to be somewhat longer in β-glucose than in β-lyxose. In β-arabinose [6] the mentioned bond angles are 113.0 and $108.9 \pm 0.6°$, respectively, and the C_1-O_1 bond length there agree with that in β-lyxose.

A value for the pyranose angle $C_1-O_5-C_5$ seems now well established from structure determinations of reasonable accuracy. Thus in β-arabinose [6] the angle was found to be $112.7 \pm 0.6°$, in β-glucose [9] $113.1 \pm 0.5°$, in α-methyl D-galactoside 6-bromhydrine [18] $113.7 \pm 0.9°$ and in the present investigation $113.8 \pm 0.4°$. The $C_1-O_5-C_5$ angle of 120° reported for α-rhamnose monohydrate [7] is almost certainly related to the poor accuracy of the y-coordinates in that structure.

The average values of C—H and O—H bond lengths in β-lyxose are 1.00 and 0.80 Å, respectively, and thus smaller than would be expected from currently accepted values, which are 1.09 Å for C—H and 0.96 Å for O—H.[19] There are numerous examples in the literature where such bonds by X-ray

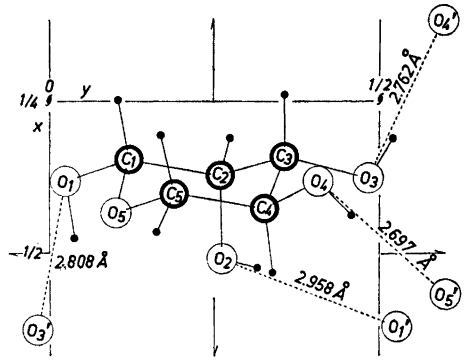

Fig. 3. The β-lyxose molecule as seen along the *c* axis with hydrogen bonds indicated.

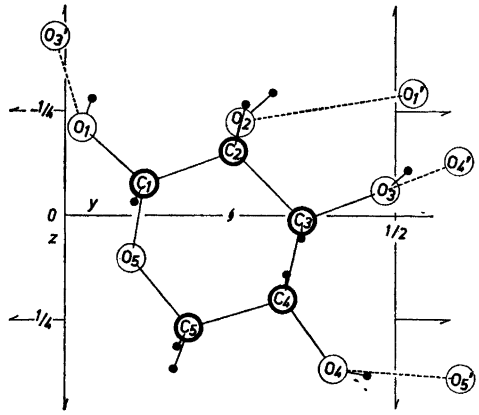

Fig. 4. The β-lyxose molecule as seen along the *a* axis with hydrogen bonds indicated.

methods have been found "too short", and the generally accepted explanation is that the X-ray method localizes the centroid of the electron cloud on hydrogen rather than the position of the nucleus.

It is tempting to point to a coincidence between the $C_3-C_4-H_4$ bond angle and the C_4-H_4 bond length as found in β-lyxose and β-arabinose.[6] The mentioned angle is $99 \pm 4°$ in the former and $98 \pm 6°$ in the latter, and the C—H bond lengths are 1.12 ± 0.04 Å and 1.15 ± 0.05 Å, respectively. The bond angles are the smallest found at carbon atoms in the two molecules, and the C—H bonds the longest. This indicates that the bonding orbital on C_4 which participates in the C_4-H_4 bond has considerable *p*-character. It may be a real effect since the C_4-O_4 and C_4-C_3 bond lengths, in β-lyxose found to be 1.405 and 1.509 Å, respectively, indicate a corresponding excess of *s*-character in the orbitals on C_4 participating in those bonds.

The Δ2 condition[1] 1*e*2*a*, sometimes referred to as the "*Δ2* effect" when considering the stability of conversion forms of pyranoses, occurs in β-lyxose. Atomic distances and angles of interest for this structural detail are listed in the first part of Table 6, and from the values one may conclude that the 1*e*2*a* configuration hardly leads to serious structural irregularities.

Hydrogen bonds. The intermolecular O···O distances which correspond to hydrogen bonds are listed in the last part of Table 6 and shown in Figs. 3 and 4. Furthermore, the positions of the hydrogen atoms in the β-lyxose molecule are shown in these figures.

The $O_2\cdots O_1'$ distance is 2.958 ± 0.005 Å and the hydrogen atom H_8, bonded to O_2, lies about 0.35 Å off the straight line between O_2 and O_1'. Although the distance is rather long it may correspond to a weak hydrogen bond; the distance between H_8 and O_1' is 2.38 ± 0.05 Å and thus shorter than the sum, 2.60 Å, of the van der Waals radii[19] for hydrogen and oxygen. $O_1\cdots O_3' = 2.808 \pm 0.005$ Å represents a relatively weak hydrogen bond. H_7, bonded to O_1, lies about 0.30 Å off the straight line between O_1 and O_3',

1.95 ± 0.05 Å from O_3' and 2.35 ± 0.05 Å from O_2. The latter value shows that H_7 apart from being engaged in the $O_1 \cdots O_3'$ hydrogen bond, is also in contact with O_2. The $C_1-O_1 \cdots O_3'$ angle is 131.1 ± 0.4° and the $C_1-O_1-H_7$ angle is 110 ± 4°; the latter angle relative to the former prevents H_7 from being close to the straight line between O_1 and O_3'. H_7 then compromises by locating itself as close as possible to both O_3' and O_2, *cf.* Figs. 3 and 4. The hydrogen bonds, $O_3 \cdots O_4' = 2.762 \pm 0.005$ Å and $O_4 \cdots O_5' = 2.697 \pm 0.005$ Å, are of normal lengths for sugar structures, with the hydrogen atoms located close to the straight lines connecting the oxygen atoms.

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