Crystal Structure Information for UM # 1255

Prepared by: Peter Y. Zavalij Feb. 25, 2006

Crystal No. & ID	:	**1255**: M.M. sugar
Compound name	:	Sucrose
Chemical formula	:	$C_{12}H_{22}O_{11}$
Final R_1 [I>2σ(I)]	:	**2.43%**

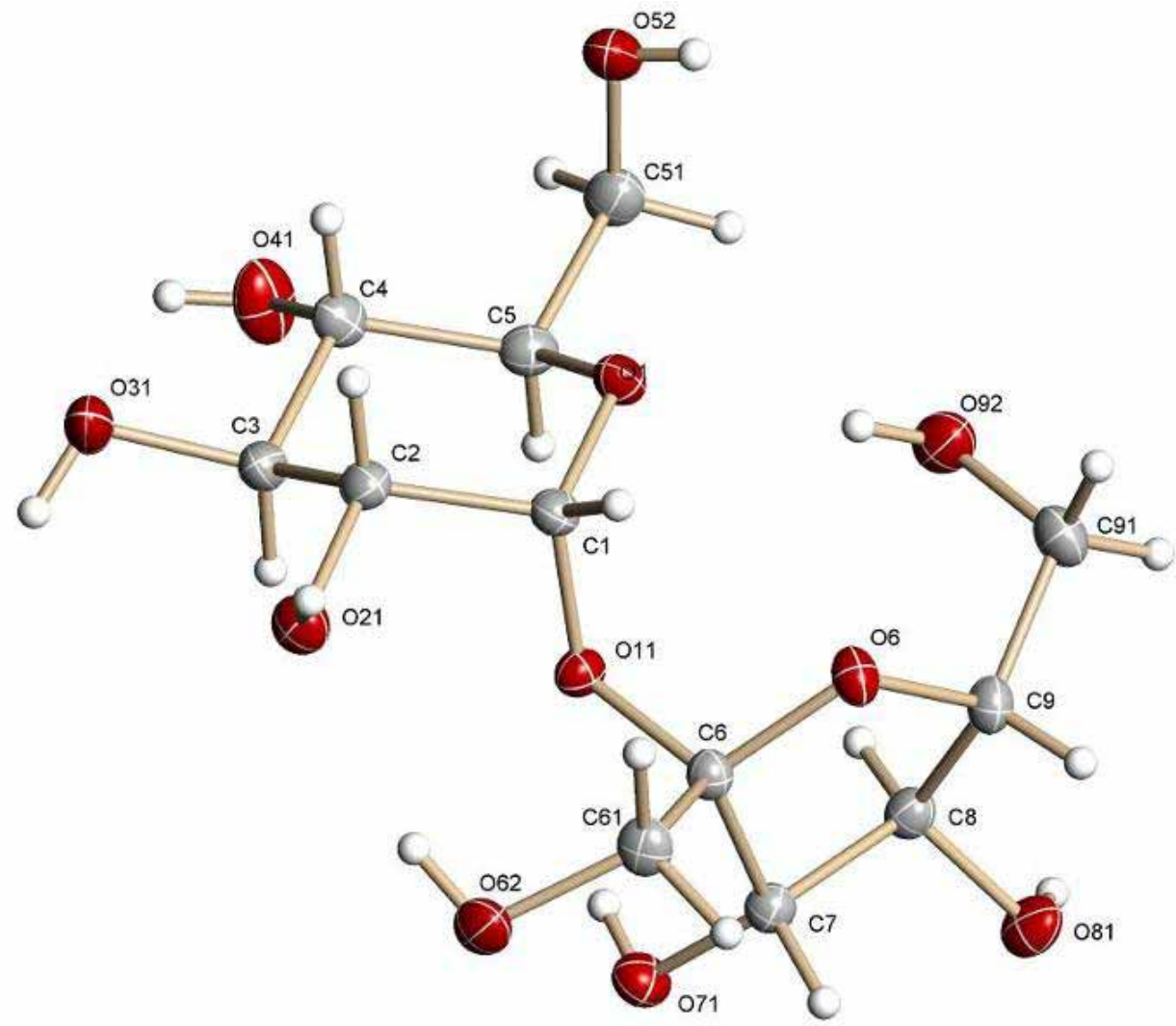


Figure 1. A view of sugar showing the numbering scheme employed. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 30% probability level. H atoms are displayed with an arbitrarily small radius.

A colorless prism of $C_{12}H_{22}O_{11}$, approximate dimensions 0.38 × 0.40 × 0.42 mm^3, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 223(2) K on a three-circle diffractometer system equipped with Bruker Smart1000 CCD area detector using a graphite monochromator and a MoKα fine-focus sealed tube (λ= 0.71073 Å) operated at 20 kV and 5 mA. The detector was placed at a distance of 4.99 cm from the crystal.

A total of 1150 frames were collected with a scan width of 0.5° in ω and an exposure time of 8 sec/frame using SMART (Bruker, 1999). The total data collection time was 4.8 hours. The frames were integrated with SAINT software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 6859 reflections to a maximum θ angle of 27.98°, of which 3324 were independent (completeness = 99.3%, R_{int} = 1.52%, R_{sig} = 1.76%) and 3274 were greater than 2σ(I). The final cell dimensions of a = 7.7441(5) Å, b = 8.6932(6) Å, c = 10.8537(7) Å, α= 90°, β= 102.9300(10)°, γ= 90°, V = 712.16(8) $Å^3$, are based upon the refinement of the XYZ-centroids of 5971 reflections with 2.7 < θ < 29.1° using SAINT. Analysis of the data showed -0.27 % decay during data collection. Data were corrected for absorption effects with the Semi-empirical from equivalents method using SADABS (Sheldrick, 1996). The minimum and maximum transmission coefficients were 0.875 and 0.947.

The structure was solved and refined using the SHELXS-97 (Sheldrick, 1990) and SHELXL-97 (Sheldrick, 1997) software in the space group $P2_1$ with Z = 2 for the formula unit $C_{12}H_{22}O_{11}$. The final anisotropic full-matrix least-squares refinement on F^2 with 276 variables converged at R_1 = 2.43 % for the observed data and wR_2 = 5.33 % for all data. The goodness-of-fit was 1.000. The largest peak on the final difference map was 0.282 ē/$Å^3$ and the largest hole was -0.152 ē/$Å^3$. On the basis of the final model, the calculated density was 1.596 g/cm^3 and F(000), 364 ē.

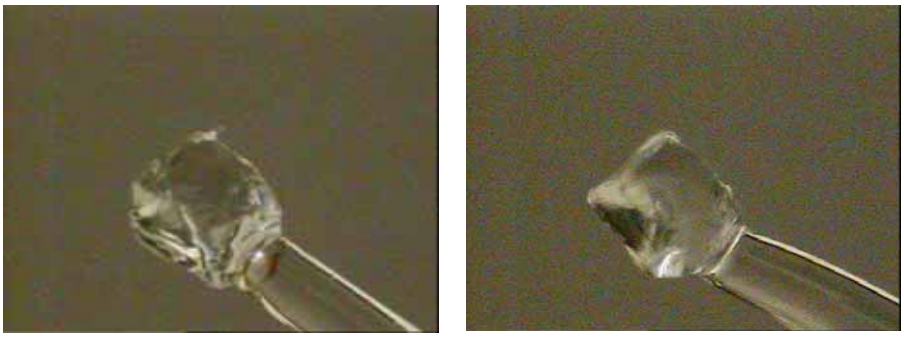
Overall structure quality considerations:

1. Strong data set, no disorder, R_1 4% maximum. Publishable quality.
2. Good data set, perhaps some minor disorder, R_1 6% maximum. Publishable quality.
3. Average data set and/or easily modelled disorder or twinning. Publishable with care.
4. Weak data and/or major disorder or twinning that is not easily modelled. Publishable in some cases.
5. Very weak data and/or unexplained features of data or model. Not of publishable quality.

A structure with a quality factor of 4 or 5 should not be used for a regulatory document without prior consultation.

Comments:

Data set quality	:	very good
Twinning	:	none
Disorder	:	none
H-atoms refinement	:	coordinates but not Uiso constrained
Residual density	:	at the middle of bonds
Structure quality	:	excellent
Check CIF	:	Platon
Publishable	:	No, known structure.



View of the crystal along two perpendicular directions

Table 1. Crystal data and structure refinement for sugar.

X-ray labbook No.	1255	
Crystal ID	M.M. sugar	
Empirical formula	C12 H22 O11	
Formula weight	342.3	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal size	0.42 × 0.40 × 0.38 mm^3	
Crystal habit	colorless prism	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 7.7441(5) Å	α = 90°
	b = 8.6932(6) Å	β = 102.930(1)°
	c = 10.8537(7) Å	γ = 90°
Volume	712.16(8) $Å^3$	
Z	2	
Density, ρ_{calc}	1.596 g/cm^3	
Absorption coefficient, μ	0.143 mm^{-1}	
F(000)	364 ē	
Diffractometer	Bruker Smart1000 CCD area detector	
Radiation source	fine-focus sealed tube, MoK_α	
Generator power	20 kV, 5 mA	
Detector distance	4.99 cm	
Detector resolution	8.33 pixels/mm	
Total frames	1150	
Frame size	512 pixels	
Frame width	0.5 °	
Exposure per frame	8 sec	
Total measurement time	4.8 hours	
Data collection method	ω scans	
θ range for data collection	2.70 to 27.98°	
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 11$, $-14 \le l \le 13$	
Reflections collected	6859	
Independent reflections	3324	
Observed reflection, I>2s(I)	3274	
Coverage of independent reflections	99.30%	
Variation in check reflections	-0.27%	
Absorption correction	Semi-empirical from equivalents	
	SADABS (Sheldrick, 1996)	
Max. and min. transmission	0.947 and 0.875	
Structure solution technique	direct	
Structure solution program	SHELXS-97 (Sheldrick, 1990)	
Refinement technique	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 1997)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	3324 / 1 / 276	
Goodness-of-fit on F^2	1	
D/smax	0	
Final R indices: R_1, I>2σ(I)	0.0243	
wR_2, all data	0.0533	
R_{int}	0.0152	
R_{sig}	0.0176	
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0093P)^2 + 0.3P]$,	
	$P = [\max(F_o^2, 0) + 2F_o^2]/3$	
Absolute structure parameter	0.2(5)	
Extinction coefficient	0.055(2)	
Largest diff. peak and hole	0.282 and -0.152 ē/$Å^3$	

$R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$

Table 2. Atomic coordinates and equivalent* displacement parameters (Å^2) for sugar.

Atom	***x/a***	***y/b***	***z/c***	$\mathbf{U_{eq}}$
O1	0.36842(12)	0.22565(10)	0.37782(8)	0.01881(19)
C1	0.48593(16)	0.26651(14)	0.29995(11)	0.0161(2)
C2	0.63682(17)	0.14925(15)	0.31305(12)	0.0180(2)
O21	0.74897(13)	0.18838(12)	0.22894(9)	0.0227(2)
C3	0.56508(17)	-0.01297(15)	0.28551(12)	0.0194(3)
O31	0.70425(15)	-0.12470(13)	0.30889(10)	0.0292(2)
C4	0.44331(18)	-0.04735(15)	0.37476(12)	0.0204(3)
O41	0.35526(16)	-0.19069(12)	0.34880(12)	0.0339(3)
C5	0.29546(17)	0.07205(15)	0.35968(12)	0.0193(2)
C51	0.18465(18)	0.05284(17)	0.45849(13)	0.0240(3)
O52	0.28694(15)	0.07850(13)	0.58253(9)	0.0274(2)
O11	0.39148(11)	0.27659(10)	0.17102(8)	0.01626(18)
C6	0.36988(15)	0.43172(14)	0.12389(11)	0.0157(2)
C61	0.54506(16)	0.49276(15)	0.10252(13)	0.0199(3)
O62	0.62176(12)	0.38698(13)	0.02884(9)	0.0232(2)
C7	0.21435(16)	0.43410(15)	0.00673(11)	0.0166(2)
O71	0.20341(12)	0.30637(12)	-0.07509(9)	0.0211(2)
C8	0.05494(16)	0.45829(15)	0.06515(12)	0.0176(2)
O81	-0.09000(12)	0.52866(13)	-0.02157(9)	0.0238(2)
O6	0.31752(11)	0.52989(11)	0.21236(8)	0.01868(19)
C9	0.12882(16)	0.56372(15)	0.17671(12)	0.0182(2)
C91	0.04796(18)	0.54302(17)	0.29011(13)	0.0233(3)
O92	0.04017(13)	0.38555(13)	0.32689(10)	0.0262(2)
H11	0.534(2)	0.363(2)	0.3309(15)	0.019
H21	0.707(2)	0.1536(19)	0.3994(16)	0.022
H211	0.833(3)	0.245(2)	0.2680(17)	0.034
H31	0.499(2)	-0.019(2)	0.1989(16)	0.023
H311	0.738(3)	-0.139(2)	0.244(2)	0.044
H41	0.515(2)	-0.043(2)	0.4592(15)	0.025
H411	0.422(3)	-0.252(3)	0.348(2)	0.051
H51	0.222(2)	0.061(2)	0.2777(15)	0.023
H511	0.140(2)	-0.052(2)	0.4497(17)	0.029
H512	0.084(2)	0.120(2)	0.4386(17)	0.029
H521	0.283(3)	0.164(3)	0.600(2)	0.041
H611	0.524(2)	0.592(2)	0.0555(15)	0.024
H612	0.622(2)	0.511(2)	0.1846(15)	0.024
H621	0.655(2)	0.312(2)	0.0755(18)	0.035
H71	0.227(2)	0.526(2)	-0.0439(14)	0.02
H711	0.183(2)	0.232(2)	-0.0367(17)	0.032
H81	0.021(2)	0.363(2)	0.0946(15)	0.021
H811	-0.177(3)	0.486(3)	-0.0154(19)	0.043(6)
H91	0.114(2)	0.668(2)	0.1530(15)	0.022
H911	-0.073(2)	0.576(2)	0.2698(16)	0.028
H912	0.114(2)	0.606(2)	0.3633(16)	0.028
H921	0.143(3)	0.354(2)	0.3444(18)	0.039

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3. Anisotropic atomic displacement parameters * (Å^2) for sugar.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0221(4)	0.0174(4)	0.0189(4)	-0.0001(3)	0.0086(4)	-0.0006(3)
C1	0.0174(5)	0.0164(6)	0.0149(5)	-0.0007(4)	0.0043(4)	-0.0004(4)
C2	0.0176(6)	0.0195(6)	0.0169(5)	0.0015(5)	0.0035(4)	0.0017(5)
O21	0.0183(4)	0.0257(5)	0.0255(5)	0.0011(4)	0.0080(4)	-0.0003(4)
C3	0.0239(6)	0.0177(6)	0.0176(6)	0.0006(5)	0.0065(5)	0.0028(5)
O31	0.0395(6)	0.0241(5)	0.0286(5)	0.0068(4)	0.0171(5)	0.0138(4)
C4	0.0256(6)	0.0154(6)	0.0217(6)	-0.0006(5)	0.0083(5)	-0.0017(5)
O41	0.0370(6)	0.0164(5)	0.0524(7)	-0.0045(5)	0.0190(5)	-0.0053(4)
C5	0.0204(6)	0.0191(6)	0.0183(6)	0.0010(5)	0.0045(5)	-0.0033(5)
C51	0.0223(6)	0.0254(7)	0.0264(6)	0.0048(5)	0.0099(5)	0.0003(5)
O52	0.0364(6)	0.0270(5)	0.0216(4)	0.0037(4)	0.0124(4)	0.0059(5)
O11	0.0179(4)	0.0136(4)	0.0167(4)	0.0007(3)	0.0025(3)	-0.0011(3)
C6	0.0148(5)	0.0148(6)	0.0178(5)	-0.0001(5)	0.0047(4)	0.0001(4)
C61	0.0150(5)	0.0203(6)	0.0255(6)	0.0010(5)	0.0067(5)	-0.0027(5)
O62	0.0197(4)	0.0281(5)	0.0246(5)	0.0037(4)	0.0104(4)	0.0021(4)
C7	0.0162(5)	0.0165(6)	0.0177(5)	0.0009(5)	0.0046(4)	-0.0009(4)
O71	0.0241(5)	0.0205(5)	0.0189(4)	-0.0030(4)	0.0051(4)	-0.0023(4)
C8	0.0149(5)	0.0183(6)	0.0197(6)	0.0027(5)	0.0043(4)	0.0005(5)
O81	0.0140(4)	0.0292(5)	0.0272(5)	0.0056(4)	0.0022(4)	0.0016(4)
O6	0.0147(4)	0.0196(4)	0.0213(4)	-0.0052(3)	0.0032(3)	0.0011(3)
C9	0.0151(6)	0.0169(6)	0.0228(6)	0.0001(5)	0.0049(5)	0.0020(4)
C91	0.0210(6)	0.0261(7)	0.0249(6)	-0.0030(5)	0.0097(5)	0.0013(5)
O92	0.0215(5)	0.0298(5)	0.0290(5)	0.0054(4)	0.0093(4)	0.0009(4)

* The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

Table 4. Bond lengths (Å), valence and torsion angles (°) for sugar.

O1-C1	1.4188(14)	O1-C5	1.4460(16)	C1-O11	1.4293(14)
C1-C2	1.5331(17)	C1-H11	0.951(17)	C2-O21	1.4347(16)
C2-C3	1.5206(18)	C2-H21	0.974(16)	O21-H211	0.85(2)
C3-O31	1.4307(16)	C3-C4	1.5251(17)	C3-H31	0.965(16)
O31-H311	0.81(2)	C4-O41	1.4181(17)	C4-C5	1.5269(18)
C4-H41	0.961(16)	O41-H411	0.75(2)	C5-C51	1.5252(17)
C5-H51	0.948(16)	C51-O52	1.4186(17)	C51-H511	0.977(19)
C51-H512	0.958(19)	O52-H521	0.77(2)	O11-C6	1.4388(15)
C6-O6	1.4104(14)	C6-C61	1.5221(16)	C6-C7	1.5436(16)
C61-O62	1.4319(16)	C61-H611	0.995(17)	C61-H612	0.968(16)
O62-H621	0.83(2)	C7-O71	1.4124(15)	C7-C8	1.5233(17)
C7-H71	0.987(17)	O71-H711	0.80(2)	C8-O81	1.4308(15)
C8-C9	1.5243(18)	C8-H81	0.950(17)	O81-H811	0.78(2)
O6-C9	1.4555(14)	C9-C91	1.5114(17)	C9-H91	0.943(18)
C91-O92	1.4310(18)	C91-H911	0.958(18)	C91-H912	1.007(18)
O92-H921	0.83(2)				
C1-O1-C5	115.67(9)	O1-C1-O11	110.04(9)	O1-C1-C2	110.82(10)
O11-C1-C2	110.12(10)	O1-C1-H11	105.2(9)	O11-C1-H11	111.4(10)
C2-C1-H11	109.2(9)	O21-C2-C3	109.96(10)	O21-C2-C1	109.92(10)
C3-C2-C1	111.16(10)	O21-C2-H21	108.4(10)	C3-C2-H21	109.1(10)
C1-C2-H21	108.3(10)	C2-O21-H211	108.9(12)	O31-C3-C2	111.54(11)
O31-C3-C4	107.62(10)	C2-C3-C4	107.91(10)	O31-C3-H31	110.5(10)
C2-C3-H31	109.2(10)	C4-C3-H31	110.0(9)	C3-O31-H311	109.1(15)
O41-C4-C3	112.59(11)	O41-C4-C5	105.09(11)	C3-C4-C5	110.74(11)
O41-C4-H41	112.2(10)	C3-C4-H41	106.8(9)	C5-C4-H41	109.4(10)
C4-O41-H411	109.1(18)	O1-C5-C51	105.63(11)	O1-C5-C4	110.63(10)
C51-C5-C4	112.12(11)	O1-C5-H51	110.9(10)	C51-C5-H51	109.5(10)
C4-C5-H51	108.1(10)	O52-C51-C5	111.57(11)	O52-C51-H511	110.6(11)
C5-C51-H511	106.2(10)	O52-C51-H512	111.7(11)	C5-C51-H512	109.3(11)
H511-C51-H512	107.3(14)	C51-O52-H521	110.0(16)	C1-O11-C6	113.51(9)
O6-C6-O11	110.69(9)	O6-C6-C61	107.14(10)	O11-C6-C61	110.13(10)
O6-C6-C7	105.23(9)	O11-C6-C7	108.22(10)	C61-C6-C7	115.31(10)
O62-C61-C6	110.88(10)	O62-C61-H611	108.1(10)	C6-C61-H611	109.2(10)
O62-C61-H612	111.7(10)	C6-C61-H612	107.7(9)	H611-C61-H612	109.2(14)
C61-O62-H621	105.8(13)	O71-C7-C8	115.47(10)	O71-C7-C6	115.68(10)
C8-C7-C6	102.39(9)	O71-C7-H71	106.7(9)	C8-C7-H71	108.0(9)
C6-C7-H71	108.3(9)	C7-O71-H711	107.2(14)	O81-C8-C7	112.01(10)
O81-C8-C9	111.42(11)	C7-C8-C9	102.62(10)	O81-C8-H81	111.1(10)
C7-C8-H81	109.4(10)	C9-C8-H81	110.0(10)	C8-O81-H811	107.8(15)
C6-O6-C9	111.45(9)	O6-C9-C91	109.49(10)	O6-C9-C8	105.41(10)
C91-C9-C8	115.15(11)	O6-C9-H91	108.8(10)	C91-C9-H91	106.7(10)
C8-C9-H91	111.1(10)	O92-C91-C9	113.08(11)	O92-C91-H911	104.3(11)
C9-C91-H911	109.9(10)	O92-C91-H912	110.4(10)	C9-C91-H912	110.3(10)
H911-C91-H912	108.6(14)	C91-O92-H921	106.5(14)		
C5-O1-C1-O11	67.51(12)	C5-O1-C1-C2	-54.53(13)	O1-C1-C2-O21	177.12(10)
O11-C1-C2-O21	55.13(13)	O1-C1-C2-C3	55.14(13)	O11-C1-C2-C3	-66.85(13)
O21-C2-C3-O31	63.45(13)	C1-C2-C3-O31	-174.59(10)	O21-C2-C3-C4	-178.54(10)
C1-C2-C3-C4	-56.59(13)	O31-C3-C4-O41	-65.28(14)	C2-C3-C4-O41	174.22(11)
O31-C3-C4-C5	177.39(11)	C2-C3-C4-C5	56.89(13)	C1-O1-C5-C51	176.52(10)

C1-O1-C5-C4	54.97(13)	O41-C4-C5-O1	-177.20(10)	C3-C4-C5-O1	-55.36(13)
O41-C4-C5-C51	65.17(14)	C3-C4-C5-C51	-173.00(11)	O1-C5-C51-O52	-56.70(14)
C4-C5-C51-O52	63.88(15)	O1-C1-O11-C6	108.32(11)	C2-C1-O11-C6	-129.23(10)
C1-O11-C6-O6	-44.83(12)	C1-O11-C6-C61	73.46(12)	C1-O11-C6-C7	-159.66(9)
O6-C6-C61-O62	171.14(10)	O11-C6-C61-O62	50.68(13)	C7-C6-C61-O62	-72.13(14)
O6-C6-C7-O71	-157.61(9)	O11-C6-C7-O71	-39.24(13)	C61-C6-C7-O71	84.58(13)
O6-C6-C7-C8	-31.15(12)	O11-C6-C7-C8	87.21(11)	C61-C6-C7-C8	-148.97(11)
O71-C7-C8-O81	-78.53(14)	C6-C7-C8-O81	154.88(10)	O71-C7-C8-C9	161.85(10)
C6-C7-C8-C9	35.26(12)	O11-C6-O6-C9	-102.24(11)	C61-C6-O6-C9	137.66(10)
C7-C6-O6-C9	14.45(12)	C6-O6-C9-C91	132.70(11)	C6-O6-C9-C8	8.28(13)
O81-C8-C9-O6	-147.55(10)	C7-C8-C9-O6	-27.53(12)	O81-C8-C9-C91	91.66(13)
C7-C8-C9-C91	-148.32(11)	O6-C9-C91-O92	-70.06(14)	C8-C9-C91-O92	48.48(16)

Table 6. Hydrogen bond information for sugar (Å and °).

D—H…A*	**d(D—H)**	**d(H…A)**	**d(D…A)**	**∠(DHA)**
O21—H211…O92^{I}	0.85(2)	2.01(2)	2.8427(15)	168.8(17)
O31—H311…O71^{II}	0.81(2)	2.04(2)	2.8499(14)	173(2)
O41—H411…O6^{III}	0.75(2)	2.42(2)	2.8257(14)	115(2)
O52—H521…O31^{IV}	0.77(2)	2.07(2)	2.8310(16)	166(2)
O62—H621…O21	0.83(2)	1.98(2)	2.7768(14)	160.9(19)
O71—H711…O81^{V}	0.80(2)	2.07(2)	2.8483(15)	166.1(18)
O81—H811…O62^{VI}	0.78(2)	1.93(2)	2.7102(14)	171(2)
O92—H921…O1	0.83(2)	2.03(2)	2.8409(14)	166(2)

* D - donor atom, H - hydrogen, A - acceptor.

Symmetry codes: (I) x+1,y,z; (II) -x+1,y-1/2,-z; (III) x,y-1,z; (IV) -x+1,y+1/2,-z+1; (V) -x,y-1/2,-z; (VI) x-1,y,z