X-ray Diffraction Laboratory
Center for Chemical Characterization and Analysis
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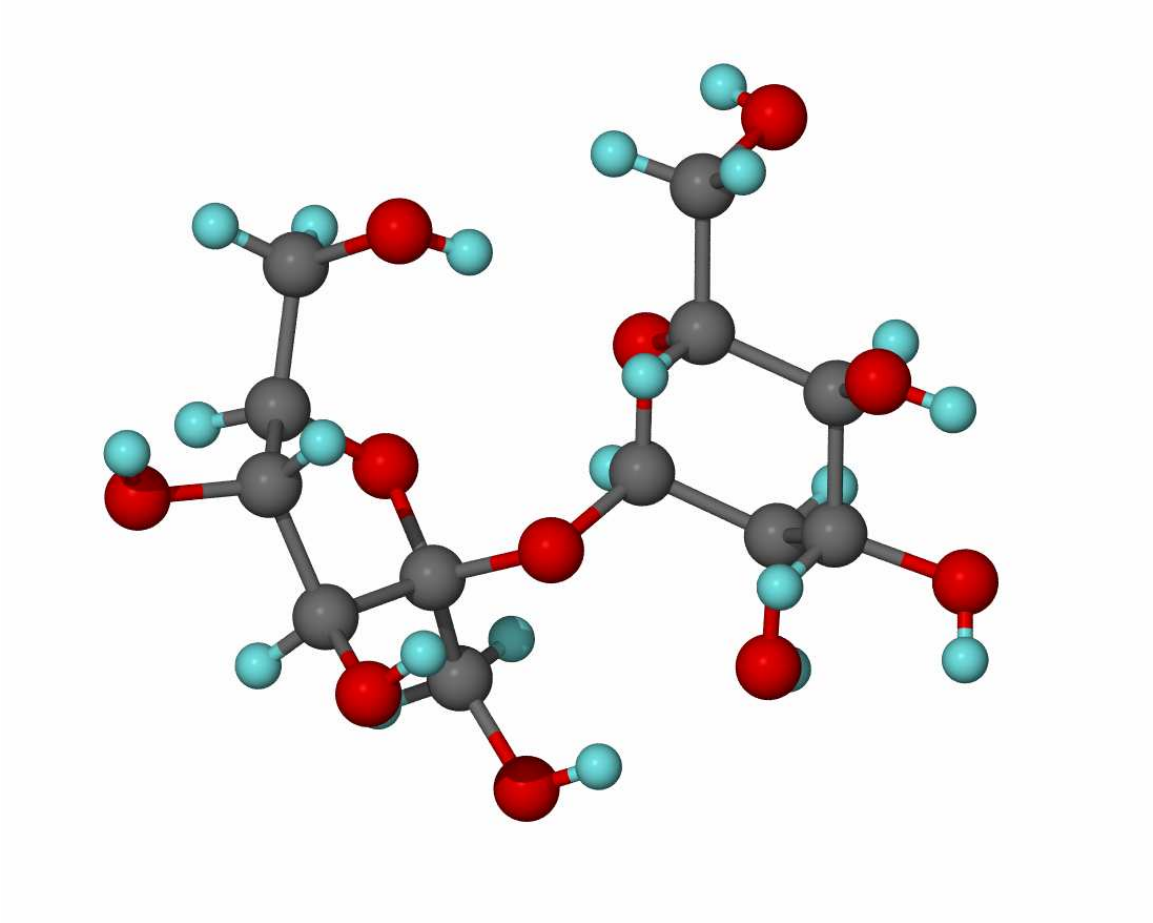
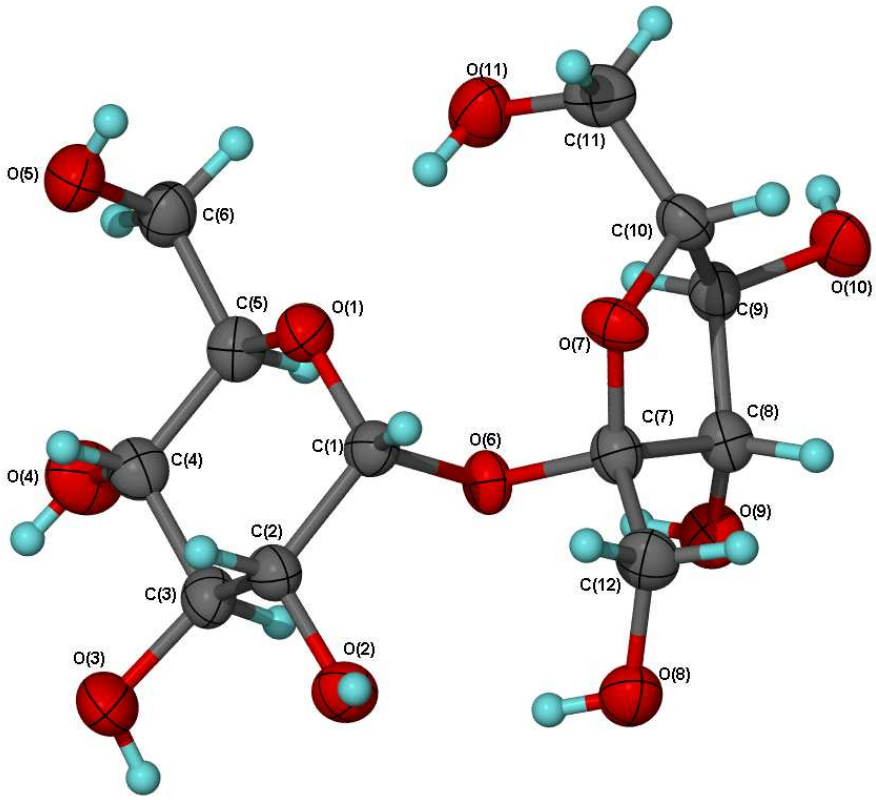
Structure Report for *J. Reibenspies*



Project Name: Sucrose
Date: January 29, 2007


O(11)
C(11)
O(5)
C(6)
C(10)
O(10)
C(5)
O(1)
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C(12)
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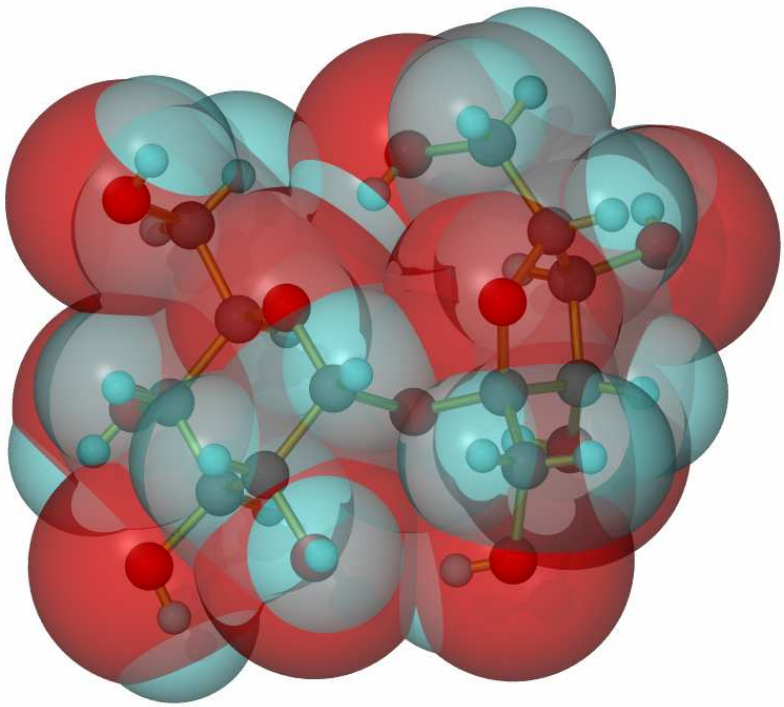

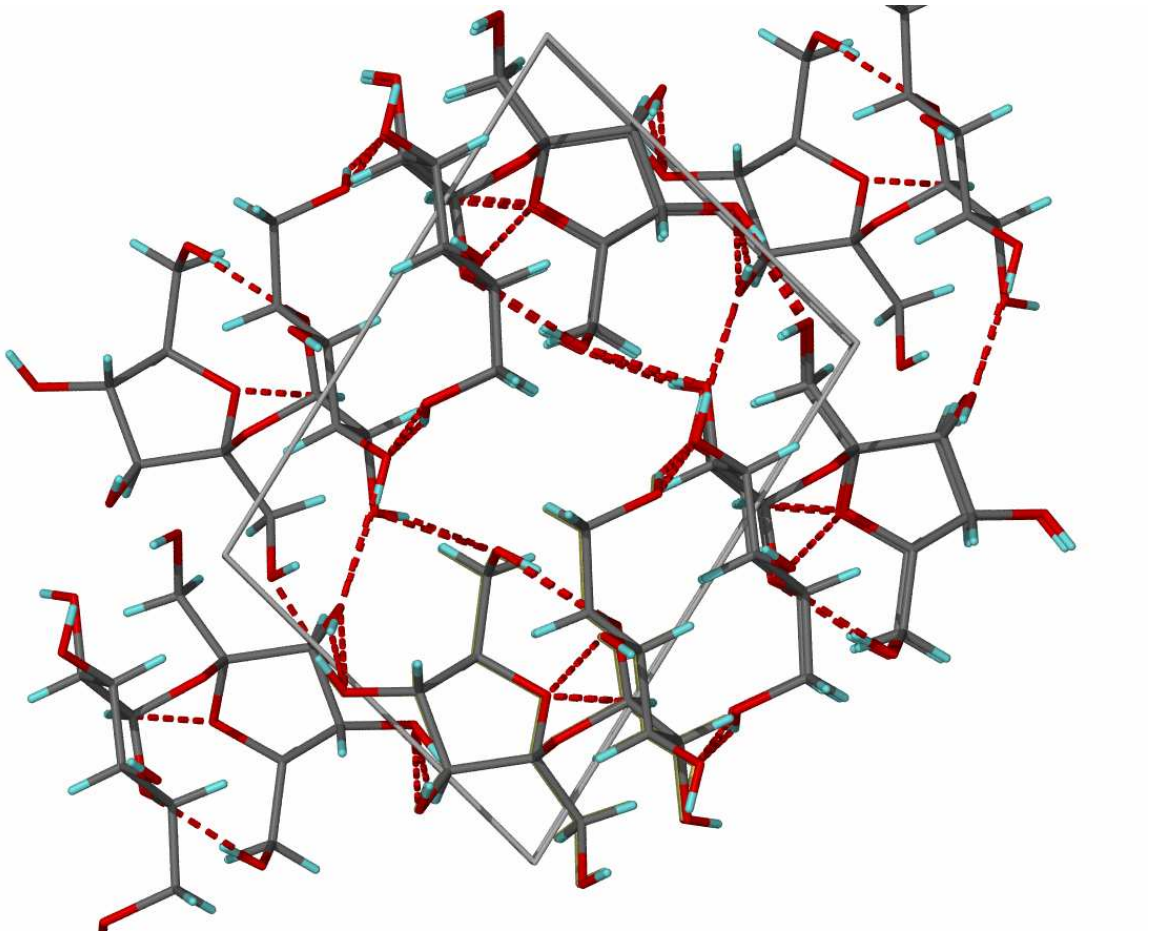

Experimental Write-up

A Leica MZ7 polarizing microscope was used to identify a suitable specimen from a representative sampling of materials. The was then fixed to a nylon loop which in turn was fashioned to a copper mounting pin. The mounted powder was then placed in a cold nitrogen stream (Oxford) maintained at 110K.

A BRUKER D8 GADDS general purpose three-circle X-ray diffractometer was employed for sample screening and data collection. The goniometer was controlled using the GADDS software suite (Microsoft Win 2000 operating system). The sample was optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions. The detector was set at 5.0cm from the crystal sample (MWPC Hi-Star Detector, 512x512 pixel). The X-ray radiation employed was generated from a Cu sealed X-ray tube (K_α = 1.54184Å with a potential of 40 kV and a current of 40 mA) and filtered with a graphite monochromator in the parallel mode (175 mm collimator with 0.5 mm pinholes).

A rotation exposure was taken to determine crystal quality and the X-ray beam intersection with the detector. The beam intersection coordinates were compared to the configured coordinates and changes were made accordingly. The rotation exposure indicated acceptable crystal quality and the unit cell determination was undertaken. Sixty data frames were taken at widths of 0.5° with an exposure time of 10 seconds. Over 200 reflections were centered and their positions were determined. These reflections were used in the auto-indexing procedure to determine the unit cell. A suitable cell was found and refined by nonlinear least squares and Bravais lattice procedures and reported here in Table 1. The unit cell was verified by examination of the hkl overlays on several frames of data, including zone photographs. No super-cell or erroneous reflections were observed.

After careful examination of the unit cell, a standard data collection procedure was initiated. This procedure consists of collection of one hemisphere of data collected using omega scans, involving the collection 2520 0.5° frames at fixed angles for ϕ, 2θ, and χ (2θ = -28°, χ = 54.73°, 2θ = -90°, χ = 54.73°), while varying omega. Addition data frames were collected to complete the data set. Each frame was exposed for 10 sec. The total data collection was performed for duration of approximately 24 hours at 110 K. No significant intensity fluctuations of equivalent reflections were observed.

After data collection, the crystal was measured carefully for size, morphology and color. These measurements are reported in Table 1.

Table 1. Crystal data and structure refinement for suc_omega.

Identification code	Sucrose	
Empirical formula	C12 H22 O11	
Formula weight	342.30	
Temperature	571(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 7.7553(4) Å	α= 90°.
	b = 8.7046(4) Å	β= 102.942(3)°.
	c = 10.8644(5) Å	γ = 90°.
Volume	714.79(6) $Å^3$	
Z	2	
Density (calculated)	1.590 Mg/m^3	
Absorption coefficient	1.242 mm^{-1}	
F(000)	364	
Crystal size	0.20 x 0.20 x 0.10 mm^3	
Theta range for data collection	4.18 to 58.91°.	
Index ranges	-8<=h<=7, -9<=k<=9, -12<=l<=11	
Reflections collected	3585	
Independent reflections	1926 [R(int) = 0.0469]	
Completeness to theta = 58.91°	95.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8859 and 0.7893	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1926 / 1 / 216	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0482, wR2 = 0.1230	
R indices (all data)	R1 = 0.0528, wR2 = 0.1361	
Absolute structure parameter	0.4(4)	
Largest diff. peak and hole	0.309 and -0.346 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for suc_omega. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3684(4)	9401(3)	8771(2)	25(1)
O(2)	7477(4)	9024(4)	7293(3)	31(1)
O(3)	7029(5)	5898(4)	8089(3)	42(1)
O(4)	3555(5)	5246(4)	8484(4)	49(1)
O(5)	2864(4)	7927(4)	10815(3)	39(1)
O(6)	3913(3)	9905(3)	6711(2)	23(1)
O(7)	3165(3)	12441(3)	7122(3)	25(1)
O(8)	6209(4)	11029(4)	5299(3)	33(1)
O(9)	2044(4)	10206(4)	4260(3)	30(1)
O(10)	-880(4)	12409(4)	4777(3)	36(1)
O(11)	392(4)	10999(4)	8263(3)	38(1)
C(1)	4863(5)	9805(5)	8002(3)	20(1)
C(2)	6367(5)	8635(5)	8134(4)	24(1)
C(3)	5649(6)	7020(5)	7859(4)	27(1)
C(4)	4415(6)	6678(5)	8738(4)	28(1)
C(5)	2953(5)	7864(5)	8606(4)	27(1)
C(6)	1830(5)	7671(6)	9579(4)	34(1)
C(7)	3687(5)	11466(5)	6245(4)	21(1)
C(8)	2134(5)	11483(5)	5061(4)	22(1)
C(9)	552(5)	11711(5)	5652(4)	27(1)
C(10)	1288(5)	12762(5)	6772(4)	24(1)
C(11)	5420(5)	12063(5)	6026(4)	28(1)
C(12)	467(6)	12572(5)	7890(4)	33(1)

Table 3. Bond lengths [Å] and angles [°] for suc_omega.

O(1)-C(1)	1.414(5)
O(1)-C(5)	1.449(5)
O(2)-C(2)	1.429(5)
O(3)-C(3)	1.429(5)
O(4)-C(4)	1.411(6)
O(5)-C(6)	1.418(5)
O(6)-C(1)	1.433(4)
O(6)-C(7)	1.447(5)
O(7)-C(7)	1.401(5)
O(7)-C(10)	1.447(4)
O(8)-C(11)	1.424(6)
O(9)-C(8)	1.404(5)
O(10)-C(9)	1.426(5)
O(11)-C(12)	1.433(6)
C(1)-C(2)	1.530(5)
C(2)-C(3)	1.517(6)
C(3)-C(4)	1.526(6)
C(4)-C(5)	1.516(6)
C(5)-C(6)	1.522(6)
C(7)-C(11)	1.509(6)
C(7)-C(8)	1.553(5)
C(8)-C(9)	1.520(6)
C(9)-C(10)	1.527(6)
C(10)-C(12)	1.502(6)

C(1)-O(1)-C(5)	116.3(3)
C(1)-O(6)-C(7)	113.2(3)
C(7)-O(7)-C(10)	111.4(3)
O(1)-C(1)-O(6)	109.5(3)
O(1)-C(1)-C(2)	111.0(3)
O(6)-C(1)-C(2)	110.3(3)
O(2)-C(2)-C(3)	109.9(3)
O(2)-C(2)-C(1)	109.7(3)
C(3)-C(2)-C(1)	111.1(3)

O(3)-C(3)-C(2)	111.8(3)
O(3)-C(3)-C(4)	107.9(4)
C(2)-C(3)-C(4)	108.1(3)
O(4)-C(4)-C(5)	105.8(4)
O(4)-C(4)-C(3)	112.6(4)
C(5)-C(4)-C(3)	111.6(4)
O(1)-C(5)-C(4)	110.7(3)
O(1)-C(5)-C(6)	106.5(4)
C(4)-C(5)-C(6)	113.0(4)
O(5)-C(6)-C(5)	110.5(3)
O(7)-C(7)-O(6)	111.2(3)
O(7)-C(7)-C(11)	107.6(3)
O(6)-C(7)-C(11)	109.6(3)
O(7)-C(7)-C(8)	105.6(3)
O(6)-C(7)-C(8)	108.0(3)
C(11)-C(7)-C(8)	114.7(3)
O(9)-C(8)-C(9)	115.6(3)
O(9)-C(8)-C(7)	115.3(3)
C(9)-C(8)-C(7)	101.6(3)
O(10)-C(9)-C(8)	110.9(3)
O(10)-C(9)-C(10)	111.9(4)
C(8)-C(9)-C(10)	102.8(3)
O(7)-C(10)-C(12)	110.5(3)
O(7)-C(10)-C(9)	105.5(3)
C(12)-C(10)-C(9)	115.4(4)
O(8)-C(11)-C(7)	112.3(4)
O(11)-C(12)-C(10)	112.7(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for suc_omega. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	22(2)	27(2)	28(2)	1(1)	8(1)	-1(1)
O(2)	13(2)	42(2)	37(2)	3(1)	7(1)	1(1)
O(3)	49(2)	38(2)	43(2)	10(2)	20(2)	21(2)
O(4)	47(2)	28(2)	76(3)	-2(2)	26(2)	-9(2)
O(5)	39(2)	44(2)	35(2)	6(1)	13(2)	6(2)
O(6)	15(2)	25(2)	26(2)	5(1)	-1(1)	2(1)
O(7)	16(2)	26(2)	32(2)	-8(1)	1(1)	4(1)
O(8)	23(2)	43(2)	37(2)	4(2)	13(1)	-2(1)
O(9)	29(2)	29(2)	31(2)	-4(1)	4(1)	-3(1)
O(10)	15(2)	50(2)	39(2)	11(2)	-2(1)	1(1)
O(11)	28(2)	43(2)	45(2)	11(2)	12(2)	4(1)
C(1)	11(2)	26(2)	22(2)	1(2)	-1(2)	0(2)
C(2)	13(2)	29(2)	27(2)	1(2)	2(2)	3(2)
C(3)	25(2)	30(3)	26(2)	2(2)	6(2)	8(2)
C(4)	27(2)	22(2)	34(2)	-5(2)	5(2)	-2(2)
C(5)	24(3)	25(2)	31(2)	0(2)	5(2)	-5(2)
C(6)	26(3)	40(3)	36(3)	6(2)	8(2)	3(2)
C(7)	12(2)	24(2)	25(2)	4(2)	3(2)	2(2)
C(8)	20(2)	23(2)	24(2)	1(2)	6(2)	-4(2)
C(9)	22(2)	28(2)	31(2)	8(2)	7(2)	4(2)
C(10)	30(2)	30(3)	38(2)	0(2)	4(2)	1(2)
C(11)	15(2)	31(3)	37(2)	2(2)	6(2)	-6(2)
C(12)	19(2)	41(3)	43(3)	-6(2)	13(2)	0(2)