

bonding distances are observed in polysulfide ions and in the sulfur rings of elemental sulfur. There is apparently a weak interaction between these nonbonded sulfurs.

A recent X-ray study (Colapietro, Vaciago, Bradley, Hursthouse & Rendall, 1970) has shown that $Ti(dtc)_4$, $V(dtc)_4$, and $Zr(dtc)_4$ all have eight coordinate dodecahedral structures in contrast to the six coordinate $Sn(dtc)_4$.

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The Crystal and Molecular Structure of the 1:1 Hydrogen Bond Complex Between α -D-Glucose and Urea

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The crystal structure of the 1:1 α -D-glucose-urea complex has been determined by the application of the tangent formula. The space group is $P2_12_1$ with four molecules in a unit cell of dimensions $a = 6.913$, $b = 9.152$ and $c = 16.509$ (all $\sigma = 0.003$) Å. The α -D-glucose and urea molecules are associated by a three-dimensional system of hydrogen bonds which involves all oxygen and nitrogen atoms. The molecular parameters found for urea and glucose in the complex are the same as those for the pure compounds, except for the conformation of the primary alcohol group of glucose.

Introduction

The possibility for donor-acceptor action enables the carbohydrates to form a variety of complexes. In general, these compounds fall into two categories; those involving metals such as alkali and alkaline earth salts

(Rendleman, 1966) and those involving simple organic bases such as pyridine (Strain, 1934, 1937), urea (Quehl, 1938) and various amides (Rendleman, Baker & Hodge, 1967). There have been a number of crystal structure investigations of complexes which fall into the first category, e.g. sucrose. NaBr. 2H₂O (Beevers & Cochran, 1947) and di-fructose. SrCl₂. 3H₂O (Eiland & Pepinsky, 1950). Glucose-urea and glucitol-pyridine (Kim, Jeffrey & Rosenstein, 1970) are the only ex-

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amples of the carbohydrate-organic base addition compounds whose crystal structures have been determined.

The 1:1 addition compound between α -D-glucose and urea was first reported by Quehl (1938). Goodman (1958) characterized the compound as a physical mixture of glucose and urea in the form of a 'molecular compound'. Hatt & Triffett (1963), who reported the space group and crystal data, concluded that the glucose and urea are not covalently bonded, but are held together principally by hydrogen bonding, a hypothesis which is confirmed by this investigation.

Experimental

A crystal $0.36 \times 0.27 \times 0.29$ mm was grown from concentrated aqueous solution and washed with γ -butyro-

lactone. The cell dimensions, obtained by measuring the 2θ values of the axial reflections using Mo $K\beta$ radiation on a four-circle diffractometer and taking a weighted average (using $\sin \theta$ as the weighting function), are $a = 6.193(3)$, $b = 9.152(3)$ and $c = 16.509(3)$ Å. The number in parenthesis here and in the rest of the text is the standard deviation in the last significant digit. The space group is $P2_12_12_1$ as reported by Hatt & Triffett (1963), with $Z = 4$, $D_m = 1.525$ g.cm $^{-3}$ and $D_c = 1.526$ g.cm $^{-3}$. Reflections were collected up to a $\sin \theta/\lambda$ limit of 0.61 with Mo $K\alpha$ radiation, using an unequal interval nine point scan technique (McGandy & Snyder, 1968a). Reflections below $\sin \theta/\lambda$ of 0.36 were counted with balanced filters at each of the nine points. The intervals between points were adjusted so that five of the nine points were taken near the top of

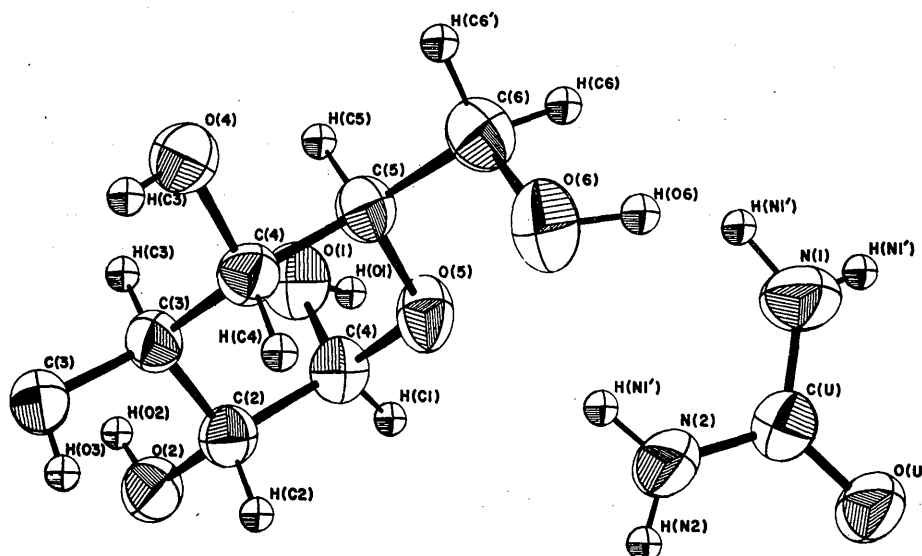


Fig. 1. Perspective drawing of the glucose and urea molecules showing the numbering of the atoms and the thermal ellipsoids. Errata: The hydrogen atom attached to O(4) should be labeled H(O4), not H(C3); the carbon atom to which H(C1) is attached should be labeled C(1), not C(4).

Table 1. Fractional atomic coordinates and thermal parameters in the glucose-urea complex

Key to atomic numbering is given in Fig. 1. The temperature factor expression used was $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$. Numbers in parentheses refer to standard deviations of the last place.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(C1)	0.6209(4)	0.3528(3)	0.1909(2)	0.0206(4)	0.0165(5)	0.0043(1)	-0.0008(5)	-0.0010(3)	-0.0001(2)
O(C2)	0.5879(4)	0.1028(2)	0.0945(1)	0.0216(6)	0.0126(4)	0.0035(1)	0.0028(4)	0.0013(2)	0.0003(2)
O(C3)	0.2320(3)	0.1855(2)	0.0167(1)	0.0211(6)	0.0099(3)	0.0026(1)	-0.0013(4)	0.0000(2)	-0.0001(1)
O(C4)	0.0202(3)	0.4196(3)	0.0968(1)	0.0212(6)	0.0112(4)	0.0031(1)	0.0021(4)	-0.0016(2)	-0.0000(2)
O(C5)	0.3369(3)	0.2940(3)	0.2583(1)	0.0176(6)	0.0148(4)	0.0024(1)	0.0031(5)	-0.0004(2)	0.0002(2)
O(C6)	-0.0695(4)	0.3473(3)	0.2956(1)	0.0201(6)	0.0168(5)	0.0013(1)	-0.0012(5)	0.0011(2)	-0.0007(2)
C(1)	0.4976(5)	0.2413(4)	0.2141(2)	0.0148(8)	0.0122(6)	0.0030(1)	-0.0004(6)	0.0001(3)	-0.0000(3)
C(2)	0.4293(5)	0.1601(4)	0.1393(2)	0.0172(8)	0.0100(6)	0.0030(1)	0.0002(6)	0.0011(3)	0.0002(2)
C(3)	0.2980(5)	0.2566(4)	0.0877(2)	0.0182(8)	0.0089(5)	0.0025(1)	-0.0014(6)	0.0000(3)	-0.0002(2)
C(4)	0.1325(5)	0.3143(4)	0.1392(2)	0.0175(8)	0.0081(5)	0.0027(1)	-0.0021(6)	-0.0004(3)	0.0002(2)
C(5)	0.2149(5)	0.3926(4)	0.2130(2)	0.0157(8)	0.0101(5)	0.0026(1)	-0.0000(6)	-0.0001(3)	-0.0003(2)
C(6)	0.0650(5)	0.4535(5)	0.2697(2)	0.0189(9)	0.0113(6)	0.0032(2)	0.0002(7)	-0.0004(3)	-0.0007(2)
C(U)	0.3777(6)	0.1788(5)	0.4886(2)	0.0188(9)	0.0125(6)	0.0033(2)	-0.0008(7)	0.0000(3)	0.0005(3)
O(U)	0.3889(4)	0.0894(3)	0.5453(1)	0.0352(9)	0.0128(4)	0.0029(1)	-0.0032(6)	-0.0011(3)	0.0006(2)
N(1)	0.3914(7)	0.3213(4)	0.5003(2)	0.0343(11)	0.0099(5)	0.0047(2)	-0.0021(8)	-0.0004(5)	-0.0002(2)
N(2)	0.3560(7)	0.1347(4)	0.4118(2)	0.0388(12)	0.0119(5)	0.0031(1)	-0.0015(8)	-0.0008(4)	0.0010(2)

Table 1 (cont.)

	x	y	z	B
H(C1)	0.593(4)	0.190(4)	0.254(2)	3.54
H(C2)	0.370(5)	0.072(4)	0.155(2)	3.54
H(C3)	0.374(4)	0.336(3)	0.069(2)	3.18
H(C4)	0.056(5)	0.231(4)	0.153(2)	3.26
H(C5)	0.299(4)	0.478(3)	0.189(2)	3.30
H(C6)	-0.009(5)	0.534(4)	0.240(2)	3.68
H(C6')	0.123(4)	0.496(3)	0.324(2)	3.68
H(O1)	0.713(5)	0.345(4)	0.233(2)	5.10
H(O2)	0.651(5)	0.170(4)	0.071(2)	4.15
H(O3)	0.193(5)	0.107(3)	0.031(2)	3.49
H(O4)	-0.031(5)	0.374(4)	0.062(2)	3.58
H(O6)	-0.099(6)	0.358(4)	0.345(2)	4.36
H(N1)	0.401(6)	0.398(4)	0.462(2)	5.00
H(N1')	0.398(7)	0.355(5)	0.544(2)	5.00
H(N2)	0.343(6)	0.046(4)	0.397(2)	4.94
H(N2')	0.356(6)	0.197(4)	0.370(2)	4.94

the reflection profile to give, in effect, a variable velocity $\theta-2\theta$ scan. These data for each reflection were integrated using an Aitken four point interpolation procedure, thereby eliminating the necessity of making any assumptions about peak shape. Standard deviations were estimated as the square root of the integrated intensities.

Two orthogonal reference reflections were measured after every forty reflections. These data were then used in an interpolation procedure to normalize all data to the initial values of the reference reflections. This procedure corrected for the observed systematic decrease

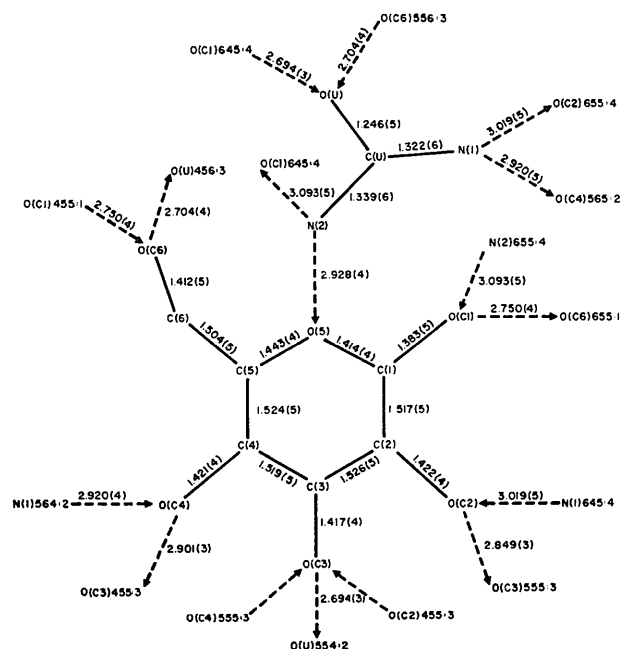


Fig. 2. Schematic diagram indicating the distances in the glucose and urea molecules (uncorrected for thermal motion) and showing the hydrogen-bonding arrangement for each atom. The symmetry code given after the atom labels (except for the central molecule) is explained in Table 5.

in intensity with exposure time. After the application of the Lorentz-polarization correction, equivalent reflections were averaged to give 1140 independent reflections, 48 of which were below the observational threshold. No absorption or extinction corrections were applied.

Solution and refinement of the structure

The structure was solved in a manner similar to that described by Karle & Karle (1966). From a list of the triplets involving the highest E 's the 450, 067 and 3,0,14 reflections were chosen as origin-defining and assigned phases of zero. The 703 reflection was assigned a phase of $\pi/2$ to specify the enantiomorph. Symbolic addition was then carried out (Karle & Hauptman, 1956) and starting phases were obtained for all reflections. These phases were refined using the tangent formula procedure of Hall (1967). Since the resulting E map failed to produce a recognizable structure, the highest five peaks were used to calculate structure factors from which the phases of 33 reflections with $E > 1.8$ and $F_c > \frac{1}{3}F_o$ were used as starting phases for another round of tangent refinement. The resulting E map contained the 16 non-hydrogen atoms as its highest 16 peaks.

The structure was refined using the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962) as modified by Shiono (1966). The atomic scattering factors for the non-hydrogen atoms were taken from Cromer & Waber (1965). The hydrogen form factor used was that of Stewart, Davidson & Simpson (1965). Initially, using $1/\sigma^2$ weights, the non-hydrogen atoms were refined isotropically. Anisotropic refinement reduced the residual ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$) to 0.097 and the weighted residual ($R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}$) to 0.092. At this stage of refinement a difference synthesis showed the location of all 16 hydrogen atoms. The final cycles of refinement were carried out with anisotropic thermal parameters for the non-hydrogen atoms and positional parameters only for the hydrogen atoms (assigning them the average isotropic B of the atom to which they are bonded). This led to $R = 0.041$ and $R_w = 0.040$. An analysis of variance, to check the validity of the $1/\sigma^2$ weighting function showed no significant dependence of the mean values of $|F_o - F_c|/\sigma(F_c)$ on the magnitude of either F_o or $\sin \theta/\lambda$. This and the low values of R and R_w is the result of the improved estimate of intensity, and standard deviation, resulting from the nine point scan technique.

The final parameters and their standard deviations are given in Table 1. The observed and calculated structure amplitudes are given in Table 2. Programs used in connection with this work, in addition to *ORFLS*, are *DIFN4* (McGandy & Seeman, 1968) and *DFOT4* (McGandy & Snyder, 1968b) diffractometer coordinate generation and processing programs for the IBM 1130; the *X-ray 63* system (Stewart, 1964), *ORTEP* (Johnson, 1965) ellipsoid plotting program and

Table 2. Observed and calculated structure factors

Columns are: Index, $10|F_{obs}|$, $10|F_{calc}|$, A_{calc} , B_{calc} . * indicates unobserved reflections.

h	k	l	$10 F_{obs} $	$10 F_{calc} $	A_{calc}	B_{calc}
0	0	0	12	35	27	27
0	12	35	27	27	0	0
0	24	20	7	7	0	0
0	36	20	7	7	0	0
0	48	20	7	7	0	0
0	60	20	7	7	0	0
0	72	20	7	7	0	0
0	84	20	7	7	0	0
0	96	20	7	7	0	0
0	108	20	7	7	0	0
0	120	20	7	7	0	0
0	132	20	7	7	0	0
0	144	20	7	7	0	0
0	156	20	7	7	0	0
0	168	20	7	7	0	0
0	180	20	7	7	0	0
0	192	20	7	7	0	0
0	204	20	7	7	0	0
0	216	20	7	7	0	0
0	228	20	7	7	0	0
0	240	20	7	7	0	0
0	252	20	7	7	0	0
0	264	20	7	7	0	0
0	276	20	7	7	0	0
0	288	20	7	7	0	0
0	300	20	7	7	0	0
0	312	20	7	7	0	0
0	324	20	7	7	0	0
0	336	20	7	7	0	0
0	348	20	7	7	0	0
0	360	20	7	7	0	0
0	372	20	7	7	0	0
0	384	20	7	7	0	0
0	396	20	7	7	0	0
0	408	20	7	7	0	0
0	420	20	7	7	0	0
0	432	20	7	7	0	0
0	444	20	7	7	0	0
0	456	20	7	7	0	0
0	468	20	7	7	0	0
0	480	20	7	7	0	0
0	492	20	7	7	0	0
0	504	20	7	7	0	0
0	516	20	7	7	0	0
0	528	20	7	7	0	0
0	540	20	7	7	0	0
0	552	20	7	7	0	0
0	564	20	7	7	0	0
0	576	20	7	7	0	0
0	588	20	7	7	0	0
0	600	20	7	7	0	0
0	612	20	7	7	0	0
0	624	20	7	7	0	0
0	636	20	7	7	0	0
0	648	20	7	7	0	0
0	660	20	7	7	0	0
0	672	20	7	7	0	0
0	684	20	7	7	0	0
0	696	20	7	7	0	0
0	708	20	7	7	0	0
0	720	20	7	7	0	0
0	732	20	7	7	0	0
0	744	20	7	7	0	0
0	756	20	7	7	0	0
0	768	20	7	7	0	0
0	780	20	7	7	0	0
0	792	20	7	7	0	0
0	804	20	7	7	0	0
0	816	20	7	7	0	0
0	828	20	7	7	0	0
0	840	20	7	7	0	0
0	852	20	7	7	0	0
0	864	20	7	7	0	0
0	876	20	7	7	0	0
0	888	20	7	7	0	0
0	900	20	7	7	0	0
0	912	20	7	7	0	0
0	924	20	7	7	0	0
0	936	20	7	7	0	0
0	948	20	7	7	0	0
0	960	20	7	7	0	0
0	972	20	7	7	0	0
0	984	20	7	7	0	0
0	996	20	7	7	0	0
0	1008	20	7	7	0	0
0	1020	20	7	7	0	0
0	1032	20	7	7	0	0
0	1044	20	7	7	0	0
0	1056	20	7	7	0	0
0	1068	20	7	7	0	0
0	1080	20	7	7	0	0
0	1092	20	7	7	0	0
0	1104	20	7	7	0	0
0	1116	20	7	7	0	0
0	1128	20	7	7	0	0
0	1140	20	7	7	0	0
0	1152	20	7	7	0	0
0	1164	20	7	7	0	0
0	1176	20	7	7	0	0
0	1188	20	7	7	0	0
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0	1212	20	7	7	0	0
0	1224	20	7	7	0	0
0	1236	20	7	7	0	0
0	1248	20	7	7	0	0
0	1260	20	7	7	0	0
0	1272	20	7	7	0	0
0	1284	20	7	7	0	0
0	1296	20	7	7	0	0
0	1308	20	7	7	0	0
0	1320	20	7	7	0	0
0	1332	20	7	7	0	0
0	1344	20	7	7	0	0
0	1356	20	7	7	0	0
0	1368	20	7	7	0	0
0	1380	20	7	7	0	0
0	1392	20	7	7	0	0
0	1404	20	7	7	0	0
0	1416	20	7	7	0	0
0	1428	20	7	7	0	0
0	1440	20	7	7	0	0
0	1452	20	7	7	0	0
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0	1788	20	7	7	0	0
0	1800	20	7	7	0	0
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0	1824	20	7	7	0	0
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0	1872	20	7	7	0	0
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0	1944	20	7	7	0	0
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0	1992	20	7	7	0	0
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0	2016	20	7	7	0	0
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0	2340	20	7	7	0	0
0	2352	20	7	7	0	0
0	2364	20	7	7	0	0
0	2376	20	7	7	0	0
0	2388	20	7	7	0	0
0	2400	20	7	7	0	0
0	2412	20	7	7	0	0
0	2424	20	7	7	0	0
0	2436	20	7	7	0	0
0	2448	20	7	7	0	0
0	2460	20	7	7	0	0
0	2472	20	7	7	0	0
0	2484	20	7	7	0	0
0	2496	20	7	7	0	0
0	2508	20	7	7	0	0
0	2520	20	7	7	0	0
0	2532	20	7	7	0	0
0	2544	20	7	7	0	0
0	2556	20	7	7	0	0
0	2568	20	7	7	0	0
0	2580	20	7	7	0	0
0	2592	20	7	7	0	0
0	2604	20	7	7	0	0
0	2616	20	7	7	0	0
0	2628	20	7	7	0	0
0	2640	20	7	7	0	0
0	2652	20	7	7	0	0
0	2664	20	7	7	0	0
0	2676	20	7	7	0	0
0	2688	20	7	7	0	0
0	2700	20	7	7	0	0
0	2712	20	7	7	0	0
0	2724	20	7	7	0	0
0	2736	20	7	7	0	0
0	2748	20	7	7	0	0
0	2760	20	7	7	0	0
0	2772	20	7	7	0	0
0	2784	20	7	7	0	0
0	2796	20	7	7	0	0
0	2808	20	7	7	0	0
0	2820	20	7	7	0	0
0	2832	20	7	7	0	0
0	2844	20	7	7	0	0

WEIGHT (Snyder, 1969) analysis of variance program, all for the IBM 7090.

Description of the structure

The conformation and numbering of the molecules are shown in Fig. 1. The urea molecule is planar within experimental error and the dimensions, shown in Fig. 2, with the exception of slightly shorter C–O bond length, agree within 3σ with those observed in the structure of urea, by Caron & Donohue (1964).

The α -D-glucose distances, also shown in Fig. 2, agree with those for the crystal structure of the parent compound (Brown & Levy, 1965) within 1σ , except for C(1)–O(5), C(5)–O(5) and C(1)–C(2). Whereas Brown & Levy (1965) observed equal C–O ring bond lengths (1.425, 1.427 Å), the difference in this structure is 7σ and even allowing for an underestimate of σ this is probably significant. Similar differences in other pyranose sugars have been reported by Berman, Chu & Jeffrey (1967). The bond angles are compared in Table 3; there are no outstanding differences. The conformation of the primary alcohol hydroxyl with respect to the ring oxygen for pure glucose, glucose–urea and the glucose hydrate (Killean, Ferrier & Young, 1962) is

shown in Fig. 3. In both the complex and the hydrate, O(6) is anti-parallel to hydrogen (*gauche-trans*). The other conformation angles between C–O bonds with respect to the C–O bonds are similar to those in the glucose structures, *i.e.* C(4)–C(5) = -178.1° , C(4)–C(3) = -61.2° , C(3)–C(2) = $+54.1^\circ$ and C(2)–C(1) = $+48.0^\circ$. Of the three conformations shown in Fig. 3, that of the α -glucose would appear to be the more stable, since it has one, rather than two, O...O or O...C third neighbour non-bonding interactions. Presumably, the more extensive hydrogen bonding in the complex and the hydrate balances the energetically less favorable conformation. This conformational difference may also be the cause of the difference in the C–O ring bond lengths which is observed in the complex but not in the pure compound.

The hydrogen bond distances are given in Table 4. The H...O or H...N distances greater than 2.0 Å involve either a nitrogen from the urea or the O(3) of the glucose, which, because it forms three hydrogen bonds, is sterically crowded.

The crystal structure is held together by a strong three-dimensional hydrogen-bond network. Each of the nine hydrogen atoms covalently bonded to oxygen and nitrogen atoms forms a hydrogen bond. As shown in

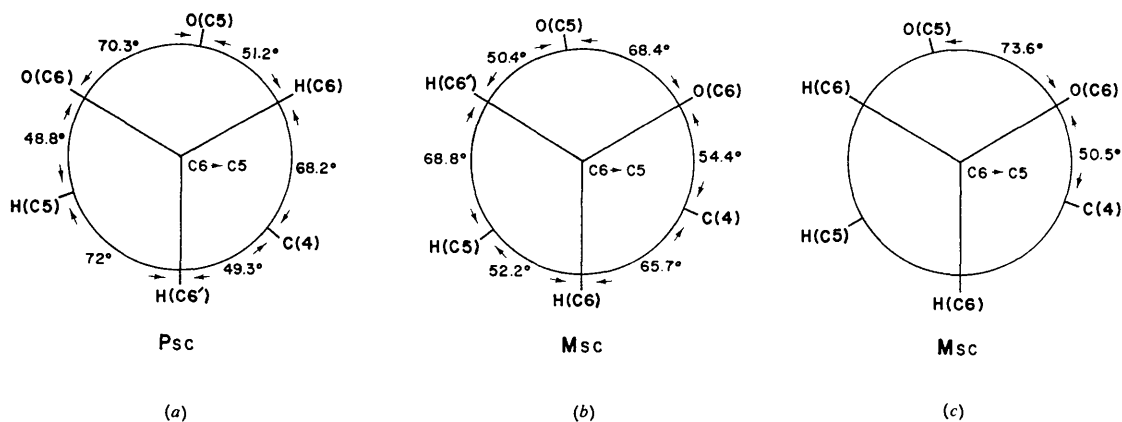


Fig. 3. Newman projections down the exocyclic C(5)–C(6) bond for (a) pure α -D-glucose (Brown & Levy, 1965), (b) α -D-glucose–urea and (c) α -D-glucose–H₂O (Killean, Ferrier & Young, 1962).

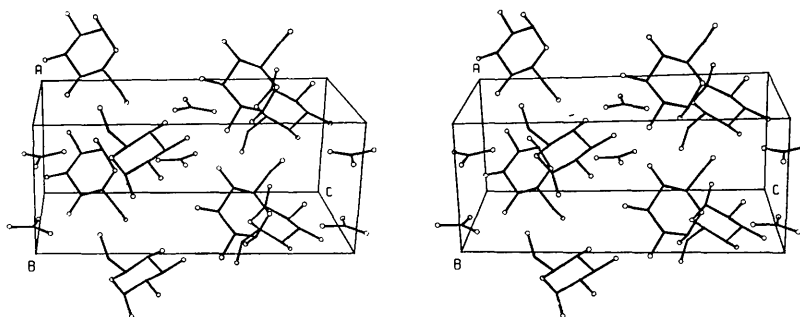


Fig. 4. The packing of the molecules in the unit cell. The pictures constitute a stereoscopic pair and may be viewed with a small hand stereoscope.

Table 3. Angles involving the non-hydrogen atoms in the α -D-glucose of the complex and the pure compound

<i>i</i>	<i>j</i>	<i>k</i>	Angle (<i>ijk</i>)	
			This work	Brown & Levy (1965)
O(1)	C(1)	O(5)	112.0 (3)°	111.6°
O(1)	C(1)	O(2)	109.2 (3)	109.4
O(5)	C(1)	C(2)	110.1 (3)	110.1
O(2)	C(2)	C(1)	111.3 (3)	110.9
O(2)	C(2)	C(3)	112.3 (5)	112.3
C(1)	C(2)	C(3)	110.8 (3)	111.1
O(3)	C(3)	C(2)	112.8 (3)	108.1
O(3)	C(3)	C(4)	112.3 (3)	110.6
C(2)	C(3)	C(4)	109.8 (3)	109.8
O(4)	C(4)	C(3)	111.9 (3)	108.3
O(4)	C(4)	C(5)	106.1 (3)	110.9
C(3)	C(4)	C(5)	109.2 (3)	111.2
O(5)	C(5)	C(4)	109.8 (3)	108.7
O(5)	C(5)	C(6)	108.2 (3)	108.1
C(4)	C(5)	C(6)	114.5 (3)	111.5
O(6)	C(6)	C(5)	112.8 (3)	110.4
C(5)	O(5)	C(1)	113.9 (3)	113.8

Figs. 4 and 5, a glucose and urea form a pair, within the same cell and symmetry operation (*i.e.* 555:1, see Table 5), which is hydrogen-bonded to 15 adjacent molecules. The urea molecules are hydrogen-bonded only to glucose molecules. The only example of two

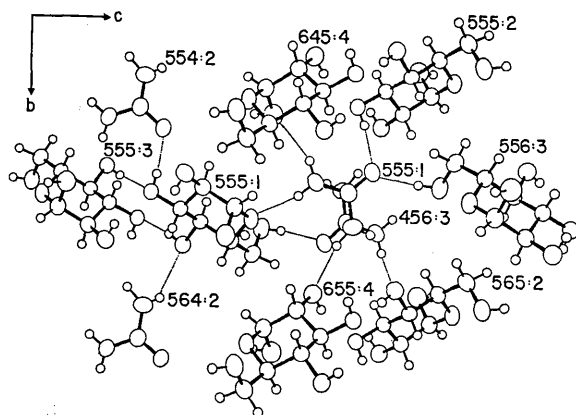


Fig. 5. View of hydrogen bonding down the *a* axis. The nine peripheral molecules all hydrogen-bond (dotted lines) to the central (555:1) glucose and urea molecules. The symmetry code is explained in Table 5.

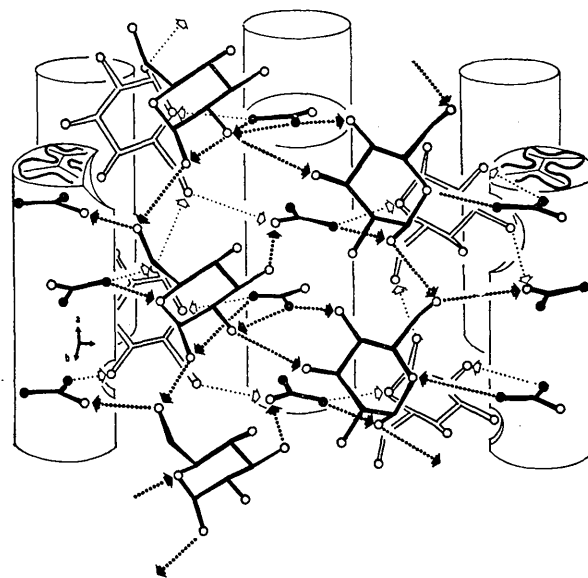


Fig. 6. Schematic representation of the crystal structure of glucose-urea showing a complete set of hydrogen bonds (dotted lines) between glucose and urea. For the purposes of clarity most of the inter-glucose hydrogen bonds have been omitted. The projection of the ureas are drawn at the top of two of the forward cylinders.

hydrogen bonds linking the same type of molecule occurs between the 555:1 and 555:3 glucoses, through the O(2) and O(3) of the 555:1 molecule.

The packing of the molecules in the unit cell is shown stereoscopically in Fig. 4. The urea molecules are staggered about the *a* screw axis with their planes nearly normal to it. This arrangement of a urea and its environment of glucose molecules in a projection down the *a* axis is shown in Fig. 5. The principal features of the crystal structure and hydrogen-bonding arrangement are illustrated in Fig. 6. Each glucose, in the infinite rows of glucose molecules, links together a pair of urea cylinders. It also bridges a pair of adjacent urea molecules within each of the two cylinders. In addition each glucose row is held together by hydrogen bonds from the anomeric hydroxyl of one glucose to the primary alcohol of the next molecule in the row. This arrangement then forms a stepping linkage in the *a* direction with the O(6) of a glucose donating to the oxygen of urea and accepting from the O(1) of the

Table 4. Hydrogen bonds in glucose-urea

C	X	H	O	<i>D</i> (X-H)	<i>D</i> (H···O)	\angle (C-X···O)	\angle (X-H···O)
C(1)	O(C1)	H(O1)	O(C6)	0.95 (4) Å	1.81 (4) Å	107.0 (2)°	166 (3)°
C(2)	O(C2)	H(O2)	O(C3)	0.85 (4)	2.04 (4)	110.7 (2)	159 (3)
C(3)	O(C3)	HO(3)	O(U)	0.80 (3)	1.91 (3)	112.5 (1)	169 (3)
C(4)	O(C4)	H(O4)	O(C3)	0.79 (3)	2.16 (3)	117.9 (2)	156 (3)
C(6)	O(C6)	H(O6)	O(U)	0.85 (4)	1.87 (4)	102.5 (2)	166 (3)
C(U)	N(1)	H(N1)	O(C2)	0.95 (4)	2.09 (4)	140.3 (3)	166 (3)
C(U)	N(1)	H(N1')	O(C4)	0.79 (4)	2.31 (4)	153.6 (3)	135 (4)
C(U)	N(2)	H(N2)	O(C1)	0.85 (4)	2.31 (4)	140.0 (3)	154 (4)
C(U)	N(2)	H(N2)	O(C5)	0.89 (4)	2.06 (4)	132.4 (3)	165 (4)

X=O or N

Table 5. *Symmetry code*

The positional parameters of atoms related by symmetry to those listed in Table 1 are given by the four digit code $T_aT_bT_c:S$. The first three digits refer to a lattice translation with respect to the origin cell at $T_aT_bT_c=555$, e.g. 564 means a translation of $(5-5)a+(6-5)b+(4-5)c$ or $(0, 1, -1)$ after the application of the symmetry operator. The fourth digit refers to one of the following symmetry operations:

- (1) x, y, z
- (2) $\frac{1}{2}-x, \bar{y}, \frac{1}{2}+z$
- (3) $\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
- (4) $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$.

glucose above it. The cross linking between the columns of glucose molecules is shown in Fig. 5.

The repeating hydrogen-bond scheme is shown in Fig. 7, the top half of which shows the relation of the hydrogen-bonding scheme to the crystal structure. The bottom half of the figure is a schematic drawing showing the hydrogen-bonding scheme in the form of a Y. The stem of the Y [from N(2) to O(3)] is a closed system of H bonds terminating within itself. The repeating hydrogen-bond scheme is composed only of the five atom sequence forming the top of the Y.

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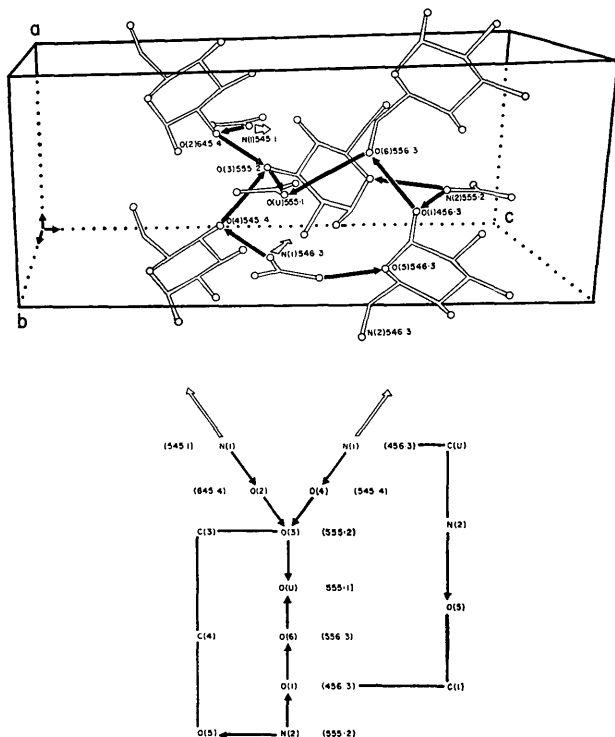


Fig. 7. Repeating hydrogen bond scheme in the α -D-glucose-urea structure. The symmetry code is explained in Table 5.

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