

3-O-Methyl- α -D-glucopyranose, C₇H₁₄O₆

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Abstract. $M_r = 194.08$, orthorhombic, $P2_12_12_1$, $a = 13.942$ (1), $b = 11.381$ (1), $c = 5.354$ (1) Å, $V = 849.6$ Å³, $Z = 4$, $D_x = 1.52$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 1.045$ mm⁻¹, $F(000) = 416$. Final $R = 0.0708$ from 835 unique reflections measured at 293 K. All O atoms, except for the ring O, are involved in hydrogen bonding. O(4)··O(4') bonds link molecules together along the c direction.

Introduction. The title compound is a monosaccharide with one substituted hydroxyl group. The structure was determined to establish the variation of the hydrogen bonds compared with the unsubstituted sugar (Brown & Levy, 1979) and the α -substituted methyl α -D-glucopyranoside (Berman & Kim, 1968).

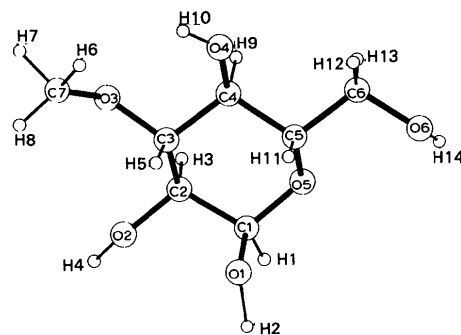
Experimental. Material supplied by Sigma Chemical Company, recrystallized from water/2-propanol, rectangular prism, $0.36 \times 0.20 \times 0.15$ mm, Enraf-Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; reflections measured for four octants to $2\theta = 140^\circ$ for the ranges of h , k and l of -16 to 16 , -13 to 13 and 0 to 6 respectively, 2696 reflections measured plus 880 flagged as ≤ 0 ; reflection 060 was used as intensity control (99 measurements) and gave an average count of 38267 with a $\sigma(\text{calculated}) = 1077$ ($= 2.8\%$); data merged using *SHELX* (Sheldrick, 1976) to give 907 unique reflections with a merging $R = 0.038$, 72 reflections eliminated with $F < 3\sigma(F)$; structure solved with *MULTAN* 80 (Main *et al.*, 1980), non-H atoms refined by least squares first with isotropic thermal parameters then anisotropic, H-atom coordinates established from a difference Fourier map, additional refinement carried out with addition of H atoms; a damping factor of 300 was applied to H-atom coordinates, their thermal vibration parameters fixed at equivalent U_{iso} of atom to which each H atom is attached; for final cycle maximum shift/error 0.035 with an average 0.007, unit weights used, difference Fourier map showed a maximum value of 0.29 e \AA^{-3} and a minimum of -0.30 e \AA^{-3} , atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974).

Table 1. Atom coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$)
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C(1)	-1785 (5)	1179 (6)	3370 (14)	463
C(2)	-2713 (4)	1544 (6)	2118 (14)	423
C(3)	-2882 (4)	2837 (6)	2350 (14)	415
C(4)	-2014 (4)	3537 (6)	1510 (13)	398
C(5)	-1102 (4)	3083 (6)	2797 (13)	391
C(6)	-222 (4)	3692 (6)	1738 (14)	453
C(7)	-4389 (5)	3903 (7)	2000 (16)	574
O(1)	-1905 (4)	1361 (4)	6019 (9)	518
O(2)	-3495 (3)	843 (4)	2901 (10)	469
O(3)	-3699 (3)	3158 (4)	840 (9)	482
O(4)	-2122 (3)	4753 (4)	1727 (10)	483
O(5)	-1001 (3)	1858 (4)	2488 (9)	442
O(6)	642 (3)	3288 (4)	2880 (10)	511

Table 2. Bond distances (Å) and angles ($^\circ$)

C(1)–C(2)	1.515 (9)	C(2)–O(2)	1.414 (7)
C(2)–C(3)	1.495 (9)	C(3)–O(3)	1.444 (7)
C(3)–C(4)	1.518 (9)	C(4)–O(4)	1.396 (7)
C(4)–C(5)	1.535 (8)	C(6)–O(6)	1.427 (8)
C(5)–C(6)	1.519 (9)	C(7)–O(3)	1.424 (8)
C(5)–O(5)	1.411 (8)	C(1)–O(5)	1.419 (8)
C(1)–O(1)	1.443 (9)		
O(1)–C(1)–C(2)	107.2 (6)	C(3)–C(4)–C(5)	110.5 (5)
O(5)–C(1)–C(2)	111.2 (6)	C(3)–C(4)–O(4)	114.1 (5)
O(5)–C(1)–O(1)	109.8 (6)	O(4)–C(4)–C(5)	112.7 (5)
C(1)–C(2)–C(3)	111.6 (6)	C(4)–C(5)–C(6)	110.3 (5)
C(1)–C(2)–O(2)	111.9 (5)	C(4)–C(5)–O(5)	111.3 (5)
O(2)–C(2)–C(3)	114.1 (5)	O(5)–C(5)–C(6)	109.1 (5)
C(2)–C(3)–C(4)	111.5 (5)	C(1)–O(5)–C(5)	115.0 (5)
C(2)–C(3)–O(3)	109.1 (6)	C(5)–C(6)–O(6)	112.0 (6)
O(3)–C(3)–C(4)	109.3 (6)	C(3)–O(3)–C(7)	116.0 (5)

Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO* 78 (Motherwell, 1978).

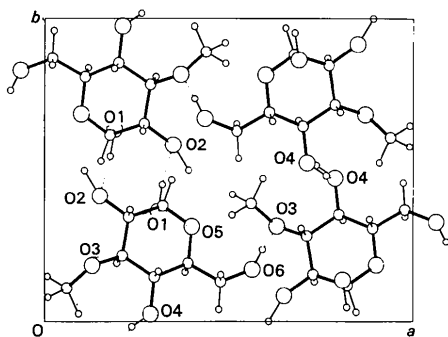


Fig. 2. Projection of the crystal structure along *c*. Broken lines indicate hydrogen bonds (PLUTO 78).

Discussion. Table 1* lists the atom coordinates and equivalent U_{iso} 's, Table 2 contains bond distances and angles. Fig. 1 shows the molecule and Fig. 2 is a *c*-axis projection of the unit cell. Hydrogen bonds are formed between O(4) and O(4') ($-\frac{1}{2}-x, 1-y, -\frac{1}{2}+z$) (forming a chain in the *c* direction), O(6) and O(3') ($-\frac{1}{2}+x,$

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38223 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$\frac{1}{2}-y, 1-z$) and O(1) and O(2') ($-\frac{1}{2}-x, -y, \frac{1}{2}+z$) with O...O distances of 2.931, 2.742 and 2.760 Å respectively. There may also be a hydrogen bond between O(2) and O(1') ($-\frac{1}{2}-x, -y, -\frac{1}{2}+z$) with the same O...O distance. O(1), O(2) and O(4) thus act as donors and acceptors while O(3) is an acceptor only and O(6) is a donor only.

In α -D-glucose O(2), O(3), O(4) and O(6) are all both donors and acceptors, with O(5) acting as acceptor from O(1), while methyl α -D-glucopyranoside has O(2), O(3) and O(6) as both donors and acceptors, the only hydrogen bond common to all three being O(6)—O(3').

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References

- BERMAN, H. M. & KIM, S. H. (1968). *Acta Cryst.* B24, 897–904.
 BROWN, G. M. & LEVY, H. A. (1979). *Acta Cryst.* B35, 656–659.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN 80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MOTHERWELL, W. D. S. (1978). *PLUTO 78*. Cambridge Crystallographic Data Centre, England.
 SHELDRIK, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

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Acta Cryst. (1983). C39, 316

Elementary patterns in protein–nucleic acid interactions. VI. Structure of 3-(7-adeninyl)propionamide monohydrate: addendum. By M. TAKIMOTO, A. TAKENAKA and Y. SASADA, *Laboratory of Chemistry for Natural Products, Tokyo Institute of Technology, Nagatsuta, Midori-ku, Yokohama 227, Japan*

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Abstract

E.s.d.'s are given for the bond distances and angles in Fig. 1 of the paper by Takimoto, Takenaka & Sasada [*Acta Cryst.* (1983), C39, 73–75]. The e.s.d.'s are 0.003–0.004 Å for bond distances and 0.2–0.3° for bond angles not involving H atoms; those for distances involving H atoms are 0.03–0.04 Å. Complete lists of these values have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38082 (22 pp.).

All the information is contained in the *Abstract*.