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Short Communication

X-Ray Crystallographic Study of α-Glucose at 140 K

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Noticing a crystalline object within a small drop of clear fluid hanging on a cactus plant on the windowsill, we were led by curiosity to extract the crystal and use it for an X-ray diffraction experiment. The result of the structure analysis showed the compound to be α-glucose and as glucose obtained from biological material usually exists in a D-configuration, the molecule is presented as α-D-glucose in the present study. The molecule was studied by X-ray methods in 1952 by McDonald and Beevers,[1] by neutron diffraction methods in 1965 (the data refined in 1979) by Brown and Levy,[2] and by Hough *et al.* (1973) as the monohydrate.[3] All previous work was carried out at room temperature whereas the data in the present study was collected at 140 K.

Table 1. Crystal data and the experimental conditions.

Compound	α-Glucose
Formula	$C_6H_{12}O_6$
Crystal system	Orthorhombic
a/Å	4.9464(7)
b/Å	10.3440(15)
c/Å	14.8472(22)
Experimental temp./K	140
Space group	$P2_12_12_1$
M/D	180.16
No. of mol. pr. cell (*Z*)	4
Crystal dimensions/mm	0.5×0.5×0.5
Apparatus	NICOLET P3/F
Scan mode	θ/2θ
Scan speed/°min^{-1}	2.0–4.0
Scan range/°	0.9–1.0
Background count time/scan time	0.7
2θ range	3.0–90.0
No. indep. meas.	3574
No. observed [$I > 3.0\sigma(I)$]	2949
Test reflections	3
COR	0.02
$R = \Sigma\|F_o - F_c\| / \Sigma\|F_o\|$	0.037[a]
$Rw = [\Sigma w(\|F_o\| - \|F_c\|)^2 / \Sigma wF_o^2]^{1/2}$	0.043[a]
$GOF = [\Sigma w(\|F_o\| - \|F_c\|)^2 / (n-m)]^{1/2}$	

[a] *w* is the inverse of the variance of observed structure factors.

Experimental

The unit cell dimensions were determined by a least-squares fit of 25 general reflections ($35 < 2\theta < 46°$). The intensity data collection was monitored by measuring three test reflections at intervals of 100 measurements. No loss of intensity in the test reflections during the experiment was observed. Standard deviation in the measured intensities were calculated as $\sigma(I) = [C_T + (0.02C_N)^2]^{1/2}$, where C_T is the total number of counts and C_N is the scan count minus the background count. The intensity data were corrected for Lorentz and polarisation effects but not for absorption.

The structure was solved by using the program system MITHRIL and refinements were made with least-squares calculations. Hydrogen atomic positions were introduced from geometrical considerations and included in the refinements which proceeded with anisotropic temperature factors for the heavier atoms and isotropic temperature factors for hydrogen atoms. The refinement converged at an *R*-factor of 0.037. The experimental conditions are summarised in Table 1. Computer programs used are described in Refs. 4 and 5.

Table 4. Comparison of unit cell dimensions from the X-ray study at 140 K and the neutron study at room temperature.

Cell axis	This study	Brown and Levy
a/Å	4.9464(7)	4.9753(3)
b/Å	10.3440(15)	10.3662(9)
c/Å	14.8472(22)	14.8506(16)
Cell volume/Å^3	759.7(14)	765.92(11)
Density/g cm^{-1}	1.575(3)	1.562(2)

Table 3. Final fractional coordinates and equivalent temperature factors with estimated standard deviations for all atoms.

Atom	*x*	*y*	*z*	U_{eq}/Å^2*
O5	0.5399(2)	0.3647(1)	0.0151(1)	0.013
O1	0.1461(2)	0.3465(1)	0.1012(1)	0.020
O2	0.4366(2)	0.3866(1)	0.2606(1)	0.018
O3	0.6671(2)	0.6328(1)	0.2161(1)	0.021
O4	0.5698(2)	0.7179(1)	0.0350(1)	0.016
O6	0.4435(2)	0.4462(1)	−0.1693(1)	0.022
C1	0.4256(2)	0.3320(1)	0.1010(1)	0.013
C2	0.5477(2)	0.4180(1)	0.1748(1)	0.013
C3	0.5170(2)	0.5606(1)	0.1519(1)	0.012
C4	0.6223(2)	0.5856(1)	0.0571(1)	0.012
C5	0.4890(2)	0.4958(1)	−0.0114(1)	0.012
C6	0.6021(3)	0.5147(1)	−0.1049(1)	0.018
Atom	*x*	*y*	*z*	*U*
H1	0.483(3)	0.234(1)	0.115(1)	0.005
H2	0.747(3)	0.399(1)	0.177(1)	0.012
H3	0.308(3)	0.585(1)	0.156(1)	0.013
H4	0.809(3)	0.570(1)	0.054(1)	0.013
H5	0.287(4)	0.511(2)	−0.011(1)	0.019
H61	0.614(4)	0.605(2)	−0.122(1)	0.026
H62	0.804(4)	0.482(2)	−0.102(1)	0.022
H01	0.082(5)	0.275(2)	0.076(1)	0.054
H02	0.297(5)	0.445(2)	0.275(1)	0.037
H03	0.631(6)	0.702(3)	0.211(2)	0.061
H04	0.686(5)	0.747(2)	0.011(2)	0.041
H06	0.549(5)	0.4202(2)	−0.207(1)	0.038

* $U_{eq}=(U11+U22+U33)/3$ for anisotropic atoms.

Discussion

The expected shrinkage of the unit cell at lower temperature is displayed in Table 2 where the cell parameters found at low temperature are compared with those reported from the neutron study at room temperature. The cell volume is decreased by about 0.8% and the largest shrinkage occurs along the shortest axis (*a*).

The final atomic co-ordinates are given in Table 3 and the corresponding bond lengths and angles in Tables 4 and 5, where a comparison with data from earlier studies is also made. The structure is shown in Fig. 1. It appears from Table 4 that the bond lengths from the low-temperature study are in close agreement with the bond lengths given by Brown and Levy. However, the results from the present structure analyses indicates that there are highly significant differences in the C–C bond lengths: the C1–C2 bond being longer and the C5–C6 bond shorter, than any other C–C bond in the compound. Such differences are not found at a significant level in the neutron diffraction study, although the C1–C2 bond still is the longest and C5–C6 the shortest C–C bond in the structure. The comparison in Table 4 also shows that the C1–C2 bond in pure α-glucose in both the X-ray and the neutron study is found to be significantly longer than that reported for the hydrate.

Table 4. Bond lengths found in the low-temperature X-ray study of α-glucose (A) compared with those reported in the room-temperature neutron study of the same compounds (B), and those from the room-temperature X-ray study of α-D-glucose hydrate (C).

	A	B	C
C1–C2	1.535(2)	1.539(2)	1.509(3)
C2–C3	1.521(2)	1.529(2)	1.522(3)
C3–C4	1.523(2)	1.523(3)	1.521(3)
C4–C5	1.526(2)	1.534(2)	1.513(4)
C5–C6	1.510(2)	1.517(2)	1.510(4)
C1–O5	1.435(2)	1.430(2)	1.427(3)
C1–O1	1.390(3)	1.402(3)	1.412(2)
C2–O2	1.424(2)	1.421(2)	1.422(3)
C3–O3	1.421(2)	1.425(2)	1.422(4)
C4–O4	1.431(2)	1.430(2)	1.435(3)
C5–O5	1.434(2)	1.431(2)	1.451(3)
C6–O6	1.426(2)	1.424(2)	1.438(5)

Table 5. Angles determined in the low-temperature study of α-glucose (A) compared with those reported in the room-temperature neutron study of the same compound (B) and in the room-temperature X-ray study fo α-D-glucose hydrate (C).

Bonds	A	Bcor	C
C(1)–O(5)–C(5)	113.4(1)	113.7(1)	113.1(2)
O(5)–C(1)–O(1)	111.6(1)	111.5(1)	110.2(2)
O(5)–C(1)–C(2)	110.01(1)	110.1(1)	110.9(2)
O(1)–C(1)–C(2)	109.1(1)	109.3(1)	110.2(2)
O(2)–C(2)–C(1)	110.7(1)	110.9(1)	110.9(2)
O(2)–C(2)–C(3)	112.5(1)	112.2(1)	112.6(2)
C(1)–C(2)–C(3)	111.3(1)	111.1(1)	112.7(2)
O(3)–C(3)–C(2)	107.9(1)	108.1(1)	106.8(2)
O(3)–C(3)–C(4)	110.7(1)	110.6(1)	110.5(2)
C(2)–C(3)–C(4)	109.7(1)	109.8(1)	109.0(2)
O(4)–C(4)–C(3)	108.2(1)	108.2(2)	108.7(2)
O(4)–C(4)–C(5)	110.6(1)	110.9(1)	109.2(2)
C(3)–C(4)–C(5)	111.3(1)	111.1(1)	111.4(2)
O(5)–C(5)–C(4)	108.5(1)	108.7(1)	108.9(2)
O(5)–C(5)–C(6)	108.5(1)	108.1(1)	106.6(2)
C(4)–C(5)–C(6)	111.9(1)	111.5(1)	114.0(2)
O(6)–C(6)–C(5)	110.3(3)	109.9(1)	112.2(2)

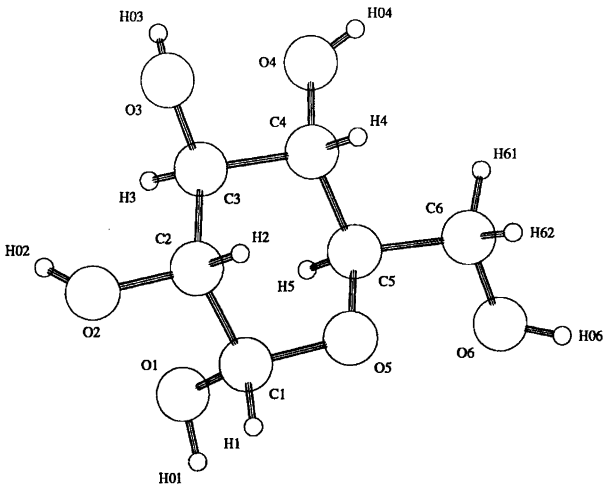

Fig. 1. Structure of α-D-glucose.

Table 6. Hydrogen bonding: A, X-ray data; B, neutron data; Δ, decrease in O---O distance as an effect of lowering the temperature. The O---O distances from the neutron study were not corrected for thermal motion and the actual Δ-values are probably somewhat greater.

Bond	Distance/Å		Distance/Å		Distance/Å		Angle/°		
O–H---O	O---O		O–H		H---O		O–H---O		
	A	B_{uncor}	A	B_{cor}	A	B_{uncor}	A	B	Δ/Å
O(1)–H---O(5)	2.833(1)	2.849(2)	0.89(3)	0.984(4)	1.99(2)	(1.915)	158.3(6)	160.9(3)	0.016
O(3)–H---O(2)	2.697(1)	2.708(2)	0.74(3)	0.975(3)	1.98(3)	(1.758)	162.1(6)	164.9(3)	0.011
O(2)–H---O(6)	2.759(1)	2.778(3)	0.77(5)	0.980(4)	1.84(2)	(1.821)	165.3(6)	170.1(3)	0.019
O(6)–H---O(3)	2.697(1)	2.714(3)	0.82(3)	0.977(4)	1.88(2)	(1.758)	171.8(6)	169.7(4)	0.018
O(4)–H---O(4)	2.763(1)	2.777(1)	0.74(3)	0.987(4)	2.08(3)	(1.819)	160.9(6)	167.7(3)	0.014

It is noteworthy that the difference in the two C–O bonds within the pyranose ring (C1–O5 and C5–O5) which is often reported[3] and shown in Table 4 for α-D-glucose monohydrate, is not present in either of the two studies of pure α-glucose.

Also for the angles, there is close agreement between the values found in the present and the neutron study, but a few significant differences when compared with the results from the study of α-D-glucose monohydrate. These differences may be a result of the difference in hydrogen bonding (Table 6).

The mean values of the C–H and O–H bonds are found in the X-ray study to be 1.02(2) Å and 0.81(3) Å, respectively, and shows the usual shortening compared with those from the neutron study which are 1.0986(3) for the C–H bonds and 0.981(4) for the O–H bonds.

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