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The Crystal Structures of the Dihydrated Potassium and Rubidium Salts of β-D-Glucuronic Acid

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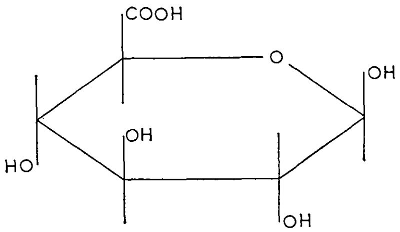
The crystal structures of the isomorphous dihydrated potassium and rubidium salts of β-D-glucuronic acid have been determined, and a two-dimensional refinement of the potassium isomorph ($C_6H_9O_7K.2H_2O$) completed. The structure has an extensive hydrogen bonding network, and there are binding forces between the positive ion and electronegative oxygen atoms.

Introduction

β-D-Glucuronic acid is a widely distributed constituent radical of the polysaccharides. It has the structural formula



The molecular dimensions of the glucuronate ion were required as part of a proposed structural study of some fibrous muco-polysaccharides, and hence this investigation of the isomorphous dihydrated potassium and rubidium salts of β-D-glucuronic acid was undertaken.

Experimental

Keihn & King (1955) have prepared a number of salts of β-D-glucuronic acid, and shown by X-ray examination that potassium and rubidium glucuronate are isomorphous and dihydrated. Crystals of these salts were prepared for this study by addition of the equivalent amount of the respective carbonate to an aqueous solution of β-D-glucuronolactone, in a similar manner to that described by Keihn & King. The solutions were decolorized with activated carbon, concentrated at room temperature under reduced pressure, and allowed to crystallize in an atmosphere of ethyl alcohol in a desiccator. Both salts crystallized as beautifully formed, colourless, orthorhombic prisms, elongated along *a*. Because the rubidium salt loses water of crystallization, these crystals were coated with collodion for the X-ray measurements.

Crystal data

The unit cell dimensions of potassium glucuronate were measured from Weissenberg films on which had also been recorded reflexions from a rock-salt crystal. In Table 1 the results are compared with those obtained by Keihn & King.

Table 1. *Density and unit cell dimensions of potassium glucuronate*

	a (Å)	*b* (Å)	*c* (Å)	g.cm^{-3}
Keihn & King	5·55 ± 0·01	16·52 ± 0·05	11·28 ± 0·05	1·718
This author	5·52 ± 0·01	16·52 ± 0·02	11·27 ± 0·02	1·718

The space group is $P2_12_12_1$.

The density was measured by flotation in a mixture of ethylene dibromide and carbon tetrachloride. The calculated number of molecules of the dihydrate in the unit cell was 3·97.

Intensity data

A Unicam Weissenberg camera, with an integrating attachment designed by Dr E. H. Medlin, of this department, was used to collect intensities for the $0kl$ zone of potassium glucuronate. The photographic densities of reflexions on the multiple film packs were measured with a microphotometer. Background measurements were made in the white radiation streak at a lower 2θ angle than the Cu $K\alpha$ reflexion, but as near to it as possible. Probably a better method is to measure the background at a higher 2θ angle than the Cu $K\alpha$ reflexion (Young, 1961), as in this way only the Bragg scattered contribution of the $K\alpha$ wavelength to the peak is measured. However, the miscellaneous scattering under the Bragg peak (Fig. 1) is, to a first approximation, proportional to the intensity of the Bragg reflexion, so that measurement of the background at a lower angle should also give correct relative intensities. The most important requirement is that the background measurement be

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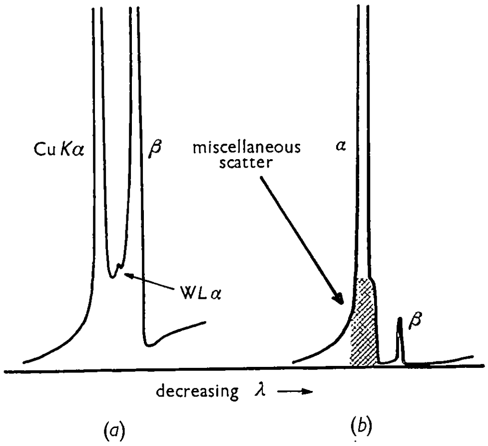

Fig. 1. Profile of a Bragg reflexion (*a*) without filter, (*b*) with a β-filter. The cross-hatched area shows the contribution of miscellaneous scatter to the peak.

made in the white radiation streak passing through the reflexion, to take account of harmonic contributions from other reflexions. It was found that, despite frequent agitation of the developer, the blackest spots were surrounded by a region of apparently low background due to depletion of the developer (the Eberhard effect: Mees, 1944), and so some discretion was needed to decide how close to the peak the background should be measured.

Intensities were derived only from those density measurements which corresponded to use of the straight line part of the characteristic film curve: *i.e.* that part for which density is proportional to exposure. This range was determined for the film and processing conditions used (Ilford Industrial G film, developed 3 minutes in ID 42), by the following method. Three separate exposures in the ratio 1:2:4 were recorded on one film for a small group of reflexions. Taking the shortest exposure of the weakest reflexion to correspond to an exposure of 1, the densities corresponding to exposures (1, 2, 4), (1*p*, 2*p*, 4*p*), (1*q*, 2*q*, 4*q*), ... were measured, each exposure range overlapping with that above and below it. In this way the characteristic film curve shown in Fig. 2(*b*) was derived from the density measurements plotted in Fig. 2(*a*). This derivation of the film curve assumed that the reciprocity law was obeyed: *i.e.* that equal exposures produced the same blackening, no matter what the time of exposure — but this is known to be true for X-rays (Bell, 1936). Fig. 2(*b*) shows that the linear range extended to densities of at least 1·5, the highest density that could be reliably measured with the photometer used.

The weaker reflexions were estimated visually from non-integrated Weissenberg films, by visual comparison with a graduated scale, prepared in the usual way. To make the comparison, the reference reflexion was placed near to the reflexion to be measured, on the white radiation streak at a lower angle than the reflexion.

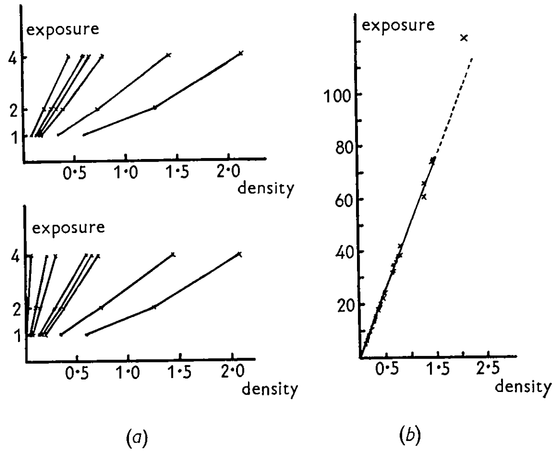

Fig. 2. (*a*) Density measurements on groups of reflexions with exposures in the ratio 1:2:4. (*b*) The characteristic film curve derived from (*a*).

To enable the precision of the measurements to be estimated, intensities $I(0kl)$, $I(0k\bar{l})$ and $I(0\bar{k}\bar{l})$ were measured four times. The average percentage error between repeated measurements on the same reflexion was 1·2%, while the agreement between the average $I(0kl)$ and $I(0\bar{k}\bar{l})$ values was 3·0%. The crystal used was a prism bounded by the {011} zone with faces 0·15 × 0·215 mm, and since the linear absorption coefficient was 50 cm^{-1} the equivalent intensities $I(0kl)$ and $I(0kl)$ were unequal because of absorption. Absorption corrections were made by a graphical method similar to that of Albrecht (1939), except that a fixed number of points was chosen through which incident and emergent beams were drawn. It has been pointed out to the author (private communication from Dr P. Schapiro, Crystallography Laboratory, University of Pittsburgh) that this corresponds to a well known mathematical problem treated by Gauss (*e.g.* see Kopal, 1955), and that one may obtain the same accuracy with fewer points if the weights and locations of the points are properly chosen. The optimum conditions with a fixed number of points and variable weights, fixed weights and variable number of points, or variable numbers of both weights and points, may be determined for each reflexion. (This is important in writing efficient computer

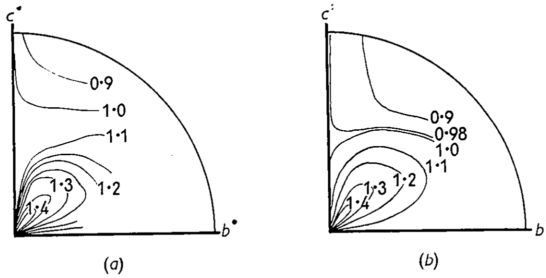

Fig. 3. (*a*) The ratio of calculated absorption factors $A(0kl) : A(0k\bar{l})$. (*b*) The ratio of observed intensities $\bar{I}(0kl) : I(0k\bar{l})$.

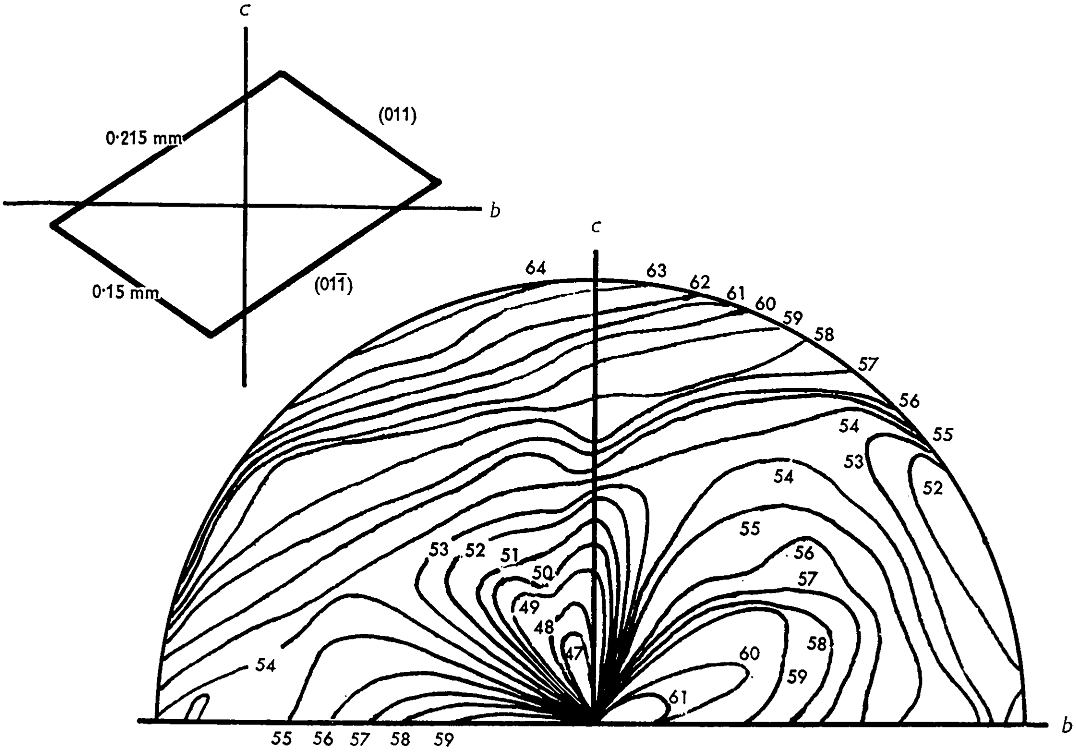

Fig. 4. The calculated absorption effect for a crystal with the shape shown at the top left.

programs for absorption corrections.) However, as 36 points were used in this case, the accuracy should be better than 1%. Confirmation that the observed discrepancies between $I(0kl)$ and $I(0k\bar{l})$ were due to absorption was given by plotting the ratio $I(0kl)/I(0k\bar{l})$ in reciprocal space, and comparing it with a similar plot of the calculated ratio of absorption factors, $A(0kl)/A(0k\bar{l})$. This is shown in Fig. 3, while Fig. 4 shows the absorption factor plotted in the $0kl$ section of reciprocal space.* Application of the absorption correction improved the agreement between $I(0kl)$ and $I(0k\bar{l})$ from 9·0% to 4·9%.

Intensities in the $hk0$ zone of potassium glucuronate and both the $0kl$ and $hk0$ zones of rubidium glucuronate were measured in a similar way, but were only repeated twice. No absorption corrections were made; an average was taken of the measurements for equivalent reflexions. The data were scaled by the methods of Wilson (1942) and Rogers (1954), both of which gave similar results.

Structure determination

The positions of the replaceable atoms were determined from Patterson projections. Fourier syntheses for the (100) projection of rubidium glucuronate were calculated, Beevers–Lipson strips being used, with signs given by the isomorphous replacement method, and also by the heavy atom method using Woolfson's (1956) weighting scheme. The maps were very similar in appearance and allowed the assignment of coordinates to the light atoms. Two further F_o syntheses reduced the R factor to 0·21.

The (001) projection of the rubidium salt was obtained by the same heavy atom method. The R factor was 0·20 in this projection when attention was shifted to the potassium compound.

Refinement of the potassium glucuronate structure

The structure of the potassium isomorph was refined by means of eleven difference-Fourier syntheses of the (100) projection and nine of the (001) projection. The atomic scattering factors used were those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955): the anomalous scattering correction of Dauben & Templeton (1955) was applied to the potassium scattering factor. Most of the difference syntheses and structure factor calculations made use of the programs written for the SILLIAC computer by Dr H. F. Freeman. His assistance, and that of Mr J. E. W. L. Smith and Dr F. M. Lovell, who checked the data, is gratefully acknowledged, as is that of the University of Sydney for making SILLIAC computing time available.

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* It will be obvious from Fig. 4 that the effect of absorption on the shape of the atom in a Fourier synthesis depends very much upon the way that the structure-factor contributions from that atom are distributed in reciprocal space: *i.e.* the apparent shape of an atom will be dependent on its coordinates.

Table 2. *Final atomic parameters*

	Rubidium glucuronate			Potassium glucuronate			
	x	y	z	x	y (average)	z	B (average)
C(1)	500	023	688	5000	0191	6890	2·05
C(2)	255	055	655	2509	0537	6521	2·05
C(3)	250	140	667	2500	1468	6749	1·95
C(4)	340	163	806	3346	1642	8033	1·95
C(5)	570	119	836	5750	1214	8268	2·05
C(6)	675	134	956	6730	1371	9553	2·05
O(1)	485	939	680	4850	9343	6745	2·2*
O(2)	205	038	532	1970	0406	5344	2·05
O(3)	025	178	658	0250	1829	6539	2·5
O(4)	350	247	805	3642	2490	8132	1·6*
O(5)	530	037	818	5321	0358	8105	1·95
O(6)	595	086	038	6027	0903	0345	2·0
O(7)	770	198	970	7908	1981	9660	2·0
O(W1)	060	183	417	0470	1792	4094	2·9
O(W2)	575	068	372	5750	0728	3667	3·9
Rb or K	367	1836	216	3670	1851	2119	2·2*

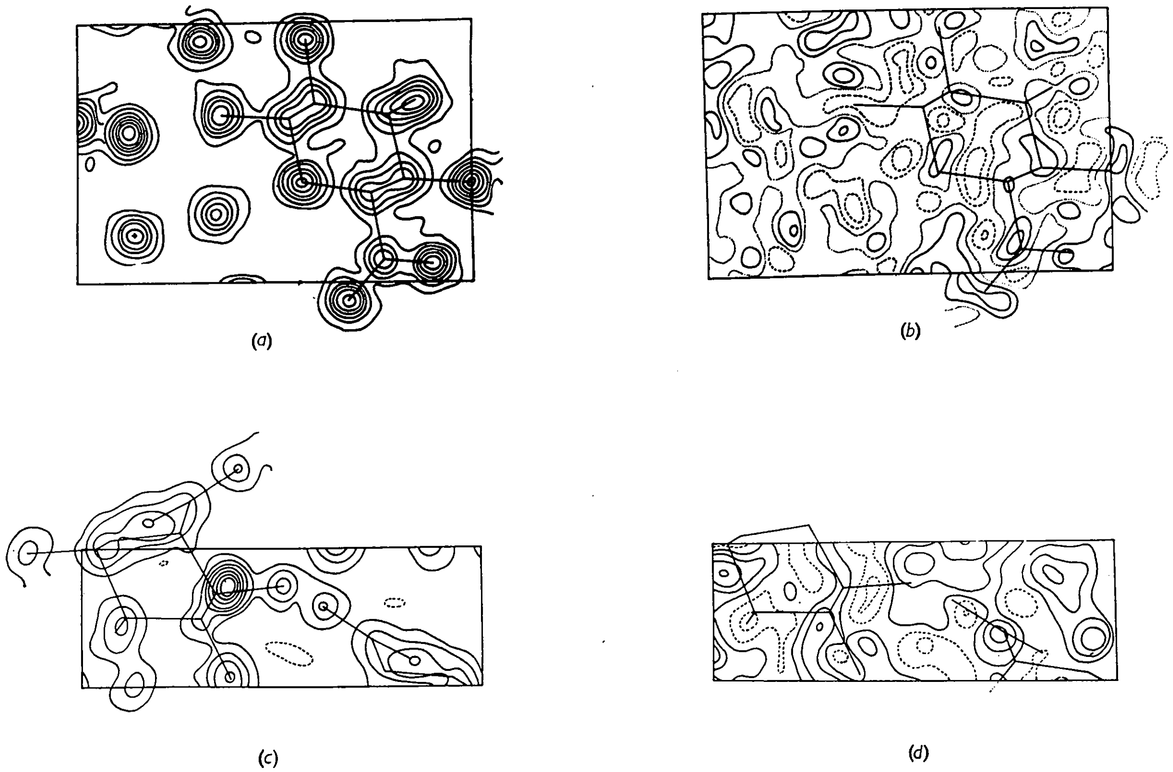
Overall $B=2{\cdot}58$.

* Anisotropic.

Anisotropic temperature factors for the potassium atom and oxygen atom O(1) were obtained by the graphical method of Cochran (1954). In the (100) projection oxygen atom O(4) was also allowed an anisotropic temperature factor. Seven of the thirteen hydrogen atoms were included in the refinement of the (100) projection, but none in the (001) projection because of overlap and the poorer data. The final R value in the $0kl$ zone was 0·071, and in the $hk0$ zone 0·095, including the unobserved reflexions as half the minimum observable $|F_o|$. The final (100) and (001)F_o and difference syntheses are shown in Fig. 5. Standard

Table 3. *Bond lengths and standard deviations*

C(1)–C(2)	$1{\cdot}54_6$ (0·026)	Average 1·545
C(2)–C(3)	$1{\cdot}55_9$ (0·012)	
C(3)–C(4)	$1{\cdot}54_8$ (0·015)	
C(4)–C(5)	$1{\cdot}52_6$ (0·025)	
C(5)–C(6)	$1{\cdot}56_7$ (0·016)	
C(5)–O(5)	$1{\cdot}44_6$ (0·011)	Average 1·427
O(5)–C(1)	$1{\cdot}40_8$ (0·011)	
C(1)–O(1)	$1{\cdot}41_3$ (0·010)	Average 1·401
C(2)–O(2)	$1{\cdot}37_7$ (0·012)	
C(3)–O(3)	$1{\cdot}39_8$ (0·022)	
C(4)–O(4)	$1{\cdot}41_5$ (0·010)	
C(6)–O(6)	$1{\cdot}24_3$ (0·012)	Average 1·224
C(6)–O(7)	$1{\cdot}20_5$ (0·015)	



(a) (b) (c) (d)

Fig. 5. (a) $0kl$ Fourier synthesis—contours at 2 e.Å^{-3} intervals. (b) $0kl$ difference Fourier—0·25 e.Å^{-3} contour intervals. (c) $hk0$ Fourier—3 e.Å^{-3} contour intervals. (d) $hk0$ difference Fourier—0·5 e.Å^{-3} contour intervals.

Table 4. *Observed and calculated structure factors*

(a): $0kl$ zone

l	k	$\|F_o\|$	F_c
0	2	14·2	−7·9
	4	16·1	−16·8
	6	50·4	+51·8
	8	(105·5)	−105·2
	10	18·9	−13·6
	12	6·8	+8·6
	14	41·2	−40·4
	16	33·5	+33·1
	18	24·7	−24·7
	20	uo	−1·8
1	1	40·6	+35·5
	2	17·2	+18·8
	3	14·3	−15·7
	4	22·4	+22·6
	5	17·3	−20·4
	6	21·9	−21·2
	7	51·1	−49·5
	8	59·3	+54·0
	9	14·6	−15·9
	10	68·0	+67·7
	11	7·7	+8·0
	12	13·9	−13·9
	13	uo	−0·6
	14	11·7	+13·2
	15	uo	+4·9
	16	uo	−2·2
	17	13·1	+11·8
	18	22·4	−21·8
	19	5·4	−8·2
	20	12·0	+9·2
	21	uo	+2·3
2	0	(79·8)	−87·9
	1	23·8	+23·5
	2	28·1	+27·0
	3	17·5	+17·2
	4	19·1	−21·3
	5	27·1	+24·8
	6	36·1	−35·8
	7	22·1	−18·9
	8	17·1	+18·7
	9	21·0	−20·5
	10	18·5	+16·0
	11	15·2	+13·3
	12	19·2	−18·0
	13	uo	−4·9
	14	24·6	+23·1
	15	uo	+4·3
	16	24·7	−24·9
	17	5·9	+4·9
	18	uo	+0·7
	19	uo	−2·2
	20	9·3	+7·7
	21	8·0	+10·4
3	1	10·1	−11·6
	2	7·3	+6·9
	3	23·9	+23·8
	4	48·2	−47·9
	5	27·6	−28·9
	6	28·9	+28·9
	7	8·2	−10·1
	8	4·0	+4·6
	9	30·3	−28·6
3	10	uo	−2·7
	11	8·4	+6·1
	12	12·7	+13·0
	13	13·4	−12·8
	14	29·8	−28·7
	15	35·7	+32·6
	16	uo	−0·3
	17	uo	0·0
	18	7·9	+6·7
	19	uo	−2·9
	20	11·4	−11·8
4	0	8·8	+6·1
	1	20·9	−21·4
	2	16·5	−18·6
	3	(80·7)	+82·2
	4	11·3	+10·2
	5	(68·2)	−68·5
	6	18·3	+18·7
	7	12·4	+11·3
	8	uo	+0·9
	9	26·3	+26·6
	10	18·8	+19·2
	11	31·6	−32·7
	12	5·8	−7·6
	13	7·1	+10·2
	14	11·3	−12·1
	15	32·0	−30·5
	16	uo	−4·4
	17	14·3	−13·6
	18	10·8	−11·8
	19	16·6	+15·0
5	1	(106·3)	+105·5
	2	24·7	−23·9
	3	8·1	−8·7
	4	30·0	+32·2
	5	4·9	−3·6
	6	9·0	+8·7
	7	33·8	+31·5
	8	uo	−5·2
	9	35·7	−38·5
	10	12·0	+11·3
	11	10·9	−11·4
	12	24·5	−23·4
	13	24·3	+21·3
	14	5·4	−5·9
	15	32·7	−32·5
	16	11·3	+11·5
	17	15·5	+16·9
	18	6·4	−6·2
	19	uo	−2·3
6	0	81·4	+85·3
	1	35·5	+33·7
	2	28·1	+27·3
	3	3·0	+2·3
	4	11·3	+8·6
	5	uo	+1·1
	6	18·2	−19·0
	7	22·6	−24·3
	8	uo	−1·0
	9	11·7	−13·0
	10	22·4	−22·4
	11	32·9	+31·6
6	12	uo	−4·1
	13	uo	−5·6
	14	uo	−0·3
	15	uo	−2·0
	16	5·3	+3·9
	17	6·3	+5·9
	18	uo	−3·3
	19	12·8	−14·5
7	1	51·1	−50·4
	2	41·6	+40·4
	3	uo	+0·5
	4	21·6	−21·9
	5	uo	+1·9
	6	21·1	+21·2
	7	36·3	−35·9
	8	37·5	+38·3
	9	17·8	+18·3
	10	23·7	+22·3
	11	6·7	+7·1
	12	6·7	+7·5
	13	8·3	−9·2
	14	8·2	−8·7
	15	21·0	+20·9
	16	uo	−1·9
	17	9·3	−9·8
8	0	61·5	−63·1
	1	21·4	+19·6
	2	6·1	+5·5
	3	27·1	+27·7
	4	5·1	−6·6
	5	19·3	−19·2
	6	15·2	−12·6
	7	3·8	−3·8
	8	5·4	−5·7
	9	18·0	−15·5
	10	14·1	+13·4
	11	12·3	−11·7
	12	uo	−1·1
	13	14·6	+13·9
	14	13·4	+12·3
	15	15·2	−15·0
	16	4·2	−5·0
9	1	10·9	+9·1
	2	9·8	+10·2
	3	13·8	−13·8
	4	34·0	−32·9
	5	12·4	−14·7
	6	21·0	+21·6
	7	33·5	+30·9
	8	uo	+2·5
	9	12·8	−10·8
	10	15·2	−13·8
	11	uo	+3·7
	12	7·4	+9·0
	13	8·2	+9·2
	14	25·0	−24·1
	15	8·1	−8·8
	16	5·1	−5·0
10	0	7·5	+9·3
	1	8·0	+6·4
10	2	15·4	−15·0
	3	4·7	−7·5
	4	12·4	+11·8
	5	5·4	+7·0
	6	18·1	+17·5
	7	uo	−0·1
	8	uo	−2·3
	9	uo	−1·3
	10	uo	+2·3
	11	7·2	+9·5
	12	14·7	+11·3
	13	12·7	−17·1
	14	15·4	−16·1
	15	12·9	−12·8
11	1	7·8	−7·9
	2	14·8	−14·4
	3	5·5	+8·8
	4	16·7	+15·3
	5	28·0	+27·6
	6	uo	+1·2
	7	7·9	−8·3
	8	7·0	+5·9
	9	13·6	+13·3
	10	14·6	+14·3
	11	8·8	−9·8
	12	7·8	−9·5
12	0	18·8	−19·2
	1	uo	−1·5
	2	6·1	+6·1
	3	14·1	+12·9
	4	uo	−1·5
	5	13·0	+13·4
	6	6·7	−7·7
	7	uo	+2·9
	8	13·0	+13·0
	9	6·0	+6·9
	10	10·6	−9·6
	11	3·8	−3·6
13	1	22·1	−18·9
	2	13·8	+10·9
	3	uo	+0·5
	4	8·9	−11·8
	5	uo	+0·4
	6	5·7	+5·6
	7	uo	−1·8
	8	uo	+2·9
	9	4·9	+4·0
14	0	13·5	−11·0
	1	7·0	+6·3
	2	10·0	−9·1

$R = 0{\cdot}071$

uo = unobserved

i.e. $|F_o| < 3{\cdot}0$ approx.

The five $|F_o|$ in brackets have been corrected for extinction

$\Sigma F_o = 3775{\cdot}7$

$\Sigma F_o = 3750{\cdot}0$ excl. uo

Table 4 (*cont.*)

(*b*): *hk*0 zone

h	k	$\lvert F_o\rvert$	F_c	h	k	$\lvert F_o\rvert$	F_c	h	k	$\lvert F_o\rvert$	F_c	h	k	$\lvert F_o\rvert$	F_c
0	2	14·6	−16·9	2	0	33·9	+32·2	3	11	34·9	+34·9	5	4	uo	− 4·1
	4	16·3	−18·4		1	36·9	+32·6		12	7·7	+ 5·7		5	13·2	−13·9
	6	50·4	+48·4		2	35·4	+36·2		13	15·9	−15·2		6	14·9	+15·7
	8	105·5	−109·4		3	109·0	−106·6		14	5·2	− 7·7		7	6·4	+ 4·8
	10	17·5	− 9·3		4	6·6	+ 6·2		15	8·3	+ 6·8		8	9·6	−12·0
	12	6·9	+ 7·1		5	17·2	−25·1		16	5·5	− 5·3		9	uo	+ 0·4
	14	38·7	−41·6		6	8·7	− 7·8		17	11·8	+10·4		10	18·0	−17·6
	16	33·5	+34·5		7	29·6	+29·3		18	16·9	+17·8		11	12·1	−11·9
	18	24·4	−23·0		8	17·6	−16·1		19	8·2	− 9·8		12	8·1	+ 8·5
	20	uo	− 2·5		9	20·5	−19·1						13	11·7	+12·1
					10	16·5	+16·2	4	0	9·4	− 1·9		14	10·9	−10·8
1	1	32·5	+36·2		11	12·2	− 6·6		1	39·2	−43·7		15	6·9	+ 5·0
	2	48·8	−54·8		12	14·8	+15·2		2	37·0	+42·1				
	3	22·7	−23·4		13	27·5	+22·8		3	uo	− 2·6	6	0	8·2	+ 8·8
	4	40·1	−35·1		14	9·7	− 8·9		4	10·6	+11·0		1	22·1	−21·7
	5	59·3	+59·7		15	22·5	−23·1		5	4·5	+ 4·3		2	4·7	− 3·9
	6	13·3	+11·4		16	5·1	+ 4·6		6	uo	+ 5·5		3	uo	+ 1·3
	7	12·5	+ 7·8		17	13·8	+14·9		7	13·9	−14·3		4	6·5	+ 9·4
	8	10·1	− 4·9		18	uo	+ 0·6		8	1·9	− 4·7		5	uo	− 3·0
	9	57·2	−61·9		19	uo	− 1·8		9	25·5	+23·8		6	13·2	+11·8
	10	21·7	−21·5		20	15·9	−15·4		10	19·2	−16·8		7	20·4	+20·2
	11	8·9	+ 7·1						11	uo	+ 1·6		8	3·4	− 3·3
	12	24·0	−24·3	3	1	37·3	−40·2		12	17·4	−17·1				
	13	12·9	−13·4		2	52·6	+52·1		13	11·3	−11·3	7	1	uo	− 1·3
	14	12·5	− 9·1		3	46·4	−51·3		14	20·4	+16·4		2	4·4	− 5·7
	15	10·7	−11·4		4	59·4	−61·4		15	9·0	+ 7·2		3	uo	+ 0·7
	16	5·3	− 5·1		5	66·9	+63·8		16	10·9	−10·0		4	11·1	+12·2
	17	18·1	+15·2		6	8·9	− 9·2		17	3·8	− 5·8				
	18	uo	− 3·1		7	4·2	− 4·2								
	19	uo	− 3·2		8	uo	− 6·1	5	1	uo	− 4·8				
	20	10·0	+10·1		9	6·4	− 7·4		2	25·5	+27·2				
	21	8·3	+ 7·6		10	48·2	−51·7		3	4·7	+ 5·0				

$R = 0{\cdot}095$

$\Sigma F_o = 2104{\cdot}8$

$\Sigma F_o = 2093{\cdot}3$ excl. uo

deviations of the atomic coordinates were computed from the difference syntheses by the method of Cruickshank (1949). The final atomic coordinates for both potassium and rubidium glucuronate are shown in Table 2. Table 3 shows the measured lengths and estimated standard deviations of the bonds in potassium glucuronate. It is thought that the standard deviations of the (001) projection have been slightly underestimated because of overlap in projection. Table 4 contains a list of $\lvert F_o\rvert$ and F_c for potassium glucuronate.

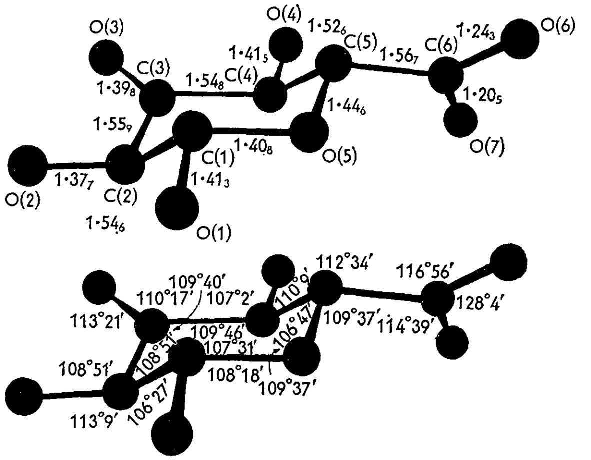


Fig. 6. The observed bond lengths and bond angles in the glucuronate ion (from the potassium salt).

Discussion of the molecular structure

The results show that the β-D-glucuronic acid molecule has the conformation $C1$ (using the nomenclature of Reeves (1951), and also the modified nomenclature of Isbell & Tipson (1960)) as was predicted by Reeves from considerations of molecular stability. The configuration of β-D-glucuronic acid is such that it is possible for all of the hydroxyl groups to be in the low-energy equatorial position, so it is to be expected that the molecule will be quite stable and free of strain.

None of the C–C or C–O bonds in the pyranose ring differ significantly from the usually accepted single-bond lengths of 1·545 and 1·427 Å respectively. The mean bond angle in the ring is 108° 42′, and the angle at O(5) is much nearer the tetrahedral angle than is the case in α-rhamnose monohydrate (McGeachin & Beevers, 1957), where the angle is about 120°. The mean of the eight C–C–OH angles is 109° 37′. The mean of the four C–OH bonds is 1·401 Å, and deviations from the mean are not significant. The C(5)–C(6) bond is long for one of sp^3–sp^2 character, while the C–O bonds of the carboxyl group are rather short, averaging 1·224 Å. The O–C–O angle in the carboxyl group is 128° 4′, and there is no significant asymmetry of the group. Fig. 6 shows the molecular dimensions.

Molecular packing

There are two types of bond involved in the packing arrangement, *viz.* electrostatic forces between the K^+ ion and the electronegative oxygen atoms, and hydrogen bonds between pairs of oxygen atoms.

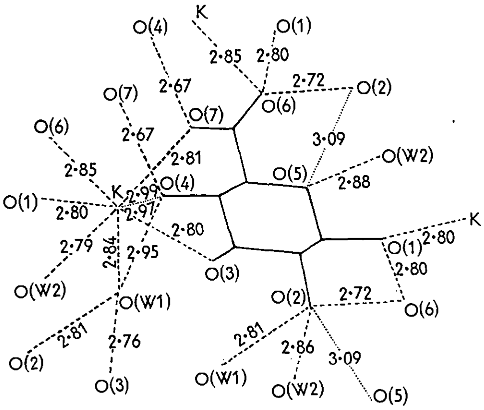


Fig. 7. (100) projection of potassium glucuronate showing the intermolecular bond distances.

The (100) projection of the bonding system is shown in Fig. 7, from which it may be seen that all oxygen atoms outside the pyranose ring form at least two external bonds, and even O(5) of the ring acts as an acceptor in a hydrogen bond with the oxygen atom of the second water molecule, O(W2). Oxygen atoms O(2), O(4), and O(6) each form three external bonds.

The high temperature factor of O(W2) may be understood from consideration of the intermolecular bonding. O(W2) forms only three bonds, compared with the four of O(W1), obviously resulting in looser bonding of O(W2) and consequently a larger thermal vibration parameter.

The K^+ ion has sixfold coordination, the K^+–O bond lengths all being very nearly equal and ranging from 2·79–2·85 Å with an average value of 2·815 Å. There are also close approaches of 2·97–2·99 Å, between the K^+ ion and two different O(4) atoms. The environment of the K^+ ion is illustrated in Fig. 8.

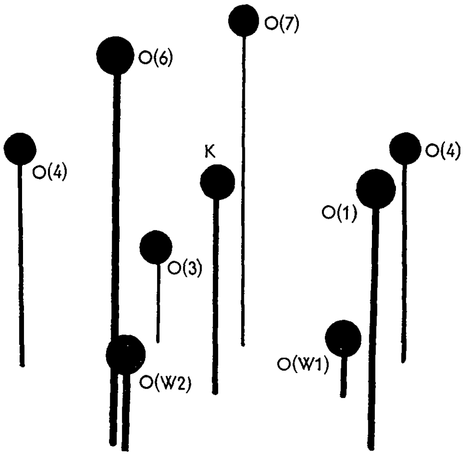


Fig. 8. Illustration of the environment of the potassium ion in potassium glucuronate.

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References

Albrecht, G. (1939). *Rev. Sci. Instrum.* **10**, 221.
Bell, G. E. (1936). *Brit. J. Radiol.* **9**, 578.
Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.
Cochran, W. (1954). *Acta Cryst.* **7**, 503.
Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 154.
Dauben, C. H. & Templeton, D. H. (1955). *Acta Cryst.* **8**, 841.
Isbell, H. S. & Tipson, R. S. (1960). *J. Res. Nat. Bur. Stand.* **64A**, 171.
Keihn, F. & King, A. J. (1955). *Acta Cryst.* **8**, 354.
Kopal, Z. (1955). *Numerical Analysis*, **VII**, 9. New York: Wiley.
McGeachin, H. McD. & Beevers, C. A. (1957). *Acta Cryst.* **10**, 227.
Mees, C. E. K. (1944). *The Theory of the Photographic Process*. New York: MacMillan.
Reeves, R. E. (1951). *Advances in Carbohydrate Chemistry*, **6**, 108.
Rogers, D. (1954). *Acta Cryst.* **7**, 628.
Wilson, A. J. C. (1942). *Nature, Lond.* **150**, 152.
Woolfson, M. M. (1956). *Acta Cryst.* **9**, 804.
Young, R. A. (1961). Technical Report No. 2, Engineering Experiment Station, Georgia Inst. Tech., Atlanta, Ga.