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The Crystal and Molecular Structure of β-D-Glucose

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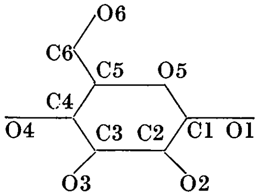
β-D-Glucose ($C_6H_{12}O_6$) consists of a pyranose ring with substituents which are all equatorial. It crystallizes in the orthorhombic system with $a = 9{\cdot}29 \pm 0{\cdot}04$, $b = 12{\cdot}65 \pm 0{\cdot}02$, $c = 6{\cdot}70 \pm 0.02$ Å. The space group is $P2_12_12_1$ with 4 molecules in the cell. The crystal structure has been solved by a combination of a direct method and trial and error applied to two-dimensional data. The final refinement was obtained by an anisotropic least-squares analysis of approximately 850 observed three-dimensional structure factors. The three positional and six thermal parameters of all atoms except hydrogens have been determined.

The pyranose ring is in the Sachse *trans* configuration and the ring oxygen bond angle is determined as 113°. The detailed stereochemistry is compared with that of α-D-glucose and cellobiose.

Introduction

β-D-glucose ($C_6H_{12}O_6$) consists of a pyranose ring with substituents which are all equatorial:



Cellulose is a polymer of β-D-glucose in which glucose residues are linked 1 to 4. The author has undertaken a detailed examination of the crystal structure of β-D-glucose as a preliminary to a study of other more complex cellulose oligosaccharides. McDonald & Beevers (1950, 1952) determined the crystal structure of α-D-glucose and more recently Killean, Ferrier & Young (1962) determined the structure of α-D-glucose monohydrate. Both structures showed that the pyranose ring possessed the Sachse *trans* configuration. Sponsler & Dore (1931) studied the X-ray powder diagram of β-D-glucose but made no deductions regarding the size of the cell. The author published a preliminary report on this structure (Ferrier, 1960) in which the Sachse *trans* configuration was confirmed. In the present publication it will be seen that the detailed structure has been obtained with reasonably high accuracy despite experimental difficulties both in obtaining and in preserving crystals.

Experimental

The β-D-glucose was prepared by the method of Hudson & Dale (1917). Crystals, which are reasonably satisfactory for X-ray work, were found to be much more difficult to obtain than crystals of either α-D-glucose or α-D-glucose monohydrate. The crystals used were grown by using acetone to precipitate them from a solution of β-D-glucose in water, the temperature being maintained just above the freezing point. The crystals obtained in this way showed (101) and (10$\bar{1}$) as faces and were found to absorb water from the atmosphere with the resultant formation of a powder deposit of α-D-glucose monohydrate on the surface. This process took place sufficiently slowly to permit normal X-ray photography, although clearly this continuous change must affect the X-ray intensities. In this investigation the crystals, which were kept dry under vacuum when not in use, were frequently renewed and the effect was ignored.

The unit-cell dimensions were determined as

$$a = 9{\cdot}29 \pm 0{\cdot}04,\ b = 12{\cdot}65 \pm 0{\cdot}02,\ c = 6{\cdot}70 \pm 0{\cdot}02\ \text{Å}.$$

The molecular weight of β-D-glucose is 180. Assuming 4 molecules in the unit cell, the density is calculated as 1·520 g.cm^{-3} (*cf.* observed density of 1·535 g.cm^{-3}). The space group was determined as $P2_12_12_1$.

The intensities of the X-ray reflexions were measured visually from equi-inclination Weissenberg photographs obtained with Cu $K\alpha$ radiation. No attempt to correct for absorption was made, as the crystals used were all small and of reasonably uniform dimensions.

Determination of the structure

The intensities of the X-ray reflexions were placed approximately on the absolute scale by the method of Wilson (1942). The space group $P2_12_12_1$ is non-centrosymmetric but each of the three principal zones is centrosymmetric. The method of Grant, Howells & Rogers (1957) was applied to each of these zones.

This method is essentially a sorting procedure for handling the well established relationship between three unitary structure factors $U(h)$, $U(h')$, $U(h+h')$, *viz.*

$$S(h').S(h+h') \approx S(h) \qquad (1)$$

where S means 'the sign of'.

In this approach 'coincidences' are recorded using a variant of (1)

$$S(A).S(B).S(C) \approx +1 \qquad (2)$$

e.g. $S(A).S(B)$ can give several $S(C)$ and in this case the signs of $S(C)$ are said to coincide. In this way it is possible to obtain for each reflexion a set of relationships from which it may be possible to derive a tentative set of signs. These are then used in an iterative process which is continued until there is no significant change in the signs given for each plane.

Each principal zone can be considered in the centrosymmetric plane group *pgg* and each zone is conveniently subdivided into 4 groups according to whether the Miller indices are even or odd. The groups are (even, even), (odd, odd), (even, odd) and (odd, even). In practice it was found convenient to follow Grant, Howells & Rogers (1957) and multiply the unitary factors by 20 in order to round up each factor to a single integer. This made computation of the weight of the products very much simpler.

In *pgg* one sign can be chosen from two of the groups (odd, odd), (even, odd) and (odd, even). This establishes the origin. For the *hk*0 zone 120 and 210 were both chosen as positive. The results for this zone are given in Table 1; this shows the signs given after five iterations, at which stage there were no further significant changes. Thirty-nine unitary factors greater than 0·10 were used and 33 signs were regarded as determined, taking due account of weights. It was subsequently discovered that 27 of the 33 signs were correct and that the 6 incorrect signs had a relatively low unitary value.

The *h*0*l* zone gave 25 reflexions with unitary factors greater than 0·10. Nineteen signs were regarded as determined in five iterations. In this case 4 of the 6 omitted had a clear majority sign (Number + > Number — or *vice versa*) in opposition to the sign given by taking account of the weight. This made the analysis in this zone much less consistent than in the *hk*0 zone, where this trouble did not arise. Seventeen of the 19 signs determined were subsequently found to be correct. Three of the four ambiguous terms were found to have been predicted correctly on the basis of the number of terms favouring the majority rather than the weights.

The application of the method to the 0*kl* zone was not successful presumably because there were many fewer reflexions of large unitary factor. The analysis was very inconsistent; 21 planes of unitary factor greater than 0·10 were used; 14 signs were regarded as determined and it was subsequently discovered that only 5 of these were correct.

Table 1. *hk*0 *zone*; *signs obtained from the direct method*

Plane	$20U$	Number +	Number −	Sign chosen
040	6	5	3	+
0,12,0	6	5	3	+
0,14,0	12	11	0	+
200	6	7	9	−
400	5	9	6	+*
600	4	5	6	omit
2,14,0	8	6	8	−
420	3	9	3	+*
4,14,0	4	8	4	+*
660	7	5	5	−†
6,12,0	6	1	3	−
6,14,0	5	6	3	+
820	6	10	0	+
860	8	10	0	+
210	4	13	4	+
270	3	4	4	omit
2,15,0	3	5	5	omit
490	4	5	3	+
4,13,0	4	6	3	+
8,11,0	5	4	2	+
170	3	6	15	−*
1,15,0	7	2	6	−
370	4	6	6	omit
3,15,0	7	5	4	+
570	4	7	9	−
710	4	4	7	−
770	4	3	6	−*
7,13,0	3	2	6	−
990	6	0	7	−
120	4	12	3	+
1,10,0	3	8	1	+
1,14,0	13	9	2	+
380	4	6	8	−*
3,12,0	4	5	4	omit
760	3	6	3	+
780	9	0	12	−
7,12,0	10	0	6	−
920	3	4	3	omit
980	7	7	2	+

* These signs were subsequently found to be incorrect.

† Chosen negative because of much superior negative weight.

The *hk*0 structure factors, the phases of which had been given by the direct method (see Table 1), were used to compute a Fourier synthesis for the (001) projection. This is shown in Fig. 1(*a*), where it can be compared with the final computation for the projection, shown in Fig. 1(*b*). A detailed examination of this projection showed that if it was to be identified with molecules of β-D-glucose, there could be only one possible interpretation of the position of the ring, containing 5 carbon atoms and 1 oxygen atom. This position is shown in Fig. 1(*a*) with the atomic centres joined by full lines. The shape of this ring allows a distinction to be made between the Sachse *trans* and *cis* configurations, since these have different projection geometry. It was found impossible to obtain a reasonable fit with the *cis* configuration, whereas the interpretation shown in Fig. 1(*a*) is a very reasonable fit to the *trans* configuration. Although the position of the ring was determined directly, there

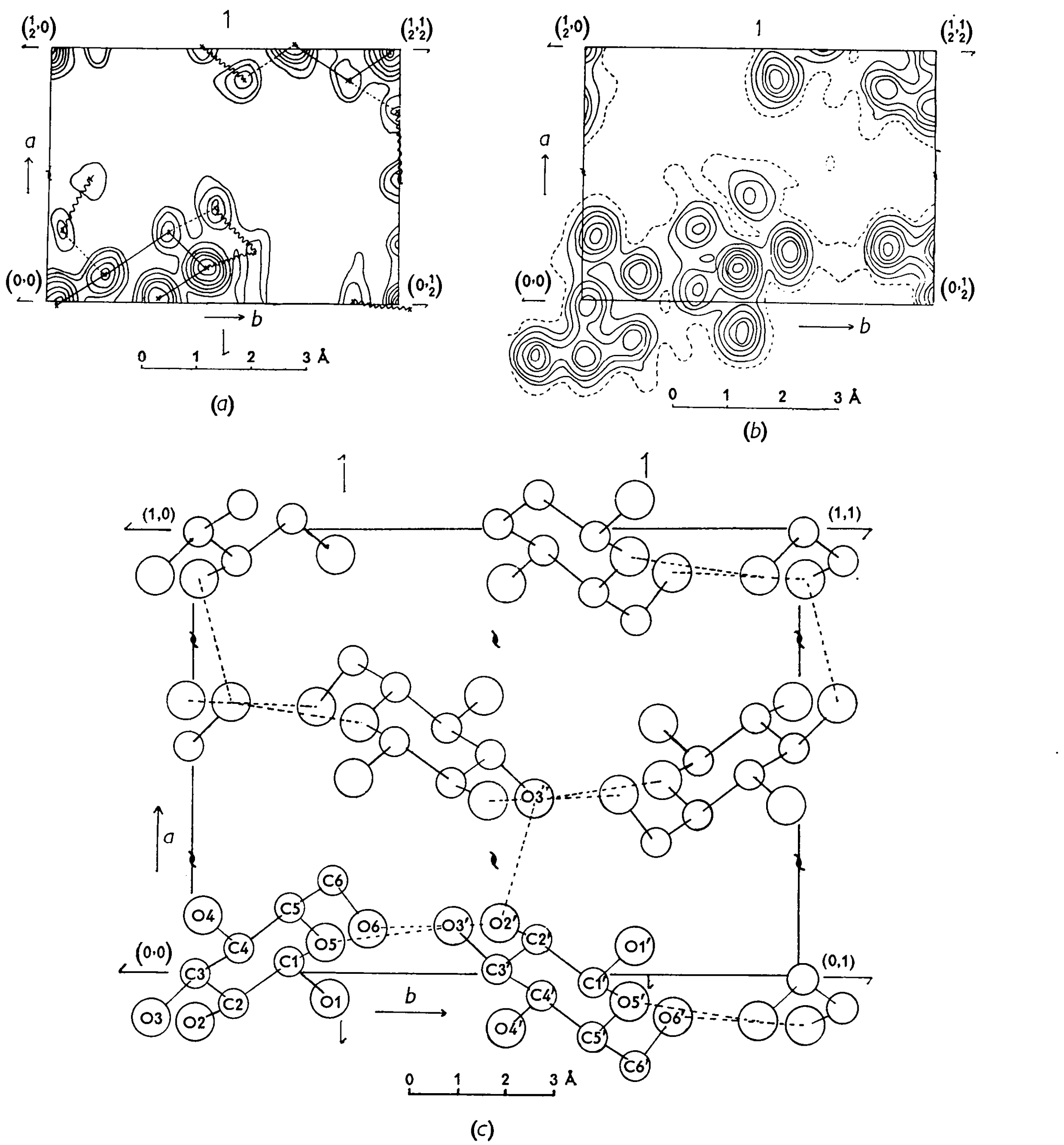


Fig. 1. (*a*) Initial Fourier synthesis on (001) projection. Contours are drawn at arbitrary intervals. (*b*) Final Fourier synthesis on (001) projection. Contours are drawn at intervals of 2 e.Å^{-2}, the dashed line corresponding to 2 e.Å^{-2}. (*c*) Positions of the atoms in the (001) projection. Hydrogen bonds are shown as dashed lines.

was still doubt as to which of the atoms within this ring is the oxygen. This is governed by the attachments to the ring. In Fig. 1(*a*) attachments regarded as certain are shown as dotted lines, while possible attachments are shown as wavy lines. It was found that four different structures were possible, two for each of the two possible positions of O(5), which has no 'certain' attachment. No attempt was made at this stage to distinguish between these structures. Instead the *h*0*l* structure factors, the phases of which had been given from the direct method, were used to compute a Fourier synthesis of the (010) projection. This gave a general confirmation of the position of the molecule as given by the (001) projection and also gave a clue as to the position of the molecule along *c*. However, there was no resolution and no distinction could be made between the 4 possible structures.

It was necessary, therefore, to resort to trial and error in the (*hk*0) zone. Attachments were added one at a time and detailed structure-factor calculations

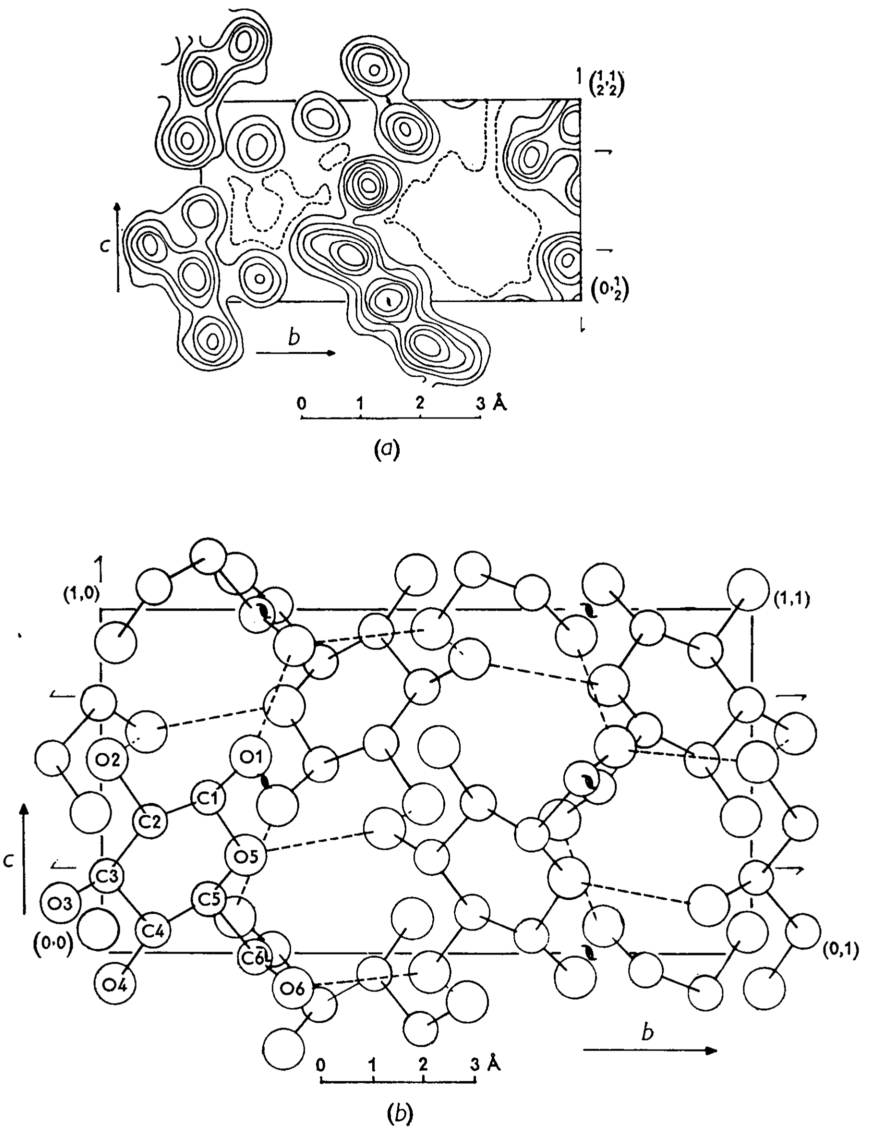


Fig. 2. (*a*) Final Fourier synthesis on (100) projection. Contours are drawn at intervals of 2 e.Å^{-2}, the dashed line corresponding to 2 e.Å^{-2}. (*b*) Positions of the atoms in the (100) projection. Hydrogen bonds are shown as dashed lines.

performed. In the initial stages the possible structures were reduced to two. Another Fourier synthesis was computed, only the phases which were confirmed by both structures being used. The result was examined in detail and more structure-factor calculations performed. In this way a single structure emerged. The final Fourier synthesis for the (001) projection was obtained after six refinements and is shown in Fig. 1(*b*). The details are best understood by reference to Fig. 1(*c*); this shows complete resolution and enables the *x* and *y* coordinates of all the atoms to be obtained with some accuracy.

The bond lengths and angles obtained by McDonald & Beevers for α-D-glucose were used for the transfer from the (001) to (100) projection. An approximate position of the molecule along *c* was given from the (010) projection. After some initial trial and error, a reasonable measure of agreement for the structure factors of this zone was obtained. Three Fourier refinements were then performed on this zone giving the result shown in Fig. 2(*a*). It shows partial resolution. The *z* coordinates of C(5), C(6) and O(6) remain undetermined although reasonable estimates can be made, particularly of the position of C(6).

A (010) projection, using phases based on the coordinates already obtained, is shown in Fig. 3. The Fourier synthesis (not shown here) gives no resolution in detail. There is partial resolution of C(5) and C(6) which allows their *z* coordinates to be determined with higher accuracy than from the (100) projection.

The outstanding parameter is the *z* coordinate of O(6), which was determined at this stage by trial

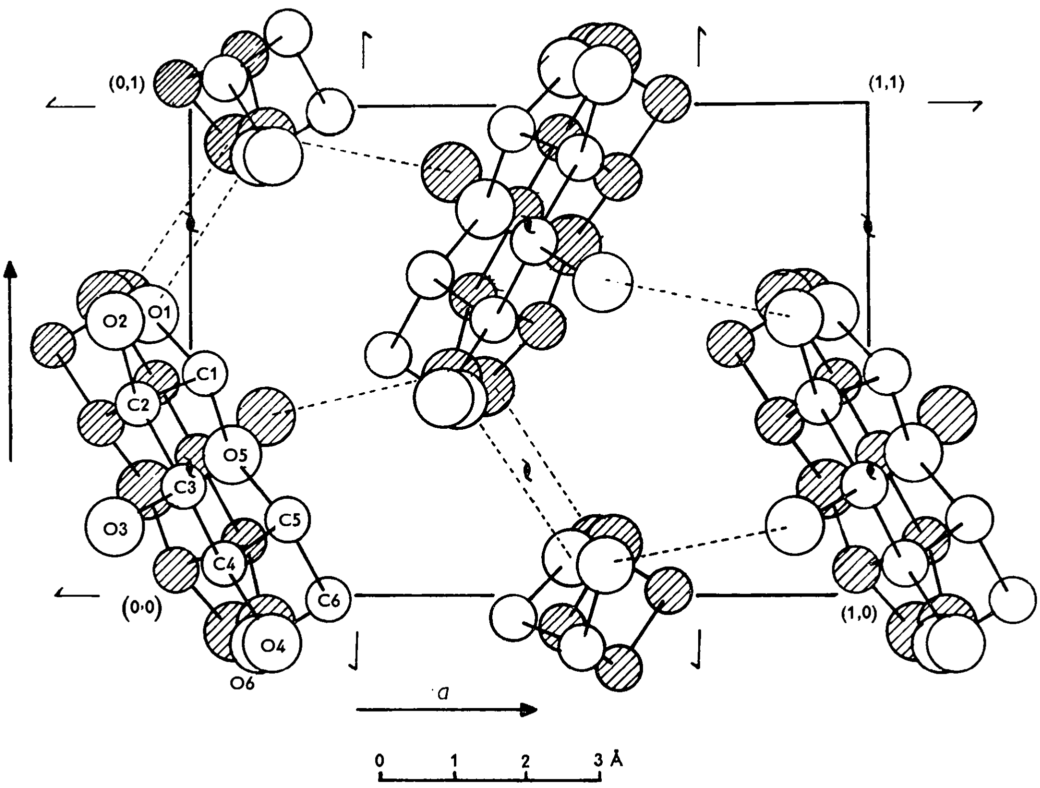


Fig. 3. Positions of the atoms in the (010) projection. Hydrogen bonds are shown as dashed lines.

and error, using bond-length and bond-angle considerations. In this way a complete set of coordinates was obtained. These were given in detail in the preliminary report on the structure. They were used to compute structure factors and the value of

$$R = (\Sigma|F_o|-|F_c|)/(\Sigma|F_o|)$$

was evaluated for all three principal zones using an isotropic temperature factor of $\exp\left[-1{\cdot}5\,(\sin\theta/\lambda)^2\right]$ for all atoms. The values obtained were

$$R(hk0)=0{\cdot}21,\ R(0kl)=0{\cdot}22,\ R(h0l)=0{\cdot}26$$

for all observed reflexions.

Refinement of the structure

The positions of all the hydrogen atoms attached to carbons were calculated from the determined carbon–oxygen positions using a C–H bond of 1·10 Å. The positions of the hydrogens attached to the oxygens were calculated from a knowledge of the hydrogen-bonding system (discussed later) by assuming the hydrogens to be located within the bond and using an O–H bond length of 1 Å. The hydrogen attached to O(4) has no part in the hydrogen bonding and its position had to be estimated. The contributions of the hydrogens to the structure factors were computed with the use of an arbitrarily assigned temperature

Table 2. *Positional parameters for carbon and oxygen atoms and their estimated standard deviations*

Atom	x (Å)	σ_x (Å)	y (Å)	σ_y (Å)	z (Å)	σ_z (Å)
C(1)	0·189	0·009	2·039	0·006	3·075	0·007
C(2)	−0·743	0·009	0·902	0·006	2·603	0·007
C(3)	−0·065	0·008	0·077	0·006	1·503	0·007
C(4)	0·444	0·007	0·972	0·006	0·388	0·007
C(5)	1·374	0·008	2·038	0·006	0·974	0·008
C(6)	1·952	0·007	3·016	0·008	−0·023	0·009
O(1)	−0·570	0·007	2·885	0·006	3·900	0·006
O(2)	−1·038	0·007	0·074	0·005	3·746	0·007
O(3)	−1·067	0·006	−0·821	0·005	0·964	0·007
O(4)	1·205	0·007	0·212	0·006	−0·579	0·007
O(5)	0·595	0·006	2·809	0·004	1·932	0·005
O(6)	0·920	0·007	3·717	0·005	−0·725	0·006

Table 3. *Calculated hydrogen coordinates*

Atom	x	y	z
H(C1)	1·00 Å	1·67 Å	3·58 Å
H(C2)	−1·69	1·34	2·21
H(C3)	0·75	−0·49	1·92
H(C4)	−0·41	1·46	−0·11
H(C5)	2·23	1·52	1·52
H(C6)	2·60	3·61	0·61
H′(C6)	2·59	2·40	−0·60
H(O1)	−0·02	3·19	4·67
H(O2)	−1·98	0·35	3·96
H(O3)	−0·90	−1·79	1·13
H(O4)	1·67	0·11	−1·46
H(O6)	0·96	4·71	−0·60

Table 4. *B_{ij} values for carbon and oxygen atoms*

Atom	B_{11}	B_{22}	B_{33}	B_{23}	B_{31}	B_{12}
C(1)	0·01663	0·00355	0·01695	0·00346	0·00148	0·00000
C(2)	0·01575	0·00306	0·01707	0·00138	0·00630	−0·00021
C(3)	0·01295	0·00143	0·02028	0·00139	0·00079	−0·00010
C(4)	0·01097	0·00353	0·01841	−0·00099	0·00656	0·00165
C(5)	0·01256	0·00355	0·02167	0·00299	0·00455	−0·00029
C(6)	0·01120	0·00630	0·02471	0·00361	0·00754	−0·00370
O(1)	0·01983	0·00699	0·02459	−0·00822	0·00543	0·00040
O(2)	0·01824	0·00393	0·03137	0·00915	0·00601	0·00007
O(3)	0·01368	0·00312	0·03465	0·00019	−0·00520	−0·00296
O(4)	0·02000	0·00654	0·02785	−0·00847	0·01480	0·00062
O(5)	0·01272	0·00331	0·01814	0·00079	0·00511	−0·00155
O(6)	0·02159	0·00466	0·01737	−0·00004	−0·00497	−0·00341

factor of $\exp[-3{\cdot}0(\sin\theta/\lambda)^2]$. These contributions were then added to the calculated structure factors.

A series of difference Fourier syntheses was then computed for the (001) and (100) projections. These led initially to a relatively fast refinement of the positional parameters of the resolved atoms. When this refinement slowed down, the three-dimensional data of approximately 850 observed structure factors were used for least-squares refinement on the DEUCE computer of the University of Glasgow, using the program written by Rollett (1960). This program allows thermal anisotropy and refines 3 positional and 6 thermal parameters for each atom.

Five cycles of refinement were performed on the parameters of the carbon and oxygen atoms. No refinement was done on hydrogen coordinates at any stage.

Table 2 gives the final coordinates of the carbon and oxygen atoms together with their standard deviations, which are estimated directly from the least squares derived variance-covariance matrix. The hydrogen parameters used in the final calculation are given in Table 3.

The least-squares program uses the expression

$$2^{-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{23}kl+B_{31}lh+B_{12}hk)}$$

instead of $2^{-B\sin^2\theta}$ for each atom. Table 4 shows the final B_{ij} values for each atom.

With these coordinates and thermal parameters a detailed calculation of the structure factors was performed. This is shown in Table 8. The final R value is 0·11.

Table 5. *Molecular dimensions with standard deviations*

C(1)–C(2) = 1·544 ± 0·011 Å
C(2)–C(3) = 1·533 ± 0·011
C(3)–C(4) = 1·517 ± 0·010
C(4)–C(5) = 1·531 ± 0·010
C(5)–C(6) = 1·512 ± 0·011
C(1)–O(1) = 1·404 ± 0·010
C(2)–O(2) = 1·442 ± 0·010
C(3)–O(3) = 1·450 ± 0·009
C(4)–O(4) = 1·447 ± 0·010
C(5)–O(5) = 1·455 ± 0·009
C(1)–O(5) = 1·437 ± 0·009
C(6)–O(6) = 1·432 ± 0·010

C(1)–C(2)–C(3) = 110·4 ± 0·5°
C(2)–C(3)–C(4) = 110·9 ± 0·5
C(3)–C(4)–C(5) = 109·5 ± 0·5
C(4)–C(5)–O(5) = 107·2 ± 0·5
C(5)–O(5)–C(1) = 113·1 ± 0·5
O(1)–C(1)–C(2) = 107·3 ± 0·6
C(1)–C(2)–O(2) = 107·7 ± 0·5
O(2)–C(2)–C(3) = 110·5 ± 0·5
C(2)–C(3)–O(3) = 107·1 ± 0·6
O(3)–C(3)–C(4) = 108·9 ± 0·5
C(3)–C(4)–O(4) = 110·9 ± 0·5
O(4)–C(4)–C(5) = 107·6 ± 0·5
C(4)–C(5)–C(6) = 115·5 ± 0·6
C(5)–C(6)–O(6) = 111·4 ± 0·5
O(1)–C(1)–O(5) = 107·3 ± 0·5
C(2)–C(1)–O(5) = 108·8 ± 0·5
C(6)–C(5)–O(5) = 107·3 ± 0·5

Discussion of the structure

(*a*) *Dimensions of the molecule*

The bond lengths and bond angles of β-D-glucose were computed from the coordinates given in Table 2.

Table 6. *The principal axes, r.m.s. displacements and orientations*

Atom	r	$\mu(r)$	$\theta(r, a)$	$\theta(r, b)$	$\theta(r, c)$
C(1)	1	0·23 Å	8°	87·5°	83°
	2	0·19	99	53·5	38
	3	0·11	93	143·5	53·5
C(2)	1	0·23	27	88·5	63
	2	0·15	113·5	53	47
	3	0·12	102	149	62
C(3)	1	0·20	11·5	89	78·5
	2	0·18	102	79	16·5
	3	0·09	91·5	170	80
C(4)	1	0·22	39·5	82·5	51
	2	0·16	108·5	142·5	58·5
	3	0·11	123	50	57·5
C(5)	1	0·22	44·5	79·5	47·5
	2	0·18	129·5	57·5	56
	3	0·12	105	144·5	58·5
C(6)	1	0·22	103·5	37	56
	2	0·23	44	103	49
	3	0·09	130	124	58
O(1)	1	0·24	136·5	126·5	69·5
	2	0·26	48·5	116·5	53
	3	0·11	101·5	48	44
O(2)	1	0·22	146·5	67	66·5
	2	0·27	57·5	66·5	42
	3	0·07	97·5	146	57
O(3)	1	0·20	42	111·5	56
	2	0·25	121	81	32
	3	0·11	62·5	32	74·5
O(4)	1	0·22	124·5	140·5	73·5
	2	0·30	45	106	49
	3	0·06	115	55	45·5
O(5)	1	0·22	32·5	98·5	59
	2	0·16	110·5	50·5	46·5
	3	0·12	112·5	147	67
O(6)	1	0·16	88·5	131	41
	2	0·27	159	73·5	77
	3	0·14	69	37·5	60

The results, together with the standard deviations, are given in Table 5.

It is profitable to compare these results with two relevant related structures. One is α-D-glucose determined by McDonald & Beevers (1952) and the other is cellobiose determined by Jacobson, Wunderlich & Lipscomb (1961). Cellobiose has two β-D-glucose residues linked 1 to 4.

Table 7. *Mean values of $\mu(r)$*

Atom	Mean $\mu(r)$	Atom	Mean $\mu(r)$
C(1)	0·18 Å	O(1)	0·20 Å
C(2)	0·17	O(2)	0·19
C(3)	0·16	O(3)	0·19
C(4)	0·16	O(4)	0·19
C(5)	0·17	O(5)	0·17
C(6)	0·18	O(6)	0·19

Table 8. *Observed and calculated structure factors*

h	k	l	F_c	F_o	cosα	sinα
0	2	0	5.2	6.3	+1.0000	0.0000
0	4	0	93.5	91.5	+1.0000	0.0000
0	8	0	7.0	8.0	+1.0000	0.0000
0	10	0	8.3	8.9	+1.0000	0.0000
0	12	0	22.6	22.4	+1.0000	0.0000
0	14	0	29.6	30.7	+1.0000	0.0000
0	16	0	2.2	2.4	+1.0000	0.0000
0	1	1	5.4	5.3	0.0000	+1.0000
0	2	1	29.6	32.6	−1.0000	0.0000
0	3	1	15.3	17.8	0.0000	−1.0000
0	4	1	40.7	42.3	+1.0000	0.0000
0	5	1	3.6	3.7	0.0000	−1.0000
0	6	1	3.2	3.7	−1.0000	0.0000
0	7	1	2.5	3.5	0.0000	+1.0000
0	8	1	13.8	15.5	+1.0000	0.0000
0	11	1	12.7	12.9	0.0000	+1.0000
0	12	1	4.4	3.9	+1.0000	0.0000
0	13	1	4.9	5.1	0.0000	+1.0000
0	14	1	6.2	6.6	−1.0000	0.0000
0	16	1	2.6	1.6	−1.0000	0.0000
0	0	2	44.6	50.8	+1.0000	0.0000
0	1	2	28.4	32.6	0.0000	+1.0000
0	2	2	1.4	0.5	−1.0000	0.0000
0	3	2	18.9	19.6	0.0000	−1.0000
0	4	2	7.1	9.0	+1.0000	0.0000
0	5	2	15.7	16.9	0.0000	+1.0000
0	6	2	0.5	2.0	+1.0000	0.0000
0	7	2	14.9	16.7	0.0000	−1.0000
0	8	2	5.9	7.1	+1.0000	0.0000
0	9	2	2.5	1.6	0.0000	+1.0000
0	10	2	3.7	1.6	−1.0000	0.0000
0	11	2	1.0	1.6	0.0000	+1.0000
0	12	2	10.8	10.9	+1.0000	0.0000
0	13	2	6.3	5.6	0.0000	+1.0000
0	14	2	2.0	1.6	−1.0000	0.0000
0	15	2	6.9	7.7	0.0000	+1.0000
0	1	3	11.6	13.8	0.0000	+1.0000
0	2	3	13.8	14.1	−1.0000	0.0000
0	3	3	6.6	6.9	0.0000	+1.0000
0	4	3	2.4	0.7	+1.0000	0.0000
0	5	3	39.2	37.3	0.0000	+1.0000
0	6	3	18.2	19.1	+1.0000	0.0000
0	7	3	18.8	20.1	0.0000	−1.0000
0	8	3	1.1	0.9	+1.0000	0.0000
0	9	3	7.2	7.7	0.0000	−1.0000
0	10	3	29.5	27.8	+1.0000	0.0000
0	11	3	6.9	5.9	0.0000	−1.0000
0	13	3	9.6	9.8	0.0000	−1.0000
0	14	3	8.4	7.8	+1.0000	0.0000
0	0	4	21.5	25.4	−1.0000	0.0000
0	1	4	2.2	2.8	0.0000	−1.0000
0	2	4	7.1	8.2	−1.0000	0.0000
0	3	4	3.7	5.5	0.0000	−1.0000
0	4	4	8.2	8.9	+1.0000	0.0000
0	5	4	8.8	10.2	0.0000	+1.0000
0	6	4	5.1	6.1	+1.0000	0.0000
0	7	4	20.8	19.1	0.0000	−1.0000
0	8	4	22.9	21.5	+1.0000	0.0000
0	9	4	19.3	18.7	0.0000	+1.0000
0	11	4	6.6	7.1	0.0000	−1.0000
0	12	4	5.7	7.1	+1.0000	0.0000
0	13	4	2.3	2.0	0.0000	+1.0000
0	14	4	4.4	4.5	−1.0000	0.0000
0	1	5	6.8	6.4	0.0000	−1.0000
0	2	5	7.0	7.9	−1.0000	0.0000
0	3	5	7.5	8.6	0.0000	+1.0000
0	4	5	10.0	10.8	−1.0000	0.0000
0	5	5	7.3	6.6	0.0000	+1.0000
0	7	5	7.7	7.8	0.0000	−1.0000
0	8	5	1.9	2.4	+1.0000	0.0000
0	9	5	3.3	2.8	0.0000	+1.0000
0	10	5	7.3	7.8	+1.0000	0.0000
0	11	5	8.9	8.2	0.0000	−1.0000
0	12	5	4.2	4.5	+1.0000	0.0000
0	13	5	3.9	3.7	0.0000	+1.0000
0	0	6	17.1	21.0	−1.0000	0.0000
0	1	6	5.0	5.3	0.0000	+1.0000
0	2	6	7.5	8.6	−1.0000	0.0000
0	3	6	23.6	26.5	0.0000	+1.0000
0	4	6	13.7	14.9	−1.0000	0.0000
0	5	6	9.3	10.9	0.0000	−1.0000
0	6	6	3.3	2.1	−1.0000	0.0000
0	7	6	7.3	7.1	0.0000	+1.0000
0	8	6	4.0	2.6	−1.0000	0.0000
0	9	6	2.7	1.8	0.0000	+1.0000
0	10	6	10.7	11.4	−1.0000	0.0000
0	11	6	10.4	9.8	0.0000	+1.0000
0	1	7	11.9	13.7	0.0000	+1.0000
0	2	7	0.7	1.6	−1.0000	0.0000
0	3	7	16.9	17.6	0.0000	−1.0000
0	6	7	2.7	1.4	+1.0000	0.0000
0	7	7	3.6	3.2	0.0000	−1.0000
0	8	7	6.4	7.0	−1.0000	0.0000
0	9	7	6.4	5.8	0.0000	+1.0000
0	0	8	0.4	2.6	+1.0000	0.0000
0	1	8	2.9	2.6	0.0000	+1.0000
0	2	8	2.7	3.2	−1.0000	0.0000
0	3	8	3.0	2.6	0.0000	+1.0000
0	5	8	4.5	4.8	0.0000	−1.0000
1	1	0	12.4	13.6	0.0000	−1.0000
1	2	0	58.1	59.6	0.0000	+1.0000
1	3	0	17.7	19.6	0.0000	−1.0000
1	4	0	7.0	9.1	0.0000	−1.0000
1	5	0	6.7	6.7	0.0000	+1.0000
1	6	0	19.5	19.2	0.0000	+1.0000
1	7	0	19.2	23.0	0.0000	+1.0000
1	8	0	1.6	1.2	0.0000	+1.0000
1	9	0	0.8	1.4	0.0000	−1.0000
1	10	0	12.1	13.2	0.0000	+1.0000
1	11	0	3.8	4.1	0.0000	−1.0000
1	12	0	6.7	7.7	0.0000	−1.0000
1	13	0	1.5	1.4	0.0000	+1.0000
1	14	0	26.9	28.7	0.0000	+1.0000
1	15	0	13.6	14.7	0.0000	−1.0000
1	0	1	51.0	52.2	0.0000	+1.0000
1	1	1	20.0	19.8	−0.0512	−1.0000
1	2	1	33.2	28.0	−0.7673	+0.6413
1	3	1	33.0	30.7	+1.0000	−0.0416
1	4	1	27.5	24.0	−0.9261	−0.3773
1	5	1	35.8	30.7	−1.0000	0.0000
1	6	1	17.6	18.7	−0.8159	−0.5782
1	7	1	4.2	2.3	+0.9593	+0.2826
1	8	1	11.6	12.0	−0.5202	−0.8540
1	9	1	14.6	16.0	−0.9962	−0.0871
1	10	1	6.1	6.4	−0.1194	−0.9928
1	11	1	10.4	10.2	+0.4461	+0.8950
1	12	1	11.6	10.5	+0.3939	+0.9192
1	13	1	4.4	3.9	−0.9205	+0.3907
1	14	1	14.4	12.6	−0.6256	+0.7802
1	16	1	2.8	2.7	−0.2264	+0.9740
1	0	2	47.8	49.4	+1.0000	0.0000
1	1	2	19.4	18.4	+0.9747	−0.2235
1	2	2	23.6	23.4	+0.4227	+0.9063
1	3	2	16.8	16.4	+0.9886	+0.1502
1	4	2	42.9	44.1	+0.9205	+0.3906
1	5	2	17.3	16.9	+0.7199	−0.6941
1	6	2	6.3	7.1	+0.5392	−0.8422
1	7	2	11.1	11.0	+0.9857	−0.1684
1	8	2	9.7	8.8	+0.4765	+0.8792
1	9	2	11.7	11.8	−0.8531	−0.5217
1	10	2	14.8	13.5	+0.6197	−0.7849
1	12	2	6.9	6.3	−0.8972	−0.4417
1	13	2	9.5	8.7	−0.9994	−0.0344
1	14	2	5.6	5.3	+0.8616	+0.5076
1	0	3	43.4	55.8	0.0000	−1.0000
1	1	3	17.6	21.0	−0.9378	+0.3472
1	2	3	20.6	22.4	+0.7313	+0.6820
1	3	3	10.6	9.6	−0.8563	−0.5166
1	4	3	60.7	52.7	+0.2011	−0.9796
1	5	3	3.5	5.8	−0.6163	+0.7875
1	6	3	18.7	18.1	−0.2569	−0.9664
1	7	3	23.6	21.9	−0.9958	+0.0921
1	8	3	30.4	26.9	+0.4010	−0.9196
1	9	3	11.8	11.3	+0.9807	−0.1957
1	10	3	24.1	18.8	−0.4803	−0.8771
1	11	3	10.8	8.8	−0.6638	+0.7480
1	12	3	7.9	6.8	−0.1432	+0.9897
1	13	3	4.4	3.5	−0.8376	−0.5462
1	14	3	4.7	3.9	−0.2541	−0.9672
1	15	3	1.3	1.6	+0.1339	+0.9911
1	0	4	15.6	18.3	+1.0000	0.0000
1	1	4	11.7	15.4	−0.8313	+0.5559
1	2	4	8.2	11.9	+0.5724	+0.8200
1	3	4	8.2	9.9	+0.7199	−0.6941
1	4	4	15.6	15.8	+0.7623	−0.6472
1	5	4	20.5	18.6	−0.9998	0.0000
1	6	4	17.0	15.4	+0.9157	+0.4019
1	7	4	24.4	20.1	+0.5405	−0.8413
1	8	4	12.0	10.9	+0.7975	−0.6033
1	9	4	5.7	4.1	+0.7043	+0.7099
1	10	4	4.9	2.9	+0.9034	+0.4289
1	11	4	5.9	5.4	+0.2419	−0.9703
1	12	4	3.6	2.5	+0.9360	−0.3520
1	13	4	6.3	5.6	+0.9060	+0.4233
1	14	4	2.0	1.7	−0.3178	−0.9481
1	0	5	3.2	3.0	0.0000	−1.0000
1	1	5	4.0	4.7	+0.9981	−0.0613
1	2	5	7.0	8.4	+0.5833	−0.8123
1	3	5	5.0	6.4	−0.1518	+0.9884
1	4	5	9.9	10.9	+0.8111	−0.5849
1	5	5	5.0	5.0	+0.7818	−0.6235
1	6	5	3.5	2.9	+0.8638	+0.5037
1	7	5	6.1	5.4	−0.7378	+0.6750
1	8	5	5.1	4.5	+0.7888	−0.6147
1	9	5	4.7	4.8	+0.9409	+0.3387
1	10	5	2.8	2.5	+0.2503	+0.9682
1	11	5	5.0	5.0	+0.6146	−0.7889
1	13	5	1.5	1.6	+0.9456	+0.3253
1	0	6	4.5	3.6	+1.0000	0.0000
1	1	6	2.9	3.5	+0.8895	−0.4568
1	2	6	4.5	4.4	+0.0466	−0.9989
1	3	6	14.4	15.2	−0.8375	−0.5465
1	4	6	10.5	10.2	+0.2219	−0.9751
1	5	6	13.2	10.9	−0.9254	+0.3791
1	6	6	7.6	7.2	+0.5256	−0.8123
1	8	6	5.3	3.4	+0.9733	+0.2297
1	9	6	7.1	5.8	+0.9406	+0.3395
1	10	6	2.6	1.6	−0.3978	−0.9174
1	0	7	7.5	7.9	0.0000	+1.0000
1	1	7	4.5	4.4	−0.1649	−0.9863
1	2	7	9.8	9.2	−0.3835	−0.9236
1	3	7	8.9	8.0	−0.9923	−0.1236
1	4	7	3.1	2.5	+0.9849	−0.1734
1	5	7	4.9	4.8	+0.9038	−0.4281
1	6	7	2.6	1.5	−0.2888	+0.9574
1	7	7	5.1	2.9	−0.8707	−0.4917
1	8	7	3.7	3.6	+0.2604	−0.9655
1	9	7	2.5	2.0	+0.4288	+0.9035
1	0	8	4.6	5.0	−1.0000	0.0000
1	1	8	2.9	2.5	−0.8683	−0.4962
1	2	8	4.6	5.6	+0.7339	−0.6793
1	4	8	2.8	2.0	−0.8461	+0.5330
1	5	8	2.9	2.9	−0.8402	+0.5423
2	6	1	21.0	23.0	−0.6575	+0.7535
2	0	0	91.0	83.4	+1.0000	0.0000
2	1	0	67.6	61.2	−1.0000	0.0000
2	2	0	7.5	8.6	+1.0000	0.0000
2	3	0	2.8	2.7	−1.0000	0.0000
2	4	0	18.0	22.1	+1.0000	0.0000
2	5	0	7.8	9.8	−1.0000	0.0000
2	6	0	2.4	3.3	−1.0000	0.0000
2	7	0	18.0	20.1	−1.0000	0.0000
2	8	0	3.5	3.6	−1.0000	0.0000
2	9	0	3.8	5.5	+1.0000	0.0000
2	10	0	8.4	10.2	+1.0000	0.0000
2	11	0	2.2	2.6	+1.0000	0.0000
2	12	0	8.8	8.6	+1.0000	0.0000
2	13	0	5.1	6.5	+1.0000	0.0000
2	14	0	17.0	18.6	+1.0000	0.0000
2	15	0	5.7	5.8	−1.0000	0.0000
2	16	0	0.4	0.6	+1.0000	0.0000
2	0	1	92.5	86.1	0.0000	+1.0000
2	1	1	48.7	44.9	+0.2825	−0.9593
2	2	1	14.6	16.0	−0.7433	+0.6690
2	3	1	54.2	50.7	+0.8129	+0.5824
2	4	1	48.1	46.2	+0.0399	+0.9992
2	5	1	34.6	36.4	−0.9823	−0.1872
2	7	1	4.5	4.9	+0.7454	+0.6666
2	8	1	16.7	18.0	+0.3585	+0.9335
2	9	1	20.5	22.0	−0.8144	−0.5807
2	10	1	14.6	14.8	−0.2103	+0.9776
2	11	1	10.3	10.7	+0.4742	+0.8804
2	12	1	8.9	9.7	+0.1217	+0.9926
2	13	1	4.3	6.4	−0.6268	−0.7792
2	14	1	8.2	7.2	−0.4610	+0.8874
2	15	1	6.7	7.9	+0.9970	−0.0776
2	0	2	6.1	9.7	−1.0000	0.0000
2	1	2	12.7	13.9	+0.7776	+0.6288
2	2	2	39.6	37.8	+0.1536	−0.9881
2	3	2	12.5	12.7	−0.8608	+0.5090
2	4	2	34.9	36.6	−0.9721	−0.2348
2	5	2	10.9	10.7	+0.5874	+0.8093
2	6	2	2.9	1.7	+0.8570	−0.5153
2	7	2	8.2	8.9	−0.3126	−0.9499
2	8	2	16.6	16.8	−0.8723	+0.4889
2	9	2	11.0	10.3	−0.0755	+0.9971
2	10	2	27.9	24.5	−0.2205	+0.9754
2	11	2	1.7	3.3	+0.0969	+0.9954
2	12	2	12.2	11.0	+0.1957	+0.9807
2	13	2	4.3	3.9	−0.9943	+0.1069
2	14	2	9.4	8.7	−0.2410	+0.9705
2	15	2	4.1	4.6	+0.3036	+0.9528
2	0	3	11.8	14.2	0.0000	+1.0000
2	1	3	2.3	2.3	+0.6461	−0.7631
2	2	3	17.1	19.6	−0.7532	+0.6578
2	3	3	19.9	17.0	+0.8654	+0.5011
2	4	3	20.8	19.6	−0.4505	+0.8928
2	5	3	15.8	15.6	−0.9733	+0.2294
2	6	3	16.0	13.8	+0.3665	−0.9304
2	7	3	3.2	3.0	−0.9496	−0.3135
2	8	3	7.5	7.6	−0.6669	+0.7451
2	9	3	11.1	10.4	−0.6989	−0.7152
2	10	3	15.6	12.6	+0.9996	+0.0282
2	11	3	3.4	4.4	−0.9980	+0.0637
2	12	3	2.1	3.1	+0.4188	+0.9080
2	13	3	6.1	5.3	−0.2847	−0.9586
2	14	3	5.8	5.8	+0.5379	+0.8430
2	15	3	2.2	1.8	−0.2648	+0.9643
2	0	4	0.9	2.8	−0.9997	0.0000
2	1	4	5.3	5.8	+0.9482	+0.3178
2	2	4	8.1	9.1	+0.9596	+0.2812
2	3	4	8.4	8.4	+0.8585	−0.5128
2	4	4	5.5	6.2	−0.5693	+0.8221
2	5	4	5.0	6.4	−0.7642	+0.6449
2	6	4	15.1	13.8	+0.9974	+0.0716
2	7	4	21.1	19.0	+0.6042	−0.7968
2	8	4	14.5	12.5	+0.2302	+0.9731
2	9	4	8.1	7.6	−0.1669	+0.9860
2	10	4	4.4	4.4	+0.7842	+0.6205
2	11	4	6.3	5.9	+0.2828	−0.9592
2	12	4	5.0	4.6	+0.7257	+0.6880
2	13	4	2.1	3.1	−0.5057	+0.8627
2	14	4	5.0	4.8	+0.2358	+0.9718
2	0	5	32.4	34.4	0.0000	−1.0000
2	1	5	15.6	15.4	−0.8490	−0.5283
2	3	5	13.4	15.4	−0.7056	+0.7086
2	4	5	13.2	12.8	+0.0752	−0.9972
2	5	5	3.3	3.3	+0.9785	−0.2061
2	7	5	9.5	8.3	+0.9622	+0.2723
2	8	5	3.8	3.3	−0.8680	+0.4965
2	10	5	7.5	6.9	+0.5005	−0.8657
2	12	5	3.3	3.6	+0.5108	+0.8597
2	0	6	15.9	16.4	−1.0000	0.0000
2	1	6	7.4	9.2	−0.6628	+0.7488
2	2	6	12.0	10.4	+0.3534	+0.9355
2	3	6	12.2	13.9	0.0000	+1.0000
2	4	6	10.3	10.4	−0.8064	−0.5913
2	5	6	4.9	3.9	−0.4618	−0.8870
2	6	6	9.8	8.3	+0.4163	+0.9092
2	7	6	2.4	3.1	+0.6055	+0.7959
2	8	6	5.1	4.6	−0.5589	−0.8293
2	11	6	9.3	7.2	+0.7691	+0.6391
2	0	7	3.4	3.3	0.0000	−1.0000
2	1	7	6.4	6.8	−0.9744	+0.2248
2	2	7	11.3	10.5	−0.2198	−0.9755
2	3	7	8.7	8.2	−0.7916	−0.6110
2	4	7	4.9	2.8	+0.5711	−0.8209
2	5	7	3.5	2.5	+0.9624	−0.2716
2	7	7	3.6	3.1	−0.9708	−0.2399
2	8	7	1.7	2.0	−0.7924	−0.6098
2	0	8	4.7	4.9	−1.0000	0.0000
2	1	8	4.0	4.1	−0.7642	−0.6450
2	2	8	4.4	4.5	+0.8911	−0.4538
2	3	8	4.5	5.5	+0.9725	+0.2327
2	5	8	4.8	3.6	+0.1178	−0.9930
3	1	0	28.5	28.7	0.0000	−1.0000
3	2	0	18.6	19.2	0.0000	+1.0000
3	3	0	4.9	5.0	0.0000	−1.0000
3	4	0	2.5	3.4	0.0000	+1.0000
3	5	0	9.2	9.3	0.0000	−1.0000
3	6	0	11.0	14.1	0.0000	+1.0000
3	7	0	19.1	23.7	0.0000	+1.0000
3	8	0	16.2	18.7	0.0000	−1.0000
3	9	0	6.3	6.5	0.0000	−1.0000
3	10	0	7.1	8.0	0.0000	−1.0000
3	12	0	10.1	8.6	0.0000	−1.0000
3	13	0	4.6	4.4	0.0000	−1.0000
3	14	0	2.0	1.2	0.0000	−1.0000
3	15	0	12.0	12.4	0.0000	−1.0000
3	0	1	19.9	19.4	0.0000	+1.0000
3	1	1	25.8	24.7	−0.3591	−0.9333
3	2	1	24.9	22.8	−0.5713	+0.8207
3	3	1	19.3	19.9	+0.8379	+0.5458
3	4	1	49.3	48.0	−0.8282	+0.5605
3	5	1	16.1	17.0	−0.6508	−0.7592
3	6	1	16.0	16.3	−0.5361	+0.8442
3	7	1	7.0	8.5	−0.3946	+0.9189
3	8	1	13.8	13.0	−0.1882	+0.9821
3	9	1	8.9	9.9	+0.9930	−0.1183
3	10	1	16.1	15.9	+0.4899	+0.8718
3	11	1	9.7	9.5	+0.7455	+0.6665
3	12	1	4.4	5.3	+0.8238	+0.5669
3	13	1	5.6	5.6	−0.8256	+0.5643
3	14	1	7.6	6.9	−0.5952	+0.8036
3	15	1	9.5	9.7	+0.9523	−0.3052
3	0	2	57.2	60.6	+1.0000	0.0000
3	1	2	5.3	5.3	−0.9772	−0.2124
3	2	2	17.0	17.3	−0.7686	+0.6397
3	3	2	8.4	7.2	−0.6866	+0.7271
3	4	2	39.2	38.8	+0.9912	−0.1324
3	5	2	37.1	33.6	−0.7434	−0.6689
3	6	2	12.3	13.6	+0.5596	−0.8288
3	7	2	11.1	11.1	+0.2807	+0.9598
3	8	2	8.8	8.1	+0.9564	+0.2920
3	9	2	19.8	18.3	+0.1868	−0.9824
3	10	2	16.0	14.4	+0.5509	−0.8345
3	11	2	10.4	9.1	−0.5613	+0.8276
3	12	2	8.1	7.4	−0.8956	−0.4449
3	13	2	6.5	6.4	+0.6310	−0.7758
3	14	2	5.4	5.5	+0.9699	−0.2437
3	15	2	2.9	4.1	−0.1488	+0.9889
3	0	3	10.4	12.7	0.0000	+1.0000
3	1	3	11.2	12.2	+0.9043	+0.4270
3	2	3	14.9	17.9	+0.6791	+0.7340
3	3	3	8.1	8.7	+0.1174	−0.9931
3	4	3	15.2	16.1	+0.9947	−0.1025
3	5	3	5.6	6.2	−0.9438	+0.3304
3	6	3	5.8	6.9	−0.4129	+0.9108
3	7	3	6.1	6.2	−0.7941	+0.6078
3	8	3	9.7	9.7	+0.9517	−0.3070
3	9	3	8.2	7.1	−0.7949	−0.6067
3	10	3	8.6	7.8	−0.9983	+0.0576
3	11	3	9.2	7.3	−0.1310	+0.9914
3	12	3	4.5	3.9	−0.9745	+0.2243
3	13	3	5.7	5.3	+0.2386	−0.9711
3	0	4	7.0	8.8	+1.0000	0.0000
3	1	4	13.1	14.9	−0.5044	+0.8635
3	2	4	4.5	6.7	−0.0485	+0.9988
3	3	4	11.9	11.4	+0.4058	−0.9140
3	4	4	13.5	13.8	+0.9570	−0.2902
3	5	4	7.4	8.1	−0.9641	+0.2656
3	6	4	18.0	17.6	+0.9772	−0.2121
3	7	4	23.4	19.9	+0.1595	−0.9872
3	8	4	16.7	14.0	+0.7059	+0.7083
3	10	4	4.4	3.6	+0.9994	−0.0352
3	11	4	3.4	3.9	−0.2582	−0.9661
3	12	4	5.1	4.1	+0.3600	+0.9329
3	13	4	3.0	4.1	+0.2129	+0.9771
3	0	5	28.0	28.7	0.0000	+1.0000

Table 8 (*cont.*)

h	k	l	F_c	F_o	cosα	sinα
3	1	5	10.2	10.8	+0.6461	-0.7633
3	2	5	13.0	14.9	+0.6971	+0.7170
3	3	5	11.1	12.8	+0.8690	+0.4947
3	4	5	6.7	5.5	+0.9329	+0.3601
3	5	5	4.9	5.0	+0.3799	-0.9250
3	6	5	13.7	12.6	+0.6653	+0.7466
3	7	5	7.9	7.2	+0.9236	+0.3833
3	8	5	4.0	4.4	-0.9914	+0.1308
3	9	5	2.2	3.0	+0.8170	+0.5765
3	10	5	4.8	4.4	+0.9920	-0.1263
3	11	5	3.4	3.2	+0.9910	-0.1342
3	12	5	13.3	12.2	-0.5377	+0.8431
3	0	6	5.8	7.0	+1.0000	0.0000
3	1	6	3.6	5.7	+0.9703	-0.2418
3	2	6	3.9	4.4	-0.4747	-0.8801
3	3	6	6.0	6.5	-0.4928	-0.8701
3	4	6	7.0	7.1	+0.5749	+0.8182
3	5	6	5.5	6.4	-0.1173	+0.9931
3	6	6	2.8	3.0	+0.9927	-0.1203
3	7	6	2.3	2.8	+0.7951	-0.6067
3	8	6	6.9	6.4	+0.8567	+0.5158
3	9	6	3.0	2.8	-0.1373	+0.9905
3	0	7	1.9	2.8	0.0000	+1.0000
3	1	7	8.9	8.5	-0.6070	-0.7947
3	2	7	3.1	2.8	-0.5684	-0.8228
3	3	7	3.0	3.9	-0.4697	-0.8829
3	4	7	3.4	3.0	+0.4650	-0.8853
3	5	7	3.7	2.4	0.0000	-0.9999
3	7	7	2.8	3.2	-0.6530	-0.7573
3	0	8	5.9	6.3	-1.0000	0.0000
3	1	8	4.9	5.3	+0.6345	-0.7729
3	2	8	7.0	6.4	+0.9247	-0.3807
3	3	8	2.4	2.8	-0.1869	+0.9824
3	4	8	6.3	4.4	-0.9922	+0.1243
4	0	0	45.7	45.4	-1.0000	0.0000
4	1	0	3.7	7.3	-1.0000	0.0000
4	2	0	30.2	20.6	-1.0000	0.0000
4	3	0	6.2	4.1	-1.0000	0.0000
4	4	0	13.3	14.2	-1.0000	0.0000
4	5	0	6.3	7.0	+1.0000	0.0000
4	6	0	8.5	7.2	-1.0000	0.0000
4	7	0	4.8	4.4	-1.0000	0.0000
4	8	0	9.1	10.8	+1.0000	0.0000
4	9	0	14.1	16.0	+1.0000	0.0000
4	10	0	5.2	4.1	-1.0000	0.0000
4	11	0	5.4	5.9	-1.0000	0.0000
4	12	0	4.0	3.3	+1.0000	0.0000
4	13	0	7.5	9.3	+1.0000	0.0000
4	14	0	6.3	7.9	-1.0000	0.0000
4	15	0	4.6	4.5	-1.0000	0.0000
4	0	1	13.1	17.5	0.0000	+1.0000
4	1	1	5.7	8.8	-0.1855	-0.9826
4	2	1	7.3	7.4	-0.4254	+0.9050
4	3	1	16.0	19.1	+0.6434	+0.7656
4	4	1	27.7	28.0	-0.9688	+0.2477
4	5	1	29.1	27.7	-0.7030	+0.7111
4	6	1	12.6	14.5	-0.3937	-0.9193
4	7	1	19.2	20.9	+0.7362	-0.6768
4	8	1	11.2	12.6	-0.7422	-0.6702
4	9	1	17.9	17.8	-0.9483	-0.2857
4	10	1	9.4	9.5	-0.2227	+0.9749
4	11	1	10.4	10.8	+0.7956	-0.6059
4	13	1	4.5	4.8	-0.3779	-0.9258
4	14	1	8.4	8.3	-0.2878	+0.9577
4	15	1	4.2	5.1	+0.7861	-0.6180
4	0	2	7.2	8.5	-1.0000	0.0000
4	1	2	21.8	17.3	+0.8709	-0.4915
4	2	2	27.9	28.0	-0.9718	-0.2357
4	3	2	34.5	32.4	-0.7736	+0.6337
4	4	2	31.6	31.5	-0.6841	-0.7294
4	5	2	19.3	19.8	+0.9495	-0.3136
4	6	2	17.8	16.1	-0.9963	-0.0857
4	7	2	13.7	13.6	-0.8812	+0.4728
4	8	2	16.5	15.6	-0.9789	+0.2044
4	9	2	10.9	9.3	+0.6403	+0.7681
4	10	2	20.7	17.9	-0.7888	+0.6147
4	11	2	3.4	3.3	+0.8509	+0.5254
4	12	2	2.3	2.7	-0.9488	-0.3159
4	13	2	3.6	3.5	-0.6316	-0.7753
4	14	2	9.0	8.5	-0.6655	+0.7464
4	0	3	5.1	5.3	0.0000	+1.0000
4	1	3	8.8	11.1	-0.6634	+0.7483
4	2	3	3.3	3.1	-0.6482	+0.7614
4	3	3	15.5	14.9	+0.1494	-0.9888
4	4	3	14.0	12.2	-0.9986	-0.0523
4	5	3	8.5	10.1	-0.9250	-0.3799
4	7	3	3.7	3.5	-0.3349	-0.9423
4	8	3	9.3	10.4	-0.9952	-0.0973
4	9	3	6.3	6.4	-0.8448	+0.5350
4	11	3	4.6	5.4	-0.5875	+0.8092
4	13	3	3.3	3.3	-0.4822	+0.8760
4	14	3	4.9	4.3	-0.7200	+0.6939
4	0	4	36.0	37.7	+1.0000	0.0000
4	1	4	4.8	5.9	-0.9655	-0.2604
4	2	4	8.3	11.4	+0.3744	-0.9273
4	3	4	4.0	3.5	-0.7434	+0.6689
4	4	4	7.7	8.0	+0.8963	-0.4435
4	5	4	11.4	10.3	+0.0907	-0.9959
4	6	4	6.5	6.4	+0.4370	-0.8994
4	7	4	8.2	8.6	-0.3090	-0.9511
4	8	4	7.8	7.3	-0.5035	+0.8640
4	9	4	8.5	7.6	+0.2562	-0.9666
4	10	4	2.2	3.5	-0.5294	+0.8483
4	11	4	4.3	4.3	+0.7916	-0.6109
4	12	4	5.8	4.9	+0.9388	+0.3445
4	13	4	3.1	2.1	+0.5320	-0.8467
4	0	5	28.0	31.6	0.0000	-1.0000
4	1	5	15.2	14.3	-0.8409	+0.5412
4	2	5	15.1	16.6	-0.4951	+0.8688
4	3	5	7.7	7.6	+0.4086	-0.9127
4	4	5	15.4	15.5	+0.5407	-0.8412
4	5	5	4.2	3.7	-0.5397	+0.8419
4	6	5	9.2	9.2	-0.0330	+0.9995
4	7	5	4.7	4.3	+0.9860	+0.1664
4	8	5	6.0	5.9	-0.9998	-0.0213
4	9	5	3.9	3.3	+0.3708	+0.9287
4	11	5	5.9	6.4	0.000	+1.0000
4	12	5	2.4	2.1	-0.9166	-0.3999
4	0	6	2.8	3.4	+1.0000	0.0000
4	1	6	3.6	4.9	+0.6687	+0.7435
4	2	6	3.7	4.1	-0.2253	+0.9743
4	4	6	8.1	8.6	+0.8545	-0.5194
4	5	6	2.3	3.5	-0.8704	+0.4923
4	6	6	5.8	5.7	+0.1772	+0.9842
4	7	6	4.7	4.0	0.0000	-1.0000
4	8	6	5.4	4.6	+0.6947	-0.7193
4	9	6	1.5	2.9	-0.7627	+0.6468
4	10	6	3.7	2.7	+0.8698	+0.4933
4	0	7	4.9	6.3	0.0000	-1.0000
4	1	7	4.6	5.0	-0.9985	+0.0543
4	2	7	5.1	4.3	-0.9888	+0.1492
4	3	7	2.3	2.9	-0.9819	+0.1890
4	4	7	3.8	3.3	+0.9800	+0.1988
4	5	7	6.1	5.4	+0.7359	+0.6771
4	6	7	3.2	3.0	-0.2439	+0.9698
4	7	7	2.6	2.7	+0.1776	-0.9841
4	1	8	7.8	6.8	+0.4090	-0.9125
4	2	8	2.3	2.1	+0.6165	+0.7873
5	2	4	9.7	10.3	-0.9978	-0.0666
5	0	5	16.5	13.2	0.0000	+1.0000
5	2	0	16.5	14.9	0.0000	+1.0000
5	4	0	5.3	6.4	0.0000	-1.0000
5	5	0	7.1	8.3	0.0000	-1.0000
5	6	0	4.8	4.7	0.0000	+1.0000
5	7	0	14.9	16.1	0.0000	-1.0000
5	10	0	6.0	5.9	0.0000	-1.0000
5	11	0	2.8	2.6	0.0000	-1.0000
5	12	0	4.5	2.9	0.0000	-1.0000
5	13	0	3.2	3.4	0.0000	+1.0000
5	14	0	2.3	1.4	0.0000	-1.0000
5	0	1	6.2	7.5	0.0000	+1.0000
5	1	1	13.7	14.4	+0.4135	-0.9105
5	2	1	5.4	7.4	+0.3678	+0.9299
5	3	1	17.4	18.1	-0.0554	+0.9985
5	4	1	14.4	16.8	-0.4519	+0.8921
5	5	1	19.1	19.0	+0.8257	-0.5641
5	6	1	22.5	24.9	-0.1408	+0.9900
5	7	1	11.9	13.7	-0.6840	+0.7294
5	8	1	23.4	25.3	+0.8033	+0.5956
5	9	1	17.7	18.5	+0.9937	-0.1117
5	10	1	12.2	12.3	+0.1500	+0.9887
5	11	1	7.4	8.2	-0.0694	-0.9976
5	12	1	6.9	6.5	+0.9067	+0.4217
5	13	1	4.6	4.3	+0.9141	+0.4055
5	0	2	0.2	1.4	-0.9885	-0.0100
5	1	2	15.1	11.1	-0.9782	+0.2075
5	2	2	33.8	28.9	-0.9936	+0.1127
5	3	2	9.7	7.3	-0.9965	+0.0833
5	4	2	13.0	12.4	-0.0858	-0.9963
5	5	2	17.4	16.0	-0.9246	-0.3809
5	6	2	7.5	7.1	+0.9708	+0.2399
5	7	2	9.3	10.3	-0.4817	+0.8763
5	8	2	8.1	6.8	+0.7283	-0.6852
5	9	2	12.8	12.9	+0.7316	-0.6818
5	10	2	3.4	1.7	-0.9908	-0.1354
5	11	2	8.4	7.9	+0.0635	+0.9980
5	12	2	6.2	5.6	-0.1810	-0.9835
5	13	2	8.0	8.5	+0.9325	+0.3610
5	14	2	3.5	3.2	-0.9830	+0.1836
5	0	3	17.1	16.9	0.0000	+1.0000
5	1	3	5.9	5.4	+0.8563	-0.5165
5	2	3	13.1	13.3	-0.6111	-0.7915
5	3	3	8.5	8.6	+0.9862	-0.1658
5	4	3	11.2	15.9	+0.0613	+0.9981
5	0	4	13.9	12.7	-1.0000	0.0000
5	1	4	11.2	11.7	+0.7065	+0.7078
5	3	4	8.4	7.9	-0.3382	-0.9411
5	4	4	6.3	8.0	-0.7902	+0.6129
5	1	5	14.2	12.7	+0.1743	-0.9847
5	2	5	11.8	11.4	+0.3984	+0.9172
5	3	5	7.8	9.4	+0.8936	-0.4489
5	4	5	4.7	5.7	-0.6241	+0.7813
5	1	6	6.9	7.5	+0.9522	+0.3053
5	2	6	4.5	5.0	-0.8779	+0.4788
5	3	6	4.6	4.9	+0.5682	+0.8229
5	4	6	5.5	7.3	+0.6075	+0.7943
5	1	7	5.6	4.9	-0.9319	-0.3628
5	2	7	4.7	3.1	+0.7469	+0.6650
5	3	7	5.0	4.7	-0.7072	-0.7070
5	4	7	6.3	7.8	+0.3539	-0.9353
6	0	0	23.6	23.7	-1.0000	0.0000
6	1	0	0.8	2.1	+0.9996	0.0000
6	2	0	13.3	10.1	+1.0000	0.0000
6	3	0	9.9	9.6	+1.0000	0.0000
6	4	0	12.4	11.7	-1.0000	0.0000
6	5	0	2.7	2.4	+1.0000	0.0000
6	6	0	23.6	26.7	+1.0000	0.0000
6	7	0	11.5	10.5	+1.0000	0.0000
6	8	0	8.1	9.6	+1.0000	0.0000
6	10	0	4.6	4.7	-1.0000	0.0000
6	11	0	4.2	2.7	+1.0000	0.0000
6	12	0	8.3	12.2	+1.0000	0.0000
6	13	0	4.0	4.0	-1.0000	0.0000
6	14	0	7.6	8.2	-1.0000	0.0000
6	0	1	1.4	2.3	0.0000	-1.0000
6	1	1	9.4	8.6	-0.9640	+0.2658
6	2	1	10.7	10.1	-0.0836	-0.9965
6	3	1	6.0	5.1	-0.4426	+0.8967
6	4	1	4.3	5.5	-0.6543	-0.7563
6	5	1	9.6	11.6	+0.2489	+0.9685
6	6	1	33.3	32.1	+0.1817	-0.9833
6	7	1	12.3	14.4	+0.9921	-0.1258
6	8	1	14.3	14.8	-0.8276	-0.5613
6	10	1	10.0	10.1	+0.2664	-0.9639
6	11	1	7.0	7.0	+0.8560	-0.5169
6	0	2	6.2	3.9	-1.0000	0.0000
6	1	2	4.4	2.3	-0.8942	-0.4477
6	2	2	12.9	11.3	-0.6513	+0.7588
6	3	2	6.5	6.8	-0.9967	+0.0807
6	4	2	14.7	12.7	-0.5258	-0.8506
6	5	2	5.3	4.2	-0.2617	-0.9651
6	6	2	6.8	5.4	-0.5708	+0.8211
6	7	2	2.8	3.5	-0.1677	+0.9858
6	8	2	7.9	6.8	-0.7883	-0.6153
6	9	2	5.0	5.4	+0.0686	-0.9976
6	10	2	6.0	5.0	-0.9749	-0.2226
6	11	2	9.1	9.6	+0.9958	+0.0916
6	12	2	10.5	10.4	-0.4946	-0.8691
6	13	2	5.0	4.8	-0.8086	+0.5883
6	0	3	5.2	3.5	0.0000	+1.0000
6	1	3	12.9	13.7	-0.6495	-0.7604
6	2	3	14.8	14.3	-0.0506	-0.9987
6	3	3	6.5	6.6	-0.8947	-0.4467
6	4	3	10.1	13.8	+0.8085	+0.5885
6	0	4	11.2	13.9	+1.0000	0.0000
6	1	4	21.8	20.6	-0.9024	-0.4310
6	2	4	9.3	9.6	+0.8245	-0.5658
6	3	4	5.4	5.5	+0.7711	+0.6368
6	4	4	11.5	13.4	+0.3543	-0.9351
6	3	5	11.5	13.3	+0.8230	-0.5680
6	0	6	5.6	6.8	+1.0000	0.0000
6	2	6	5.1	3.6	-0.8260	-0.5636
6	3	6	7.9	7.3	+0.9888	+0.1495
6	4	6	3.9	5.7	-0.1937	-0.9811
6	0	7	4.5	3.3	0.0000	+1.0000
6	2	7	4.4	3.1	-0.9726	+0.2325
6	3	7	6.6	5.3	+0.6244	+0.7811
6	4	7	7.2	7.3	+0.1107	+0.9939
7	1	0	19.9	17.4	0.0000	+1.0000
7	3	0	3.4	3.8	0.0000	-1.0000
7	4	0	4.2	6.7	0.0000	-1.0000
7	6	0	11.2	11.0	0.0000	-1.0000
7	7	0	10.3	11.5	0.0000	-1.0000
7	8	0	23.4	24.7	0.0000	+1.0000
7	9	0	6.4	6.5	0.0000	-1.0000
7	12	0	13.6	18.0	0.0000	+1.0000
7	13	0	8.5	9.3	0.0000	+1.0000
7	1	1	11.5	10.8	+0.7507	-0.6607
7	3	1	8.8	8.6	+0.3078	+0.9515
7	4	1	4.6	6.3	+0.9420	-0.3356
7	5	1	6.0	7.3	+0.9505	+0.3108
7	6	1	11.1	13.1	-0.0732	+0.9973
7	8	1	9.6	10.1	+0.9684	+0.2493
7	12	1	5.2	5.1	+0.1609	+0.9870
7	0	2	18.1	16.4	-1.0000	0.0000
7	1	2	8.8	7.3	-0.4620	+0.8669
7	2	2	11.4	9.6	-0.9993	+0.0367
7	3	2	2.6	2.5	+0.4125	+0.9110
7	4	2	5.1	5.4	-0.7547	-0.6561
7	5	2	5.5	4.6	+0.9000	+0.4359
7	7	2	4.3	4.8	-0.2205	+0.9754
7	8	2	5.9	4.0	+0.9815	+0.2491
7	9	2	3.1	3.5	+0.9967	-0.0815
7	10	2	4.1	3.5	-0.9904	+0.1383
7	11	2	6.6	7.1	+0.4605	+0.8877
7	1	3	13.4	14.2	-0.8227	-0.5685
7	2	3	22.7	18.6	-0.1005	-0.9949
7	4	3	12.4	12.2	-0.9984	-0.0573
7	0	4	5.5	3.9	-1.0000	0.0000
7	1	4	10.4	8.8	+0.9636	-0.2674
7	2	4	8.7	6.8	-0.9956	-0.0937
7	3	4	4.7	5.5	-0.9763	+0.2165
7	4	4	11.0	12.6	-0.5821	+0.8131
7	3	5	6.2	4.9	+0.1690	-0.9856
7	4	5	6.5	7.3	-0.9172	+0.3985
7	0	6	5.0	3.7	+1.0000	0.0000
7	2	6	5.3	4.3	-0.8912	+0.4535
7	3	6	5.2	5.7	+0.9896	+0.1439
8	4	3	14.8	15.0	+0.9980	-0.0637
8	11	1	5.1	4.7	+0.0511	+0.9987
8	0	0	5.5	3.3	+1.0000	0.0000
8	2	0	21.5	20.9	+1.0000	0.0000
8	3	0	12.5	8.9	-1.0000	0.0000
8	5	0	8.8	7.1	+1.0000	0.0000
8	6	0	21.3	23.0	+1.0000	0.0000
8	7	0	8.0	7.5	-1.0000	0.0000
8	8	0	3.5	3.6	+1.0000	0.0000
8	10	0	3.9	3.6	+1.0000	0.0000
8	11	0	6.7	9.5	+1.0000	0.0000
8	12	0	4.4	3.1	+1.0000	0.0000
8	0	1	7.8	5.7	0.0000	-1.0000
8	2	1	9.0	7.8	+0.5611	+0.8277
8	3	1	7.8	9.4	+0.4893	-0.8721
8	4	1	11.1	10.1	+0.6365	-0.7713
8	5	1	7.1	7.8	+0.9938	+0.1111
8	6	1	6.7	6.9	+0.9392	-0.3434
8	8	1	7.6	7.5	-0.6148	-0.7887
8	9	1	9.0	10.0	+0.9730	+0.2306
8	1	2	5.7	4.4	+0.3075	+0.9516
8	2	2	3.6	4.0	-0.7768	+0.6298
8	3	2	8.9	5.6	-0.9993	+0.0370
8	4	2	2.0	2.5	+0.4728	+0.8811
8	5	2	2.2	1.6	+0.9916	-0.1290
8	6	2	6.6	6.2	+0.7905	+0.6124
8	7	2	6.2	3.8	-0.7932	-0.6089
8	8	2	3.8	4.2	+0.1065	-0.9943
8	9	2	3.9	1.9	+0.7936	-0.6085
8	0	3	9.1	5.3	0.0000	-1.0000
8	1	3	8.3	9.2	-0.5019	-0.8649
8	2	3	8.4	5.3	+0.2195	+0.9756
8	1	4	7.0	6.7	+0.2609	-0.9654
8	2	4	4.5	4.0	+0.9068	+0.4216
8	4	4	7.2	7.3	-0.1276	-0.9918
8	2	6	3.1	3.0	-0.9276	-0.3737
8	4	6	5.3	3.0	-0.6375	-0.7704
9	1	0	9.1	8.3	0.0000	+1.0000
9	2	0	10.3	6.6	0.0000	-1.0000
9	3	0	4.9	5.3	0.0000	-1.0000
9	6	0	6.2	6.2	0.0000	-1.0000
9	7	0	0.7	2.1	0.0000	+1.0000
9	8	0	9.1	14.2	0.0000	+1.0000
9	9	0	7.7	11.5	0.0000	-1.0000
9	10	0	6.6	5.0	0.0000	+1.0000
9	1	1	3.4	4.3	-0.6176	-0.7865
9	2	1	6.8	5.9	-0.0837	+0.9965
9	7	1	6.1	6.3	-0.9478	-0.3188
9	9	1	2.9	3.4	+0.6910	+0.7229
9	1	2	10.0	8.1	+0.7648	+0.6443
9	2	2	8.5	6.6	+0.9939	+0.1099
9	3	2	3.5	2.3	+0.9978	+0.0669
9	4	2	5.4	4.8	+0.0997	-0.9950
9	5	2	4.5	3.7	+0.7008	+0.7134
9	6	2	2.8	2.5	-0.1038	+0.9946
9	7	2	2.1	1.9	+0.8966	-0.4428
9	9	2	3.5	3.4	-0.9962	+0.0871
9	0	3	5.8	2.9	0.0000	-1.0000
9	1	3	10.0	8.2	-0.7499	-0.6616
9	2	3	12.0	9.6	+0.2932	-0.9560
9	4	3	7.6	7.1	-0.9600	-0.2799
9	1	4	6.8	5.9	+0.7489	-0.6627
9	2	4	5.1	4.3	-0.1139	+0.9935
9	2	5	4.0	4.8	0.0000	-1.0000
10	1	0	7.1	5.8	-1.0000	0.0000
10	2	0	4.1	5.5	+1.0000	0.0000
10	3	0	5.1	4.7	-1.0000	0.0000
10	4	0	7.8	5.0	+1.0000	0.0000
10	5	0	5.3	2.6	-1.0000	0.0000
10	6	0	2.1	2.7	+1.0000	0.0000
10	7	0	1.3	1.7	-1.0000	0.0000
10	8	0	5.1	4.1	+1.0000	0.0000
10	3	1	11.6	10.4	+0.1404	-0.9901
10	4	1	6.8	6.2	+0.1102	+0.9939
10	5	1	4.7	5.1	+0.8406	+0.5427
10	8	1	5.8	6.2	-0.0151	+1.0000
10	1	2	8.3	5.4	-0.9917	+0.1289
10	2	2	8.5	6.0	-1.0000	-0.0143
10	3	2	6.6	5.6	+0.6938	+0.07202
10	4	2	2.6	2.3	+0.8877	+0.4604
10	5	2	4.4	3.7	-0.7127	+0.7015
10	2	3	4.8	4.0	+0.4378	+0.8991
10	4	3	6.3	4.8	+0.9031	+0.1294
11	1	0	4.4	3.4	0.0000	-1.0000
11	2	0	5.0	2.7	0.0000	-1.0000
11	3	0	2.1	1.7	0.0000	-1.0000
11	4	0	2.0	2.4	0.0000	+1.0000
11	5	0	2.8	3.2	0.0000	-1.0000
11	1	1	7.0	7.1	-0.8507	+0.5256
11	2	1	4.5	6.3	+0.4998	+0.8662
11	3	1	6.1	5.3	+0.1163	-0.9932
11	4	1	4.4	4.9	-0.8004	+0.5995
11	0	2	8.7	7.1	+1.0000	0.0000
11	1	2	5.5	5.4	+0.8855	+0.4646
11	2	2	5.6	5.7	+0.9112	-0.4119
11	3	2	4.0	4.0	+0.2884	-0.9575
11	4	2	2.3	2.6	-0.3406	-0.9403

The CC bonds in β-D-glucose agree well with each other and with the accepted value, the mean being 1·53 Å and the maximum deviation 0·015 Å. The mean CC bond in α-D-glucose is 1·54 Å and in the two residues of cellobiose is 1·53 Å and 1·51 Å. The CO bonds in β-D-glucose give a mean of 1·44 Å which compares with the accepted value of 1·43 Å. However, C(1)O(1) differs from the mean by approximately three times its standard deviation, and is therefore probably significantly short. This is extremely interesting in comparison with α-D-glucose where McDonald & Beevers found two short bonds: C(6)O(6) (not significantly different from the mean in β-D-glucose) and C(1)O(1); McDonald (1950) speculates on the possible chemical significance of these short bonds. The results from β-D-glucose indicate that shortening of the C(1)O(1) bond may well be a real effect. The quoted bond lengths of cellobiose do not show great consistency and both C(1)O(1) bonds, although short, are not the shortest quoted for their residue.

The bond angles in β-glucose are all very close to the tetrahedral angle with two exceptions.

The first is the ring oxygen bond angle C(5)–O(5)–C(1) which is calculated as 113·1°. This compares with 117·3° and 115·2° in the two glucose residues of cellobiose and with 111° in α-D-glucose. The second is C(4)–C(5)–C(6) which is calculated as 115·5°. It is interesting to note that the two glucose residues in cellobiose have only one angle (apart from the ring oxygen angle) which appears significantly greater than the tetrahedral angle in both residues. This is C(4)–C(5)–C(6) which has the values 115·3° and 115·4°. The corresponding angle in α-D-glucose, however, is 112° and it does not appear to be significantly large.

(b) *Temperature anisotropy*

The final B_{ij} values, which are given in Table 4, were used to compute the principal axes of the ellipsoids of thermal vibration for the carbon and oxygen atoms; the method used was that due to Busing & Levy (1958). The results are shown in Table 6. $\mu(r)$ are the r.m.s. displacements along the rth principal axis. Strictly according to the figures these are correct to $\pm0{\cdot}01$ Å. $\theta(r)$ are the angles made by the rth principal axis with the ith direct-lattice vector. These angles are quoted for convenience to the nearest half degree, but are certainly not reliable to better than ten degrees.

These results indicate that β-D-glucose has considerable thermal anisotropy. Alternatively it may well be that these parameters have only proved useful as a method of reducing the overall reliability to a low figure. The mean of the r.m.s. displacements of each atom gives a measure of the temperature effect considered as isotropic for each atom. These means are given in Table 7 and show considerable consistency. All ring atoms including O(5) show, as would be expected, consistently less thermal vibration than oxygen attachments.

(c) *Intermolecular bonding*

Four pairs of atoms have separation values which correspond to hydrogen bonds. Three of these O(5)–O(3′) (2·77 Å), O(6)–O(2′) (2·71 Å) and O(2′)–O(3″) (2·71 Å) are shown in Fig. 1(*c*). Still referring to this figure, the only other possible bonds are between oxygen atoms of one molecule and the oxygen atoms of the identically oriented molecule shifted one cell length along *c*. O(6)–O(1) (2·69 Å) is one such bond and corresponds to the fourth hydrogen bond. It is not drawn in Fig. 1(*c*) but can be seen in Fig. 2(*b*).

The O(1) hydrogen is approximately 1 Å along O(1)O(6) (O(1) to O(6) of identical molecule, one cell length along *c*). The O(6) hydrogen is approximately 1 Å along O(6)O(2′). The O(2) hydrogen lies approximately 1 Å along *e.g.* O(2′)O(3″) and O(3) hydrogen lies approximately 1 Å along *e.g.* O(3′)O(5). The O(2) hydrogen has no hydrogen bond and its position, as given in Table 3, had to be estimated. The structure is therefore not fully bonded and the details and significance of this are discussed elsewhere (Ferrier, 1960).

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