References

Berman, H. M., McGandy, E. L., Burgner, J. W. & VanEtten, R. L. (1969). *J. Amer. Chem. Soc.* **91**, 6177–6182.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE*. Report ORNL-TM-306, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
Gold, E. H. (1968). Schering Corporation. U.S. Patent 3600380.
Gold, E. H. (1971). Private communication.
International Tables for X-ray Crystallography (1968). Vol. III, 2nd ed., pp. 202–203. Birmingham: Kynoch Press.
Johnson, C. K. (1970). *ORTEP*. Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
McGandy, E. L., Berman, H. M., Burgner, J. W. & VanEtten, R. L. (1969). *J. Amer. Chem. Soc.* **91**, 6173–6177.
Parker, W. L., Flynn, J. J. & Boer, F. P. (1968). *J. Amer. Chem. Soc.* **90**, 4723–4729.
Simpson, P. G. (1963). Ph.D. Thesis, Harvard University.
Towns, R. L. & Trefonas, L. M. (1971). *J. Amer. Chem. Soc.* **93**, 1761–1764.

Acta Cryst. (1974). B**30**, 537

Crystal Structure of α-Glucuronamide

By Judith L. Flippen and Richard D. Gilardi

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, D.C. 20375, *U.S.A.*

(*Received* 4 *September* 1973; *accepted* 25 *September* 1973)

Abstract. $C_6H_{11}NO_6$, monoclinic, $P2_1$, $a=9·229$ (12), $b=5·179$ (4), $c=8·261$ (8) Å, $\beta=102·9$ (2)°, $Z=2$, $D_x=1·67$ g cm^{-3}. The structure was solved by routine application of the symbolic addition procedure and refined to a final R index of 5·1 %.

Experimental. Glucuronamide is the amide of glucuronic acid which is one of the components of the polysaccharide heparin. The crystal was selected from a commercial sample (Sigma Chemical Company). The 699 independent reflections were collected from a small crystal ($\sim 0·34 \times 0·12 \times 0·07$ mm) on an automatic diffractometer using Cu $K\alpha$ radiation ($\lambda=1·54178$ Å, Ni filter). The θ–2θ scan mode of data collection was used (maximum $\sin\theta/\lambda=0·567$ Å^{-1}).

The structure was solved by the symbolic addition procedure for non-centrosymmetric crystals (Karle & Karle, 1966). Full-matrix least-squares methods (Busing *et al.*, 1971) were used to refine the structure. The atomic scattering factors used were those listed in *International Tables for X-ray Crystallography* (1962). The function minimized was $\sum w(|F_o|-|F_c|)^2$ where the weights were calculated according to the procedure outlined by Gilardi (1973). The final R index is 0·051 ($R_w=0·050$). The hydrogen atoms were located in a difference map. Positional parameters for the hydrogen atoms were included in the refinement. Hydrogen thermal factors (set equal to the final isotropic value for the atom to which they were bonded) were not refined. Table 1 lists the final coordinates and thermal

Table 1. *Fractional coordinates and thermal parameters with standard deviations*

The thermal parameters are of the form $T=\exp[\frac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)]$.

Standard deviations are based solely on least-squares parameters.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
N(1)	0·3546 (6)	0·6226 (13)	−0·0444 (6)	3·9 (2)	1·7 (2)	3·0 (2)	−0·8 (2)	1·6 (2)	−0·1 (2)
C(1)	0·0692 (6)	0·6069 (16)	0·2666 (6)	2·3 (2)	1·8 (2)	2·9 (2)	0·0 (2)	1·2 (2)	−0·2 (2)
C(2)	0·1801 (6)	0·5506 (13)	0·4309 (6)	2·6 (2)	1·2 (3)	2·7 (2)	−0·0 (2)	0·9 (2)	0·1 (2)
C(3)	0·2622 (6)	0·2968 (13)	0·4205 (7)	2·4 (2)	1·6 (2)	2·0 (2)	0·1 (2)	0·3 (2)	−0·3 (2)
C(4)	0·3342 (6)	0·3082 (13)	0·2730 (7)	2·4 (2)	1·5 (2)	2·3 (2)	−0·1 (2)	0·7 (2)	−0·2 (2)
C(5)	0·2152 (5)	0·3674 (13)	0·1144 (6)	2·1 (2)	1·9 (3)	2·4 (2)	0·4 (2)	0·6 (2)	−0·3 (2)
C(6)	0·2898 (5)	0·3961 (14)	−0·0295 (6)	2·1 (2)	2·4 (3)	2·4 (2)	−0·1 (2)	0·5 (2)	0·3 (2)
O(1)	−0·0435 (4)	0·4226 (11)	0·2436 (5)	2·2 (1)	2·7 (2)	3·5 (2)	−0·2 (2)	0·5 (1)	0·7 (2)
O(2)	0·1069 (4)	0·5276 (12)	0·5642 (4)	3·6 (2)	2·8 (2)	2·7 (2)	0·6 (2)	1·5 (1)	0·0 (2)
O(3)	0·3761 (4)	0·2542 (11)	0·5641 (4)	2·4 (2)	2·6 (2)	2·0 (1)	0·2 (2)	0·4 (1)	0·0 (1)
O(4)	0·4031 (4)	0·0696 (10)	0·2453 (4)	3·1 (2)	2·2 (2)	2·1 (1)	1·0 (2)	0·5 (1)	−0·0 (1)
O(5)	0·1442 (4)	0·6078 (0)	0·1335 (4)	2·7 (2)	1·3 (2)	2·5 (2)	0·4 (1)	1·0 (1)	0·3 (1)
O(6)	0·2939 (4)	0·2094 (11)	−0·1259 (4)	3·3 (2)	2·1 (2)	2·1 (1)	−0·2 (2)	0·8 (1)	−0·4 (1)

Table 1 (*cont.*)

	x	*y*	*z*
H(1)	0·022 (7)	0·797 (17)	0·268 (8)
H(2)	0·258 (7)	0·654 (17)	0·441 (8)
H(3)	0·195 (7)	0·165 (16)	0·404 (8)
H(4)	0·412 (7)	0·448 (16)	0·296 (8)
H(5)	0·146 (7)	0·226 (17)	0·085 (7)
H(N*A*)	0·420 (7)	0·638 (16)	−0·137 (8)
H(N*B*)	0·344 (7)	0·796 (16)	0·037 (8)
H(O1)	−0·140 (7)	0·469 (18)	0·191 (8)
H(O2)	0·165 (7)	0·559 (20)	0·618 (9)
H(O3)	0·331 (7)	0·231 (17)	0·646 (8)
H(O4)	0·473 (7)	0·047 (18)	0·340 (8)

factors and bond distances and angles are illustrated in Fig. 1.*

Discussion. The molecule is illustrated in Fig. 2. The pyranose ring has the normal $^4C_1$ conformation with all conformational angles falling in the normal range (see Table 2). The average C–OH bond length (excluding the anomeric C–OH bond) is 1·422 (6) Å and the average ring C–C bond length is 1·530 (6) Å. These values agree well with the values of 1·425 Å for C–OH bonds and 1·524 Å for C–C bonds given in the review of carbohydrate structures by Strahs (1970). The anomeric C–OH bond length [C(1)–O(1) = 1·393 (6) Å] is significantly shorter than the other C–OH bonds, and is close to the mean value (1·401 Å) reported by Strahs (1970) for an axial anomeric hydroxyl distance. The amide group is staggered with respect to the C(4)–C(5) and C(5)–O(5) ring bonds, but is rotated about 20° from a position bisecting the inter-bond dihedral angle (see Table 2). The C(5)–C(6) distance of 1·508 Å is reasonable for a carbon–carbon single bond next to a C=O bond. In potassium *β*-D-glucuronate (Gurr, 1963) this bond was reported to be unusually long (1·567 Å). The carbonyl bond, C(6)–O(6), is somewhat long (1·258 Å) for an amide carbonyl bond, but this may be accounted for by the participation of O(6) as an acceptor in two strong hydrogen bonds.

Table 2. *Pertinent torsion angles*

Pyranose ring	
O(5)–C(1)–C(2)–C(3)	−55·8 (7)°
C(1)–C(2)–C(3)–C(4)	54·9 (6)
C(2)–C(3)–C(4)–C(5)	−55·9 (6)
C(3)–C(4)–C(5)–O(5)	58·7 (6)
C(4)–C(5)–O(5)–C(1)	−61·5 (5)
C(5)–O(5)–C(1)–C(2)	59·9 (6)
Amide group	
O(5)–C(5)–C(6)–N(1)	38·5 (6)°
O(5)–C(5)–C(6)–O(6)	−143·8 (5)
C(4)–C(5)–C(6)–O(6)	37·3 (6)
C(4)–C(5)–C(6)–N(1)	−80·3 (6)

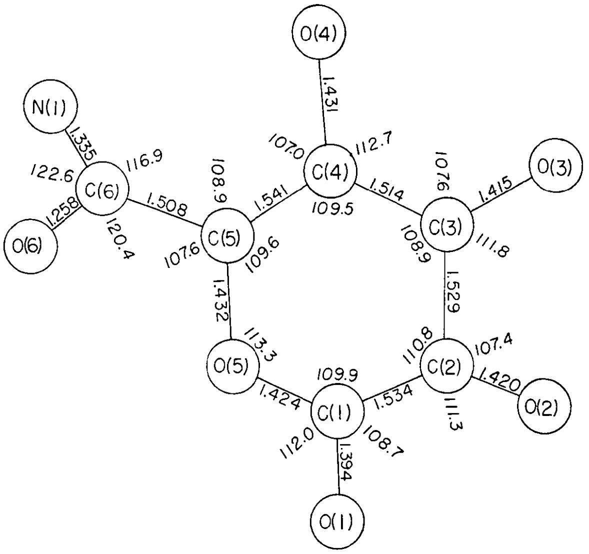

Fig. 1. Bond distances and angles for α-glucuronamide. Based solely on least-squares parameters, the standard deviations are on the order of 0·006 Å for the bond lengths and 0·4° for the bond angles.

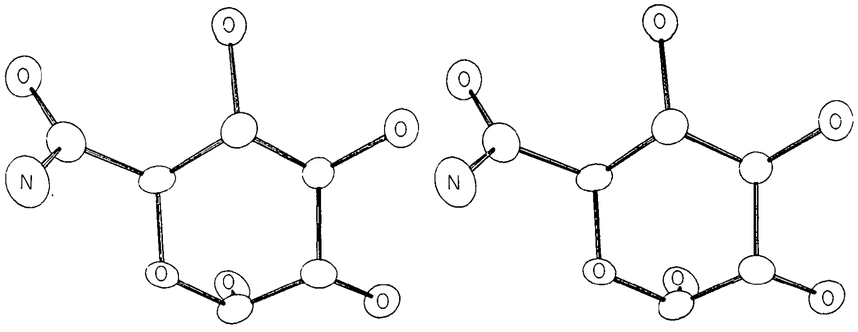

Fig. 2. Stereodiagram of a molecule of α-glucuronamide.

---

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30232 (5pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

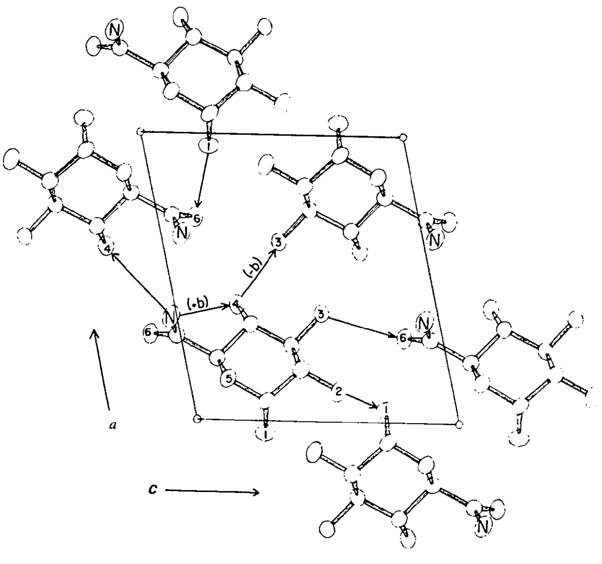

Fig. 3. A projection down the *b* axis showing the packing in the unit cell. The five unique hydrogen bonds are shown as arrows directed from donor to acceptor atoms. The notation (+*b*) or (−*b*) on an arrow indicates that the hydrogen bond is to a molecule translated by that amount parallel to the *b* axis. The close intermolecular approach between N(1) and O(4) is also indicated by an arrow. The molecule in the lower left corner of the cell is the reference molecule whose coordinates are in Table 1; the molecule in the upper right corner of the cell is related by the symmetry operation $1-x$, $\frac{1}{2}+y$, $1-z$. All others are related to these two by lattice translations of *a* or *c*.

Some aspects of the packing are illustrated in Fig. 3. On the basis of heavy atom (N, O) intermolecular contact distances, there appear to be five unique hydrogen bonds, which is one less than the maximum possible number for this molecule, and they are shown in Fig. 3. The hydroxyl on C(2) is not an acceptor for an H-bond; all other oxygen atoms are acceptors, with O(4) and O(6) each accepting two hydrogen bonds. The hydrogen atom labelled H(O2) does not lie on the indicated H-bond between O(2) and O(1), which are only 2·732 Å from one another. The angle O(2)–H(O2)···O(1) is only 96°. No appreciable density was found in the difference map on the line connecting these two atoms. N(1) is involved in close intermolecular approaches with four different oxygen atoms (see Table 2). The two hydrogen atoms located on N(1) were found to lie approximately along the lines connecting N(1) to O(4) [N(1)–H(NA)–O(4) = 156·7°] and to an O(4) atom in a different molecule [N(1)–H(NB)–O(4) = 158·3°]. However, the second N(1)–O(4) separation of 3·29 Å is too long to be called a true hydrogen bond. The hydrogen-bond distances and selected close intermolecular approaches are listed in Table 3.

Table 3. *Hydrogen-bonded intermolecular contacts*

Donor	Acceptor	Distance (±0·01 Å)
N(1)	O(4) $1-x, \frac{1}{2}+y, -z$	3·08
H(N1)	O(4)	2·06
O(1)	O(6) $-x, \frac{1}{2}+y, -z$	2·74
H(O1)	O(6)	1·88
O(2)	O(1) $-x, \frac{1}{2}+y, 1-z$	2·73
H(O2)	O(1)	2·59
O(3)	O(6) $x, y, 1+z$	2·84
H(O3)	O(6)	1·99
O(4)	O(3) $1-x, y-\frac{1}{2}, 1-z$	2·80
H(O4)	O(3)	2·09

Hydrogen-bond angles

N(1)–H···O(4)	156·7°
O(1)–H···O(6)	153·1
O(2)–H···O(1)	96·1
O(3)–H···O(6)	161·1
O(4)–H···O(3)	134·9

Selected close intermolecular approaches

N(1)	O(6) $x, 1+y, z$	3·14 Å
N(1)	O(6) $1-x, \frac{1}{2}+y, \bar{z}$	3·26
N(1)	O(4) $x, 1+y, z$	3·29

This investigation was supported in part by the Office of Naval Research Project No. P04-0095.

References

Busing, W. R., Martin, K. O., Levy, H. A., Ellison, R. D., Hamilton, W. C., Ibers, J. A., Johnson, C. K. & Thiessin, W. E. (1971). *ORXFLS*3. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
Gilardi, R. D. (1973). *Acta Cryst.* **B29**, 2089–2095.
Gurr, G. E. (1963). *Acta Cryst.* **16**, 690–696.
International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
Karle, J. & Karle, I. L. (1966). *Acta Cryst.* **21**, 849–859.
Strahs, G. (1970). *Advanc. Carbohydr. Chem.* **25**, 53–107.