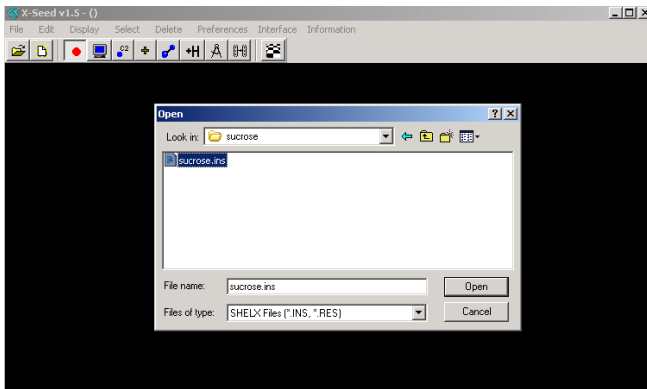


X-seed Practical

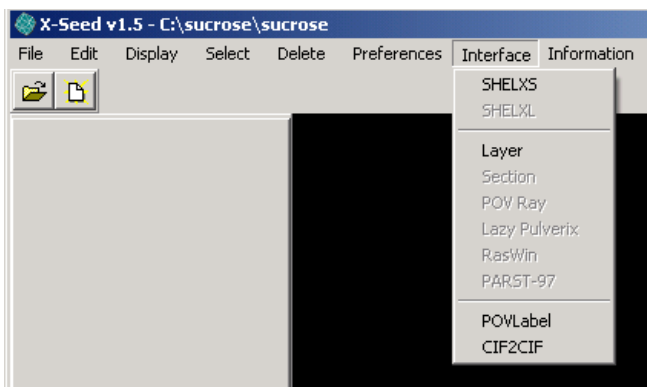
A LAPTOP university course

J. Reibenspies

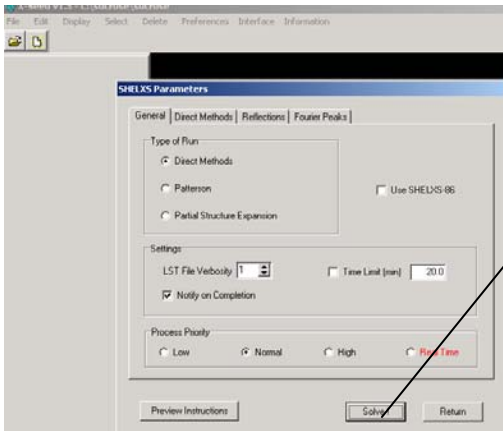
Start the program X-SEED and open “sucrose.ins”.



Point to interface and then SHELXS

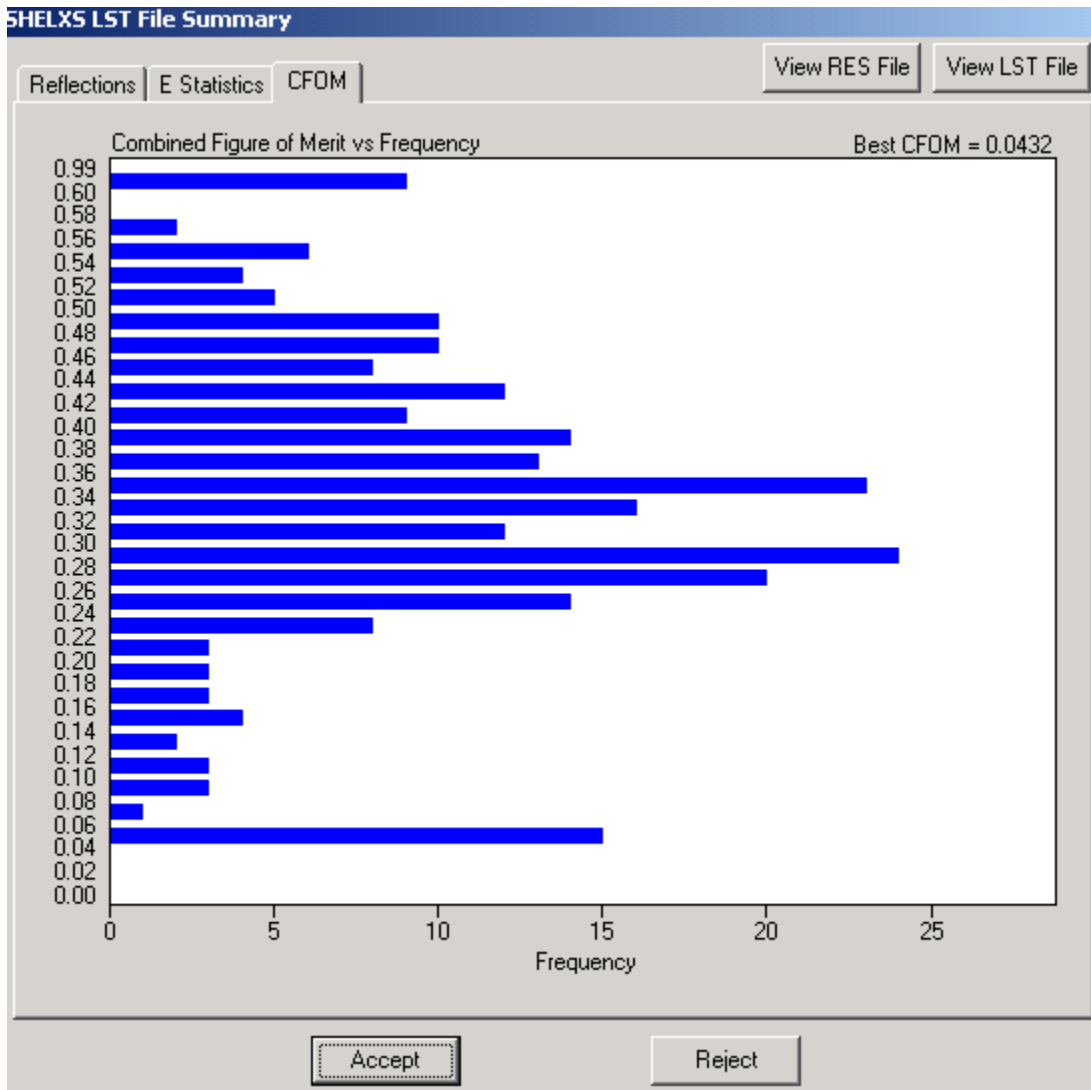


You will see the Direct Methods radio button checked, with a low output verbosity (summarized results) and normal priority on the processing. These are the defaults and are good for the first run. You should only change these settings if you cannot solve the structure on the first run.

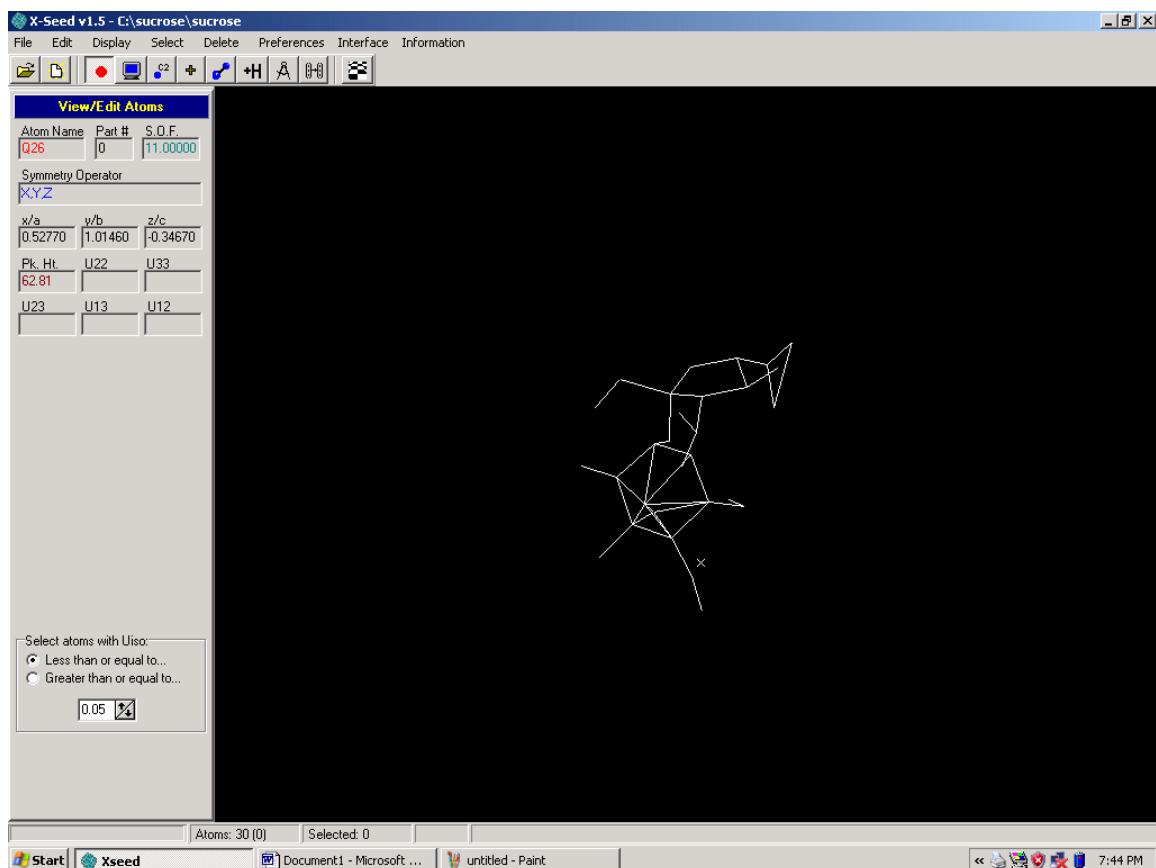


Click here to start

A SHELXS window will open and you will see a summary of the programs progress. When the SHELXS program is finished the window will close and a summary window will open. Most of this may make no sense to you for now so go straight to the CFOM tab. The smaller the CFOM number the better the result. Notice that at about 0.04 CFOM there is a high frequency of hits. This is a good sign that the structure is solved.

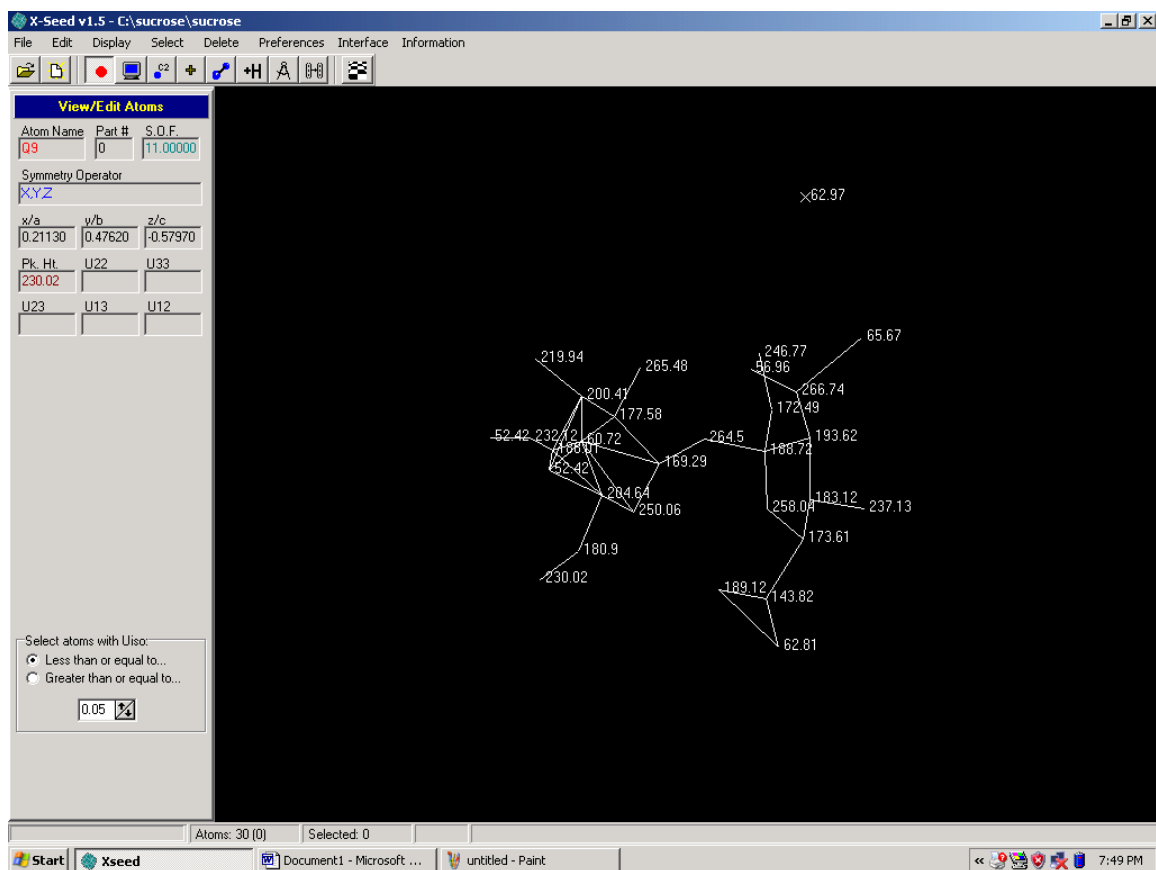


Point to the Accept button and continue. You should see something like this



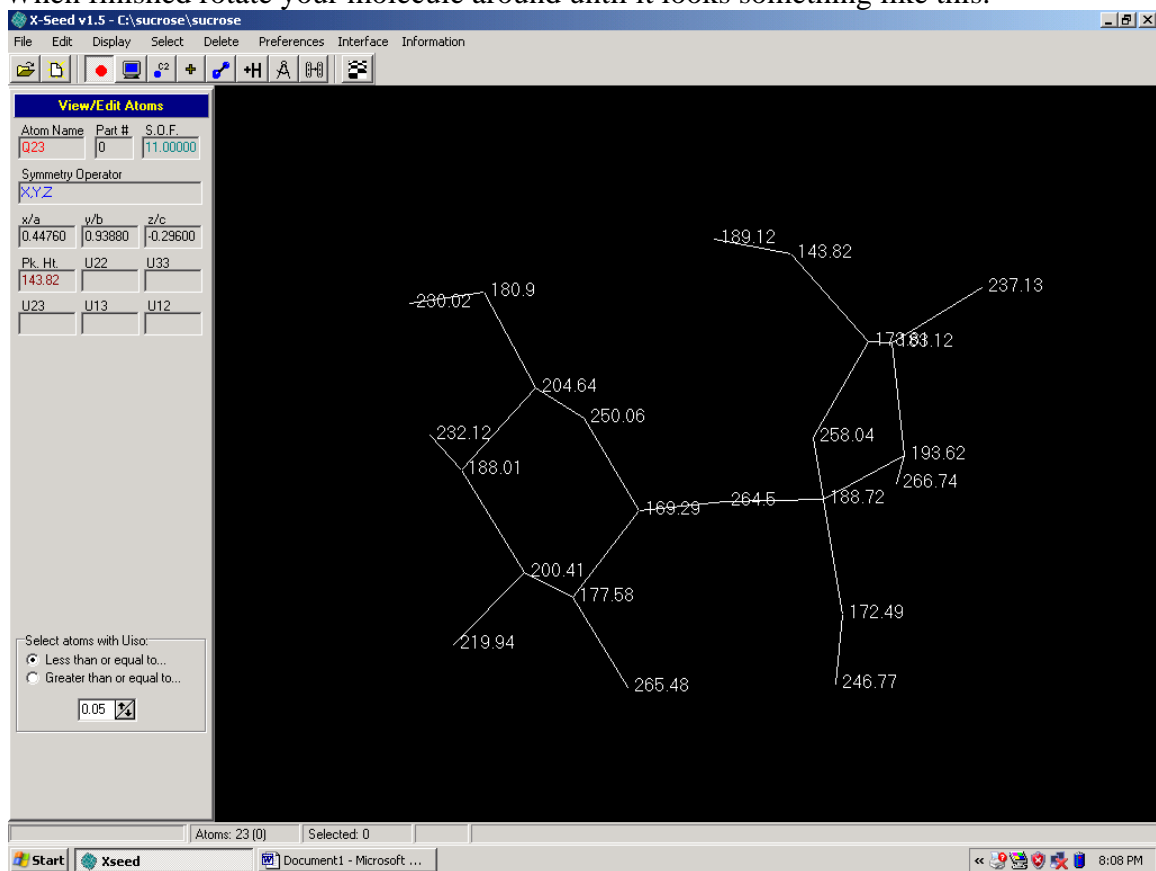
Hold the left mouse button down and move the mouse. Now hold the right mouse button down and move the mouse. Note what happens. Try the cntl and shift buttons with the mouse movement. Experiment until you feel comfortable and you know how to move and rotate the molecule.

Put labels on the peak positions by pointing to display atom labels. What you will see are numbers that display the peak intensities.

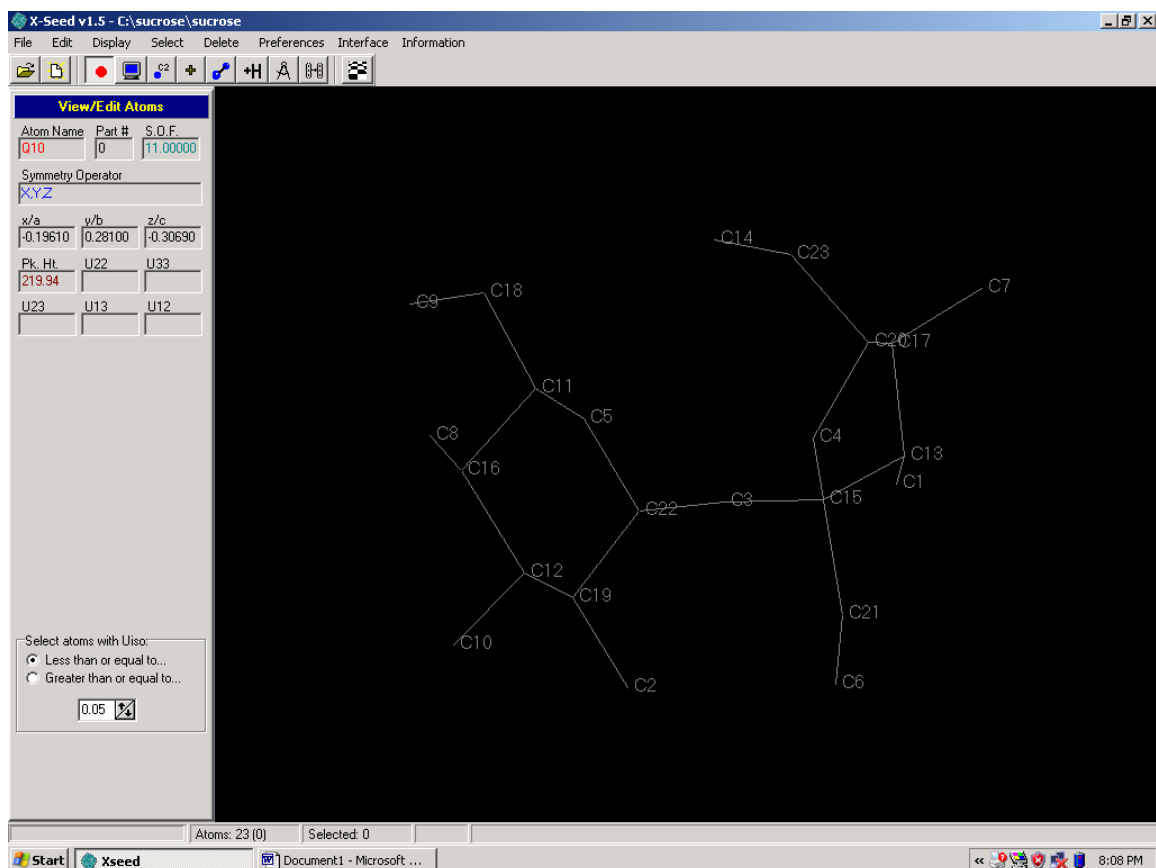


Now clean up your model. With the mouse choose and peak position that does not make good chemical sense. For example peak position 60.72 makes too many bonds. Move the cursor over that peak and double click. Repeat this procedure for all nonsensical peak positions.

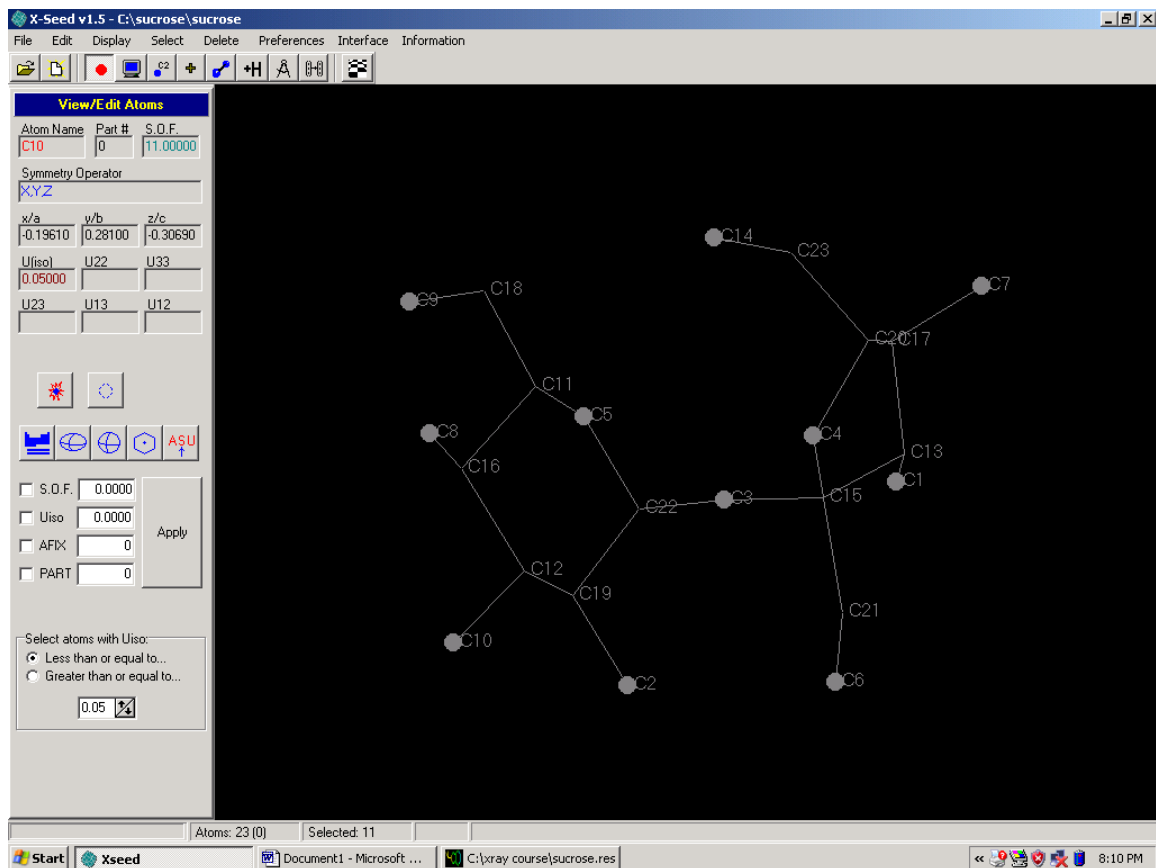
When finished type the delete (or backspace) key. The selected atoms will be erased.
 When finished rotate your molecule around until it looks something like this.



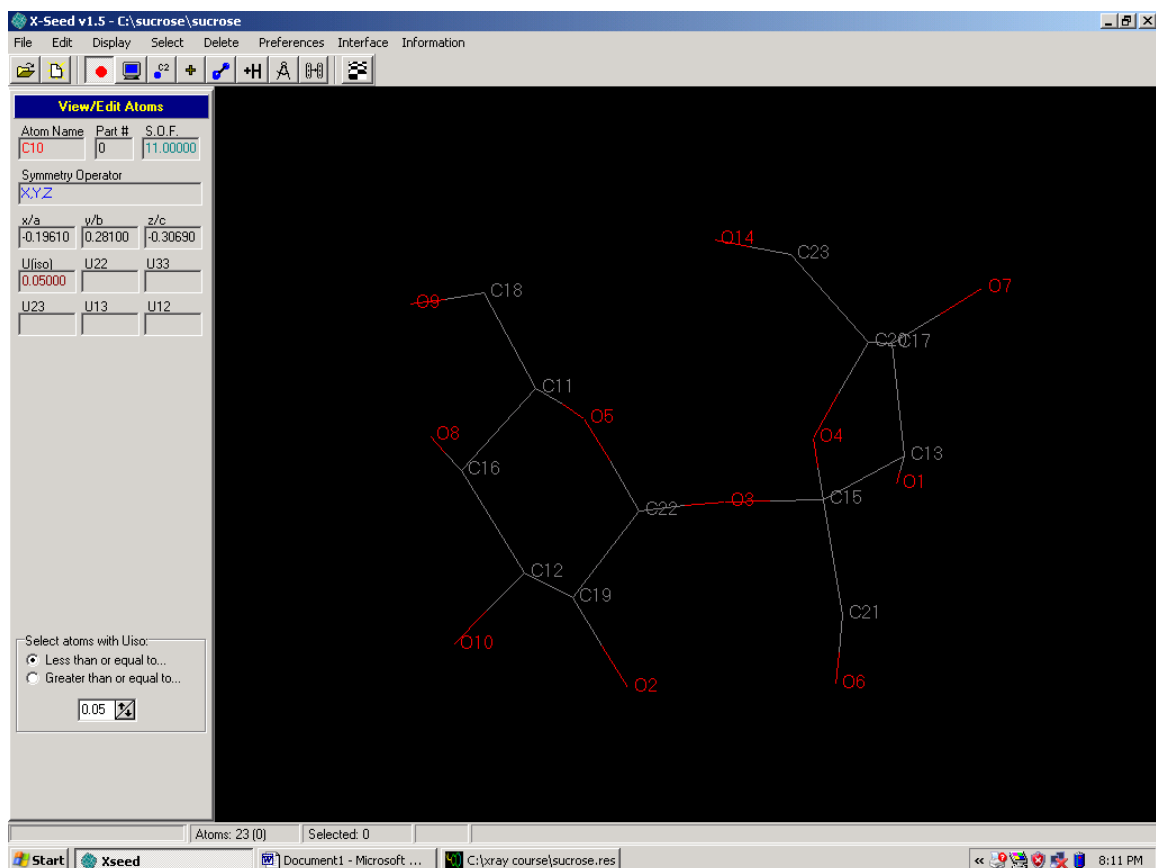
Now let's start naming atoms point to edit and choose "change Q's to C's". You should find all remaining peak positions now named Cij, where ij are integers. Don't worry if some of the carbons should be oxygens, we will change them next. For now your molecule should look something like this.



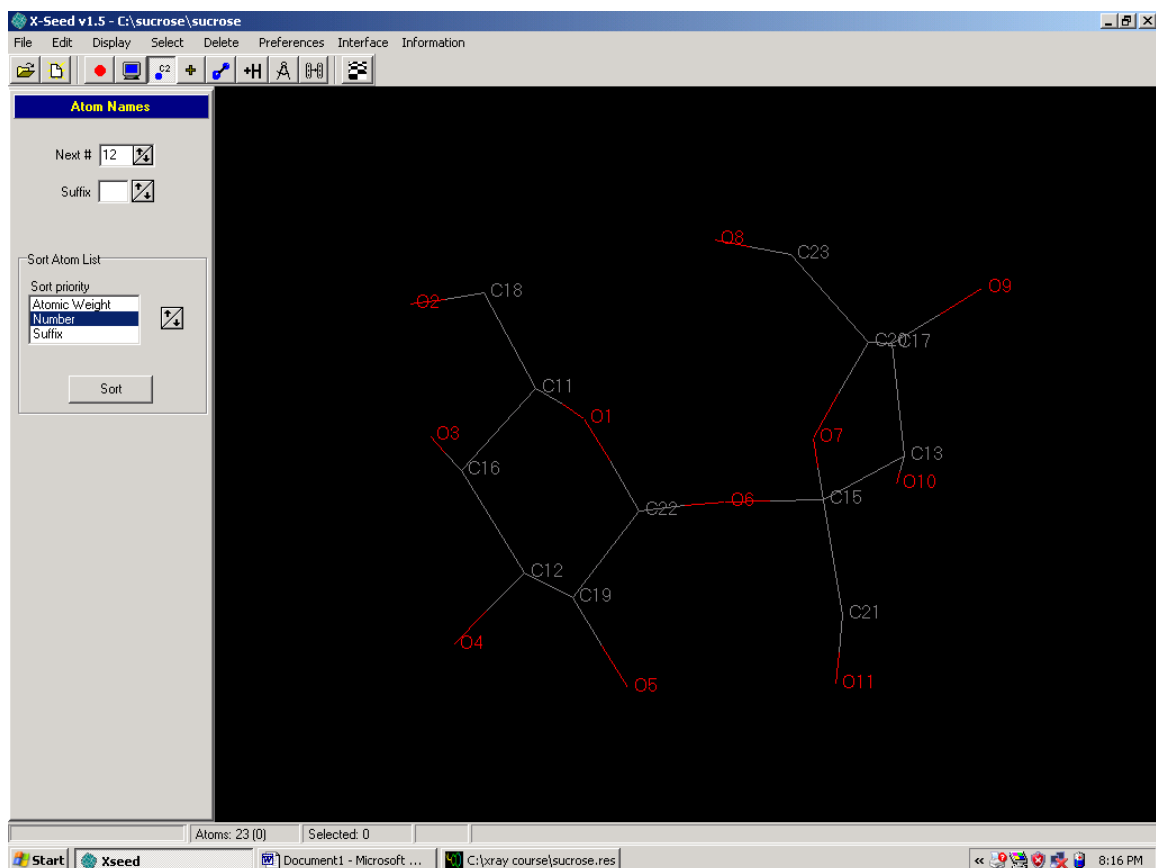
Now time to pick the oxygens. From your diagram you should know where the oxygens are placed in the molecule. You could also pick the oxygens by their peak heights, but this could lead to some errors in judgment.



Point to the icon that looks like a periodic table. A small window will appear. In the window choose Oxygen.

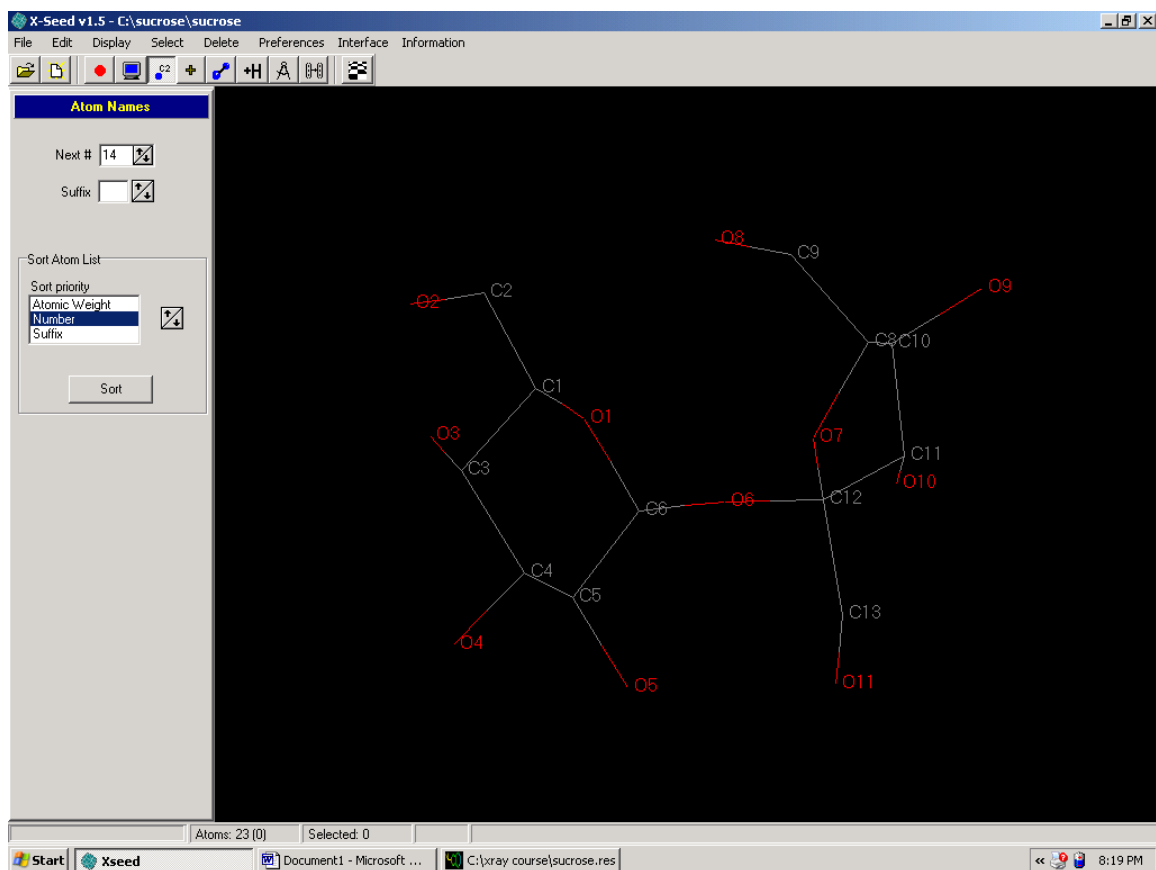


If everything looks alright then let's renumber the atoms. Point to the .C2 icon, move your cursor to O5 (glucose oxygen) and click the mouse. The O5 will now read O1. Following a counter-clockwise direction, click on each oxygen of the glucose ring. Go next to the bridging oxygen (O3 above) and click on the next ring oxygen following a clockwise direction. Should look something like this.

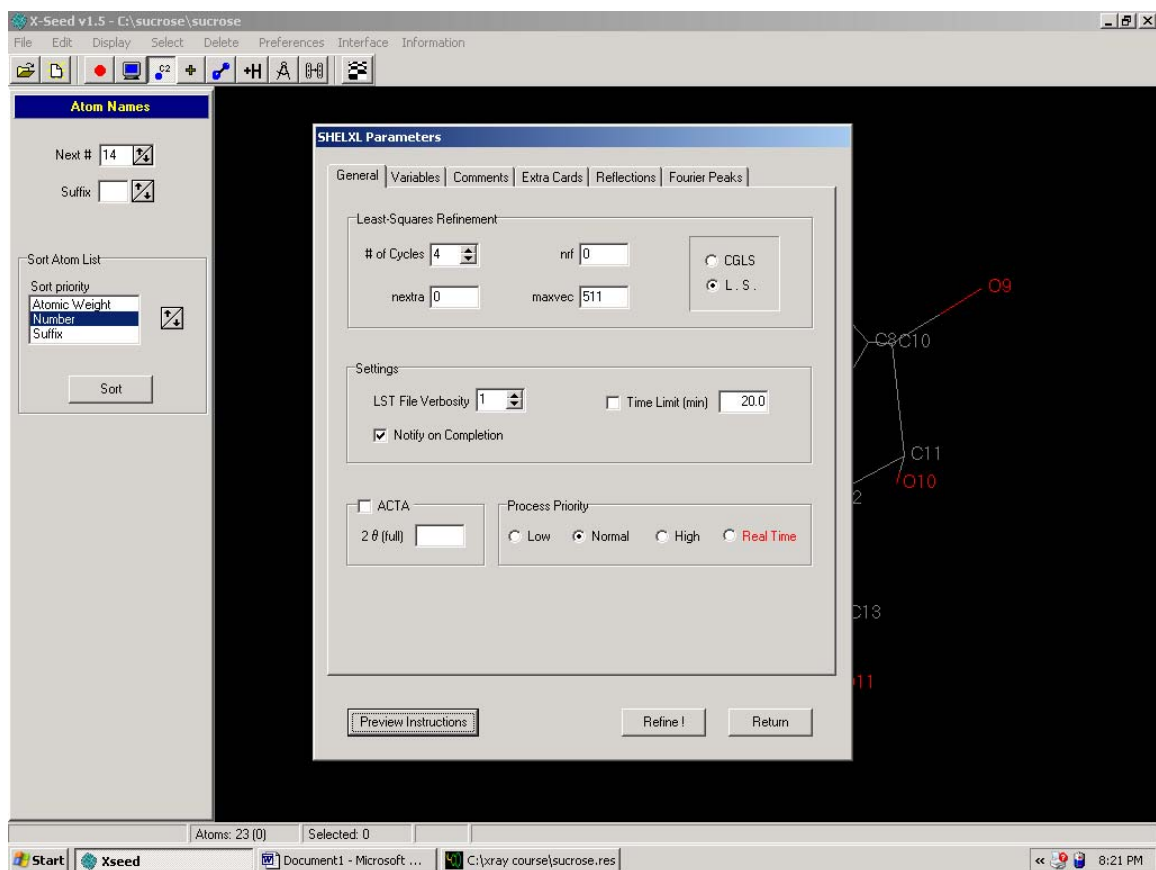


If you make a mistake then just point to the Next # box, type in the number where you made the mistake and then go back to the atom where you made the error and continue.

Now do the carbons. Go to the Next# box and type in 1. Go to the carbon next to O1 (C11 above) and click. Continue in a manner similar to the oxygen labeling.



Now you are ready to sort. Move the sort button and click. Your file is now labeled and sorted. You are ready to refine. Move to the Interface menu and select SHELXL



As before leave the defaults alone for now and select Refine!
The SHELXL window will open and refine for a while. When finished a summary window will appear.

The screenshot shows the X-Seed v1.5 interface with the SHELXL LST File Summary dialog box open. The dialog is divided into several sections:

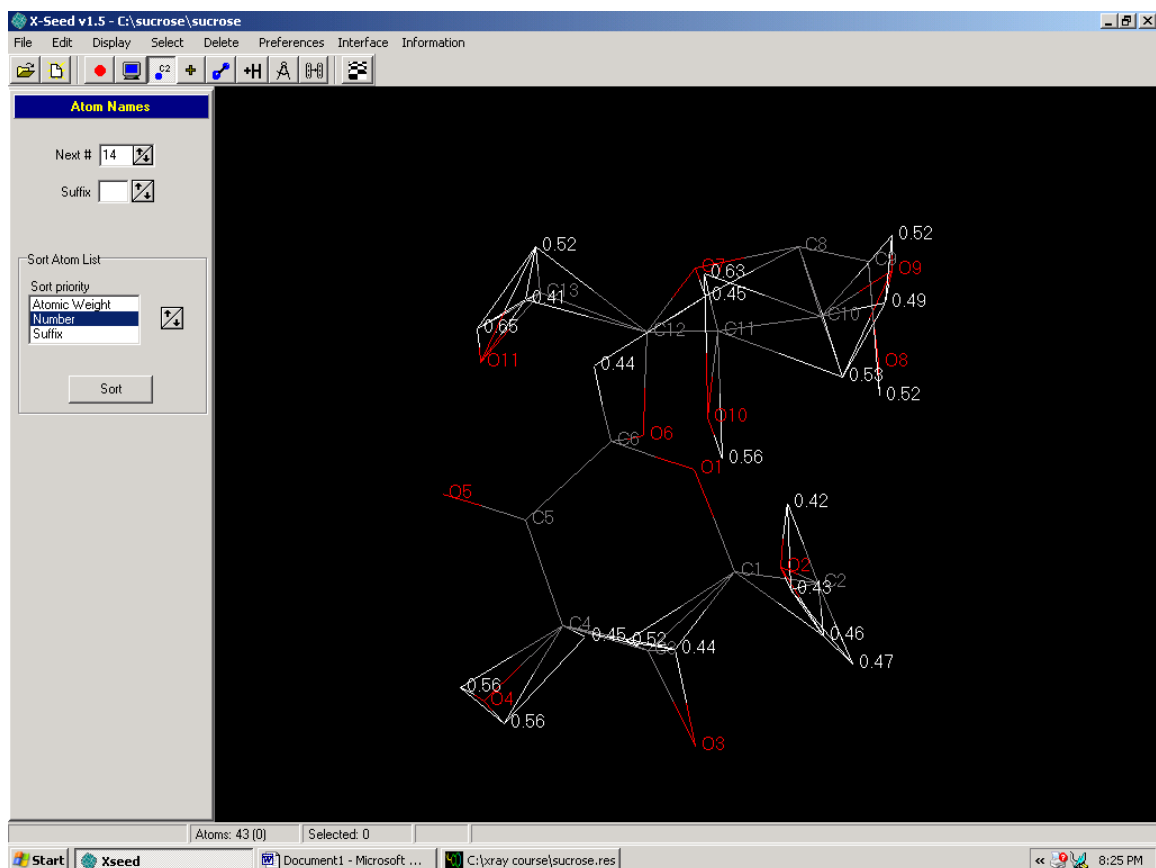
- Refinement Parameters:** Analysis of Variance, View RES File, View LST File.
- Reflection Data:** Systematic Absence Violations: 0, Unique Reflections: 3297, R_{int}: 0.0535, Bad Equivalents: 0, # Suppressed: 0, R_σ: 0.0826.
- Final Results:**

	wR2	Goof	Restrained Goof	Fo > 4 (Fo)	R1	# Data
	0.2592	1.424	1.424	All Data	0.0961	2009
				After Merging	0.1398	1803
- Statistics:** Highest Peak: 0.65, Deepest Hole: -0.46.
- Warning:** Cell contents from UNIT instruction and atom list do not agree. Flack x parameter = 0.0866 with esd 2.6315.

The 'Accept' button is highlighted, and the 'Reject' button is also visible. The background shows a partial view of a molecular structure with atoms labeled O9, C10, C11, and O10.

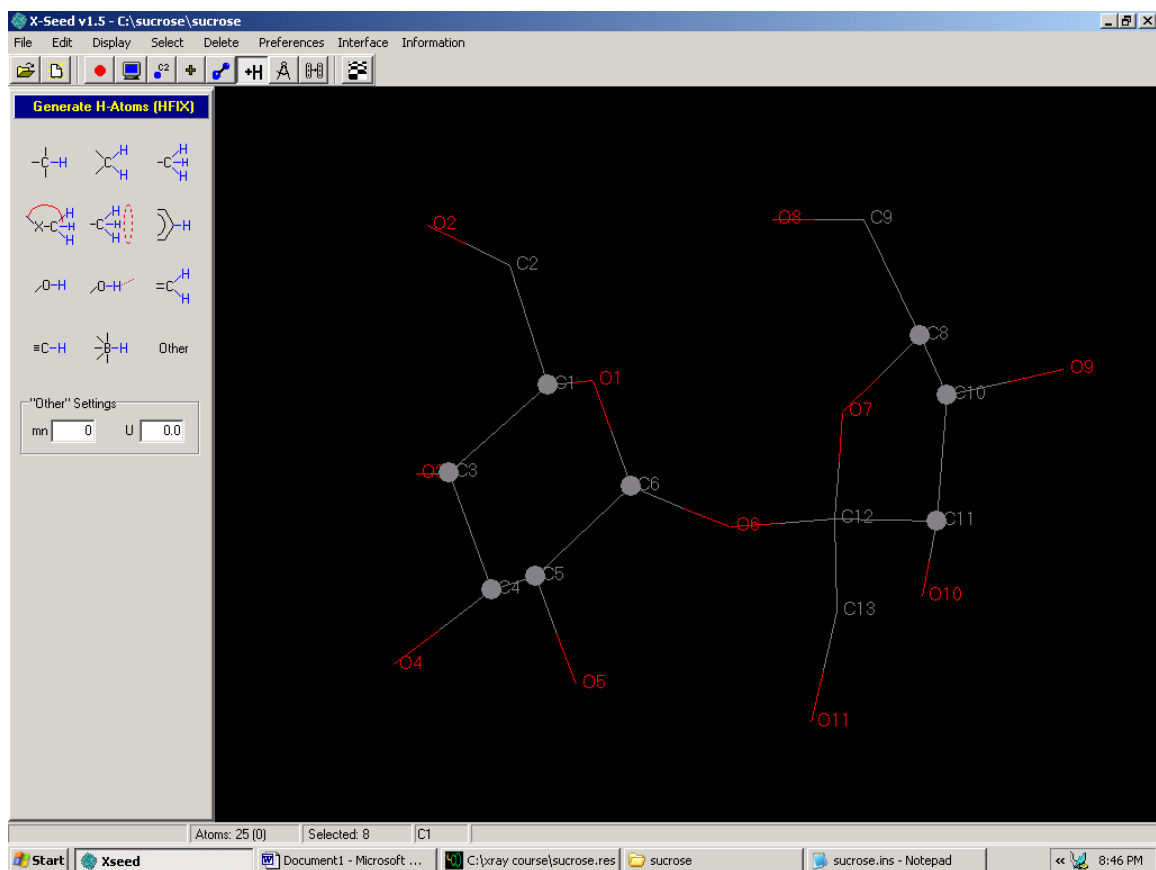
In this window you will see the R factors as well as a few comments. Were not finished with the refinement so don't worry about the R factors for now. Type Accept.

You will see a window with the residual peak heights in white. These are the Q peaks and they represent Hydrogens and lone pairs.

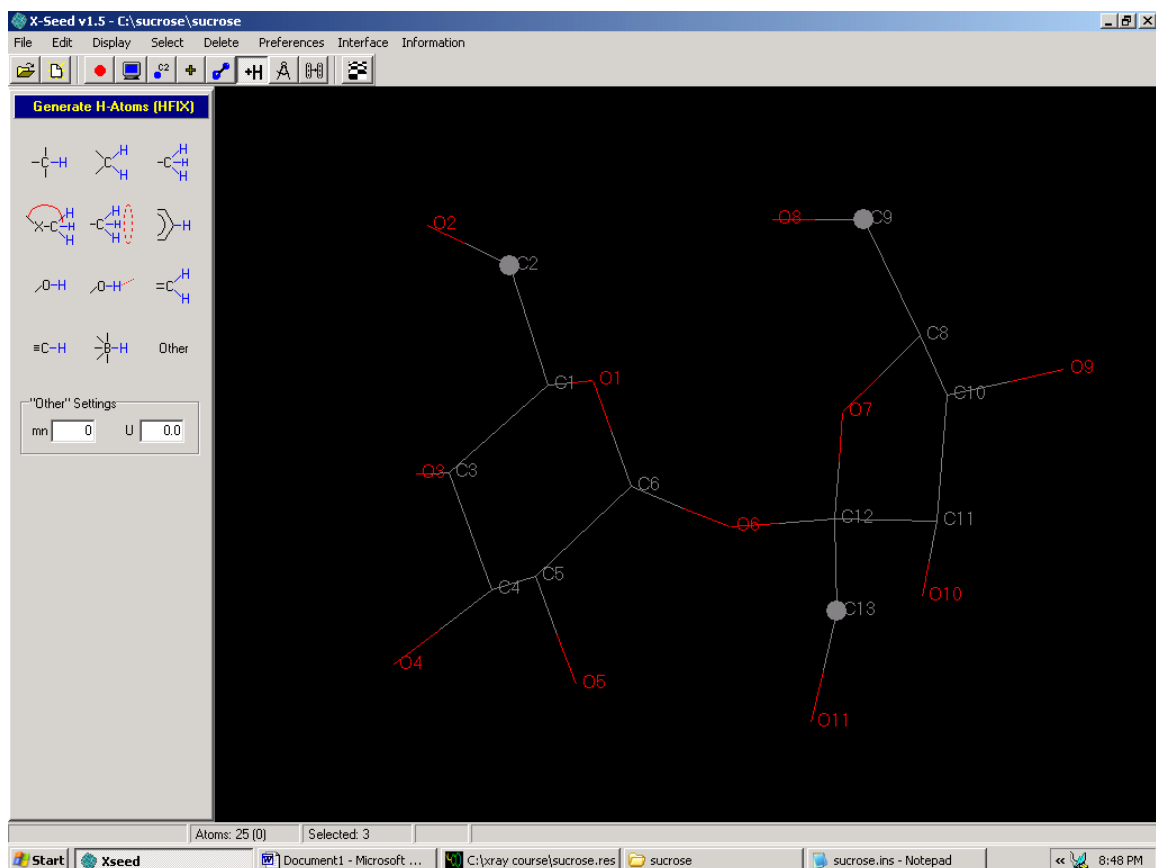


For now point to Delete and choose “all Residual Peaks” or just type a Q. If any residual peaks are left then goto the red dot icon, select the residual peaks and type the delete key.

To add Hydrogen Atoms goto the +H icon. First pick all the tertiary Carbons (R3C-H)

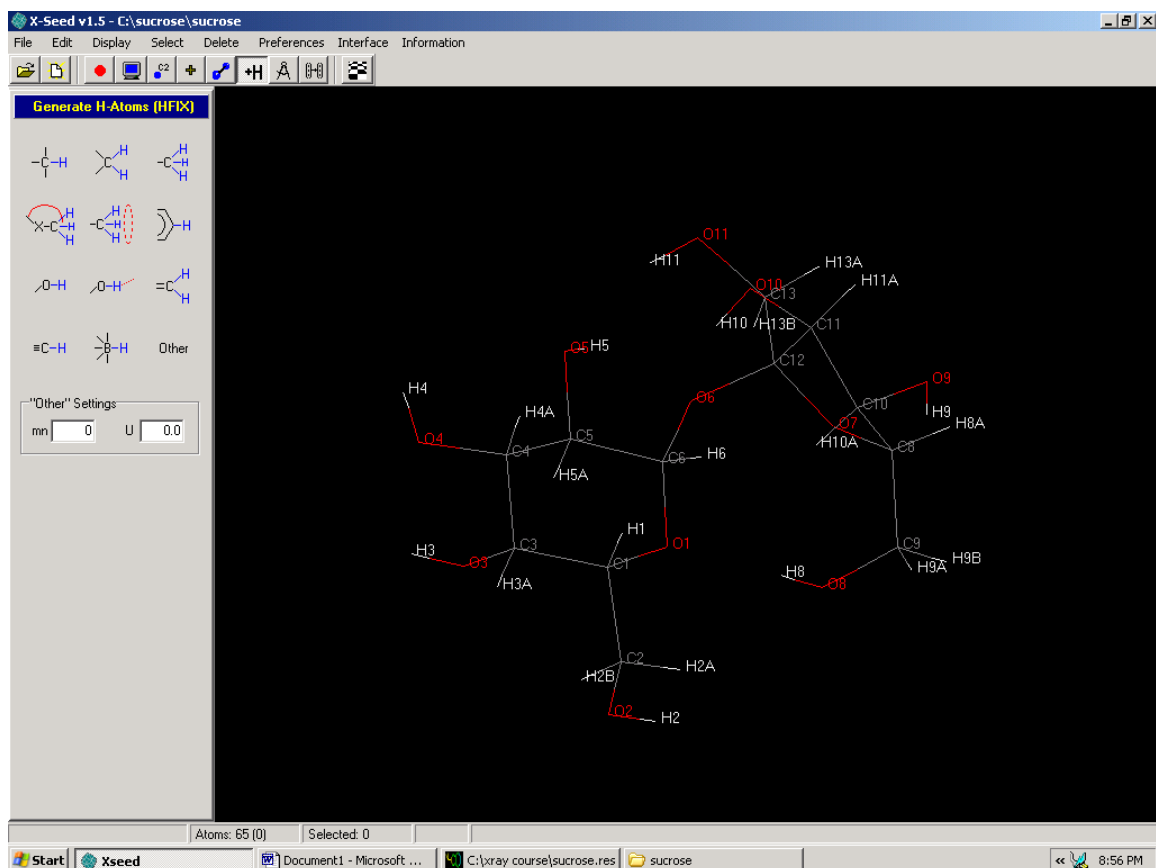


When complete type the -C-H icon. The selection will disappear. Now select the secondary Carbons



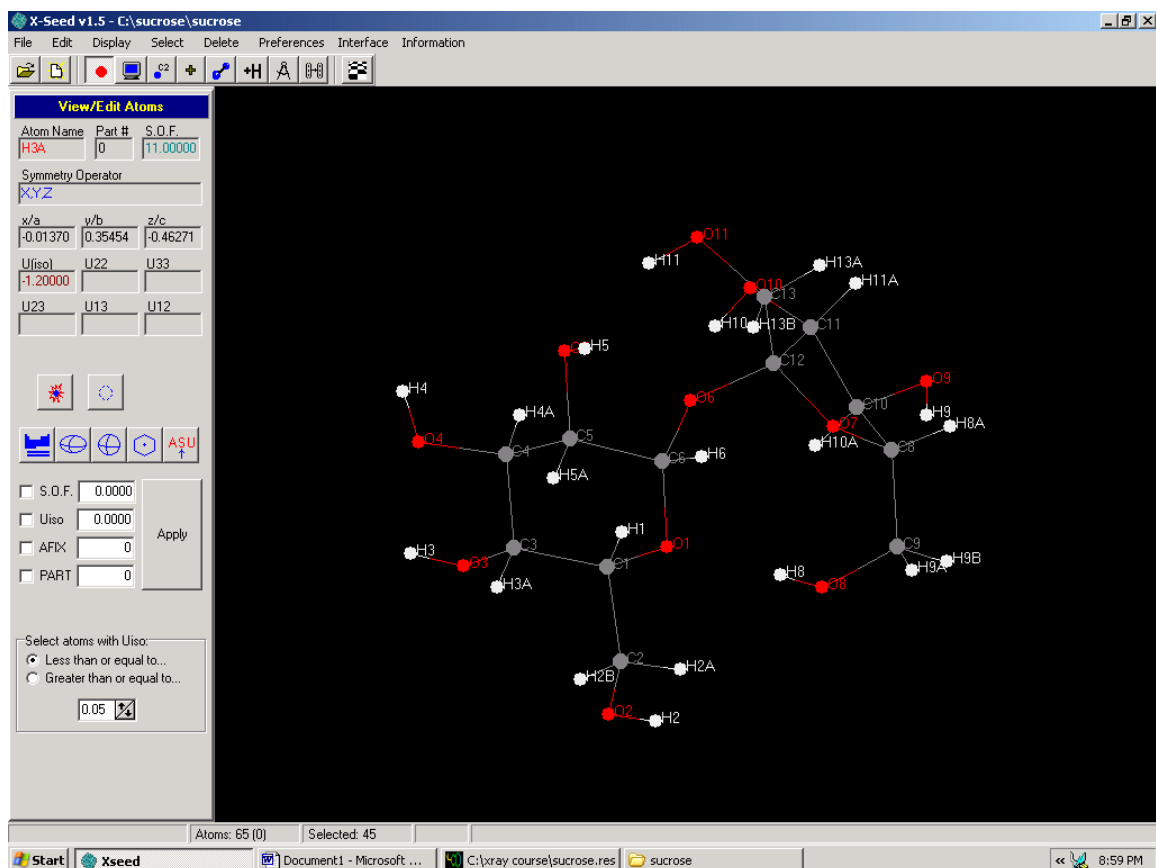
Type the CH₂ icon. The selection will disappear. Now select the terminal oxygens. Type the –O–H icon (not the –O–H.... icon). The selection will disappear.

Go back to the interface menu and select SHELXL again. If you make a mistake simply select the atom in error and click the Other button (with mn and U set to 0). This sometimes happens with C12. If SHELXL will not run then you may have C12 set wrong. Accept the results and kill the residual peaks. The structure should look like this



Inspect the structure carefully and look for mistakes.

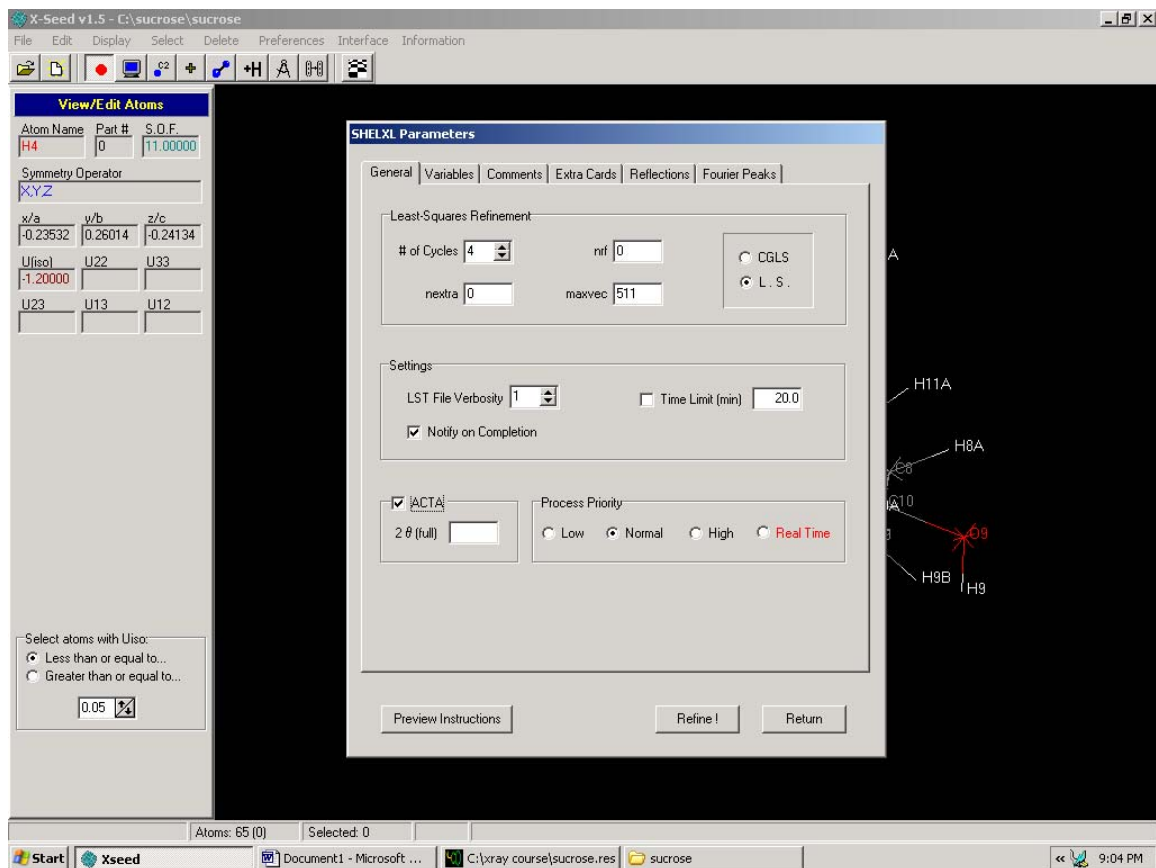
Next let's go anisotropic on all non-hydrogen atoms. To do so select all atoms. Under the select menu goto to select all and atoms. Return to the red dot icon and select the icon that looks like a football.



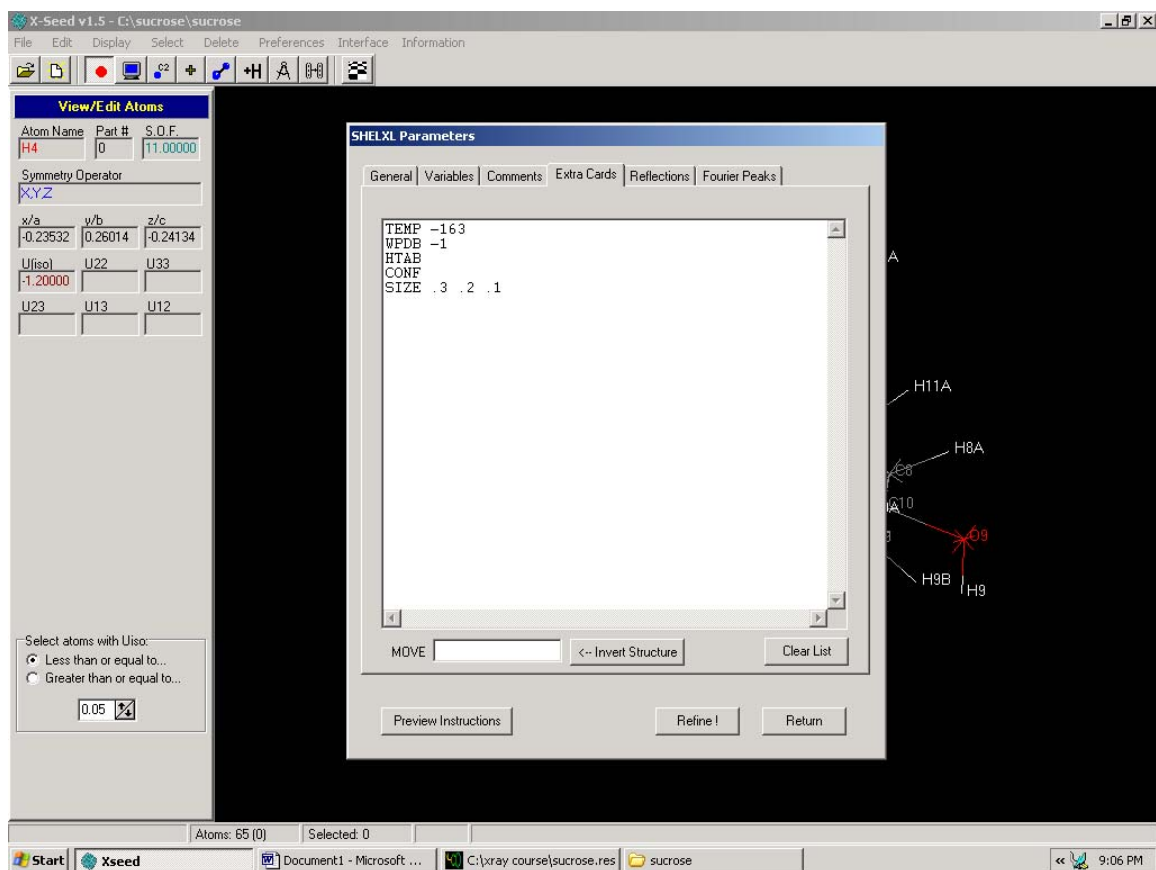
Your selections will disappear. Now go back to interface and SHELXL again. Refine the structure. Notice the R1 factor should be near .06.

Your ready for the last refinement cycles. Go back to interface. Now let's change a few things in the GUI.

First check the ACTA box



goto the Extra Cards Tab and add these commands



TEMP -163
 WPDB -1
 HTAB
 CONF
 SIZE .3 .2 .1

On the SIZE card add the correct crystal size for your sample.
 Goto the variable tab and check the WGHT box

X-Seed v1.5 - C:\sucrose\sucrose

File Edit Display Select Delete Preferences Interface Information

View/Edit Atoms

Atom Name	Part #	S.O.F.
H4	0	11.00000

Symmetry Operator
XYZ

x/a	w/b	z/c
-0.23532	0.26014	-0.24134
U[isol]	U22	U33
-1.20000		
U23	U13	U12

Select atoms with Uiso:
 Less than or equal to...
 Greater than or equal to...
 0.05

SHELXL Parameters

General Variables Comments Extra Cards Reflections Fourier Peaks

Free Variables (FVAR)
0.33102

EXTI
x 0

SWAT
g 0 U 2

WGHT
a 0.0776 b 0 c 0 d 0 e 0 f 0.3333

$$W = q / [\sigma^2(F_o^2) + (aP)^2 + bP + d + e \sin(\theta)]$$

$$P = f[\text{Max}(0 \text{ or } F_o^2)] + (1-f)F_c^2$$

Preview Instructions Refine! Return

Atoms: 65 (0) Selected: 0

Start Xseed Document1 - Microsoft ... C:\xray course\sucrose.res sucrose 9:09 PM

Now refine. The summary should look like this

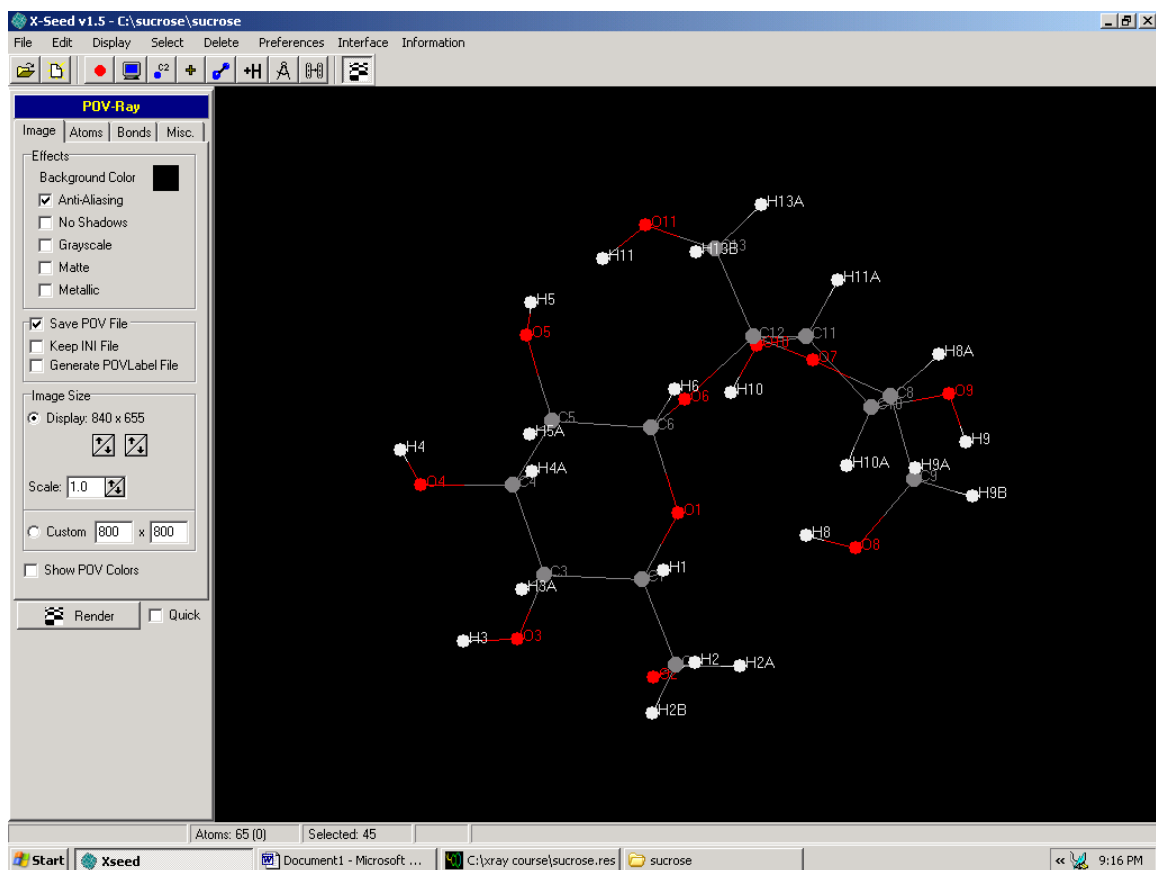
The screenshot shows the X-Seed v1.5 interface with the SHELXL LST File Summary dialog box open. The dialog box contains the following data:

Refinement Parameters		Analysis of Variance	
Reflection Data			
Systematic Absence Violations	0	Bad Equivalents	0
Unique Reflections	3297	# Suppressed	0
R _{int}	0.0535	R _σ	0.0826
Final Results			
wR2	0.1571	R1	0.0593
Goof	1.016	Fo > 4 (Fo)	0.1094
Restrained Goof	1.015	All Data	3297
		After Merging	0.0990
		# Data	1803
Highest Peak	0.31	Deepest Hole	-0.30
Flack x parameter = 0.7906 with esd 1.6510			

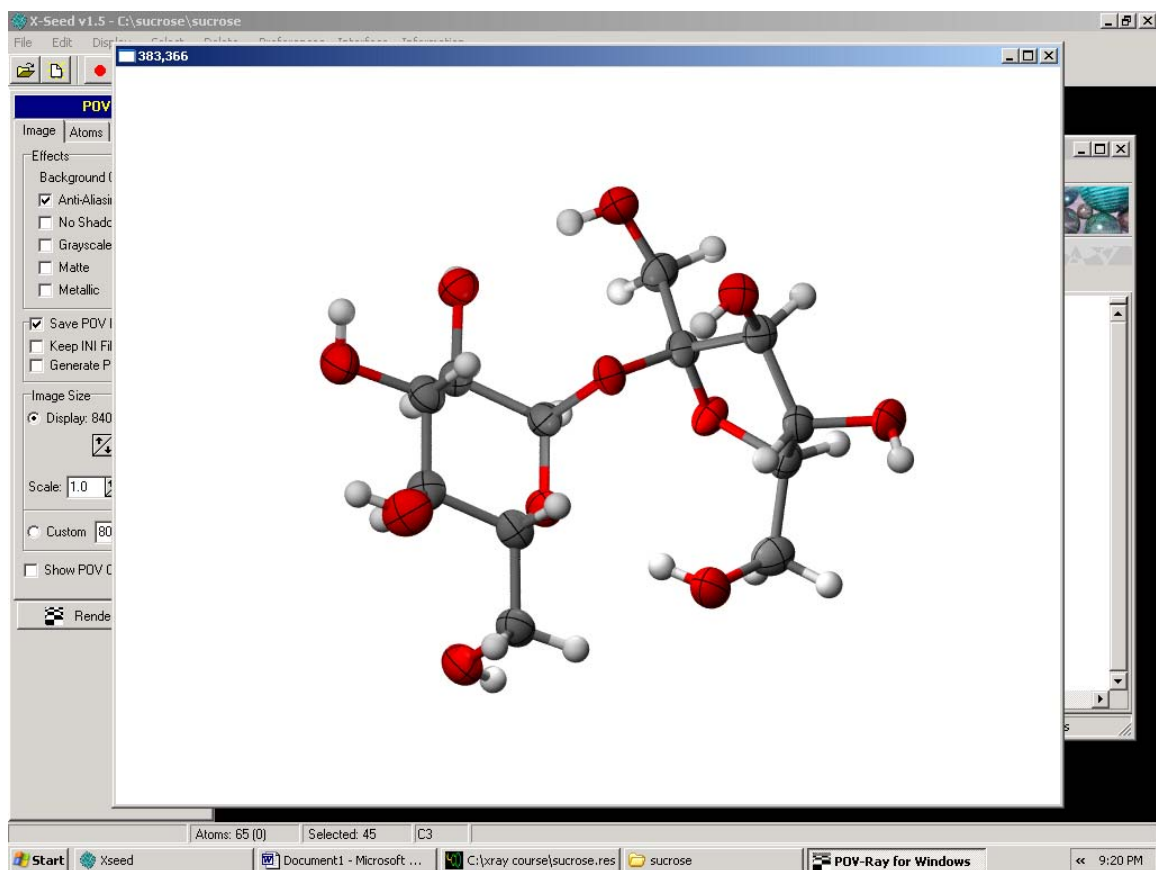
Buttons at the bottom of the dialog are 'Accept' and 'Reject'. The background shows a molecular structure with atoms labeled H11A, H8A, H9B, and H9.

The R1 factor is 0.0593 and the wR2 is 0.1571. The highest peak is 0.3 electrons
Accept the results and delete the residual peaks.

You have finished the refinement. Its time to draw an image. Select all atoms. Type the POV-RAY button (the checkered board). Goto the bond tab and check the ellipsoid button. Click APPLY. Return to the image tab. Select a white background, anti-aliasing and save pov file. Rotate the molecule until you have the view you want. Type render

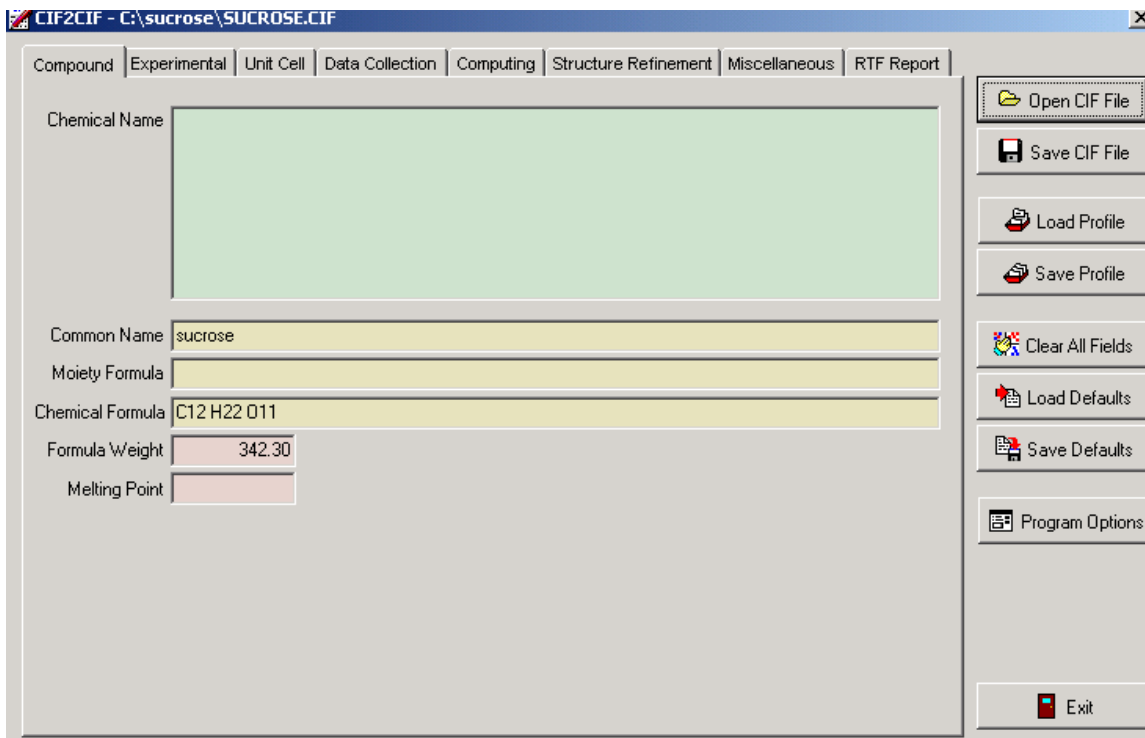


Save the file

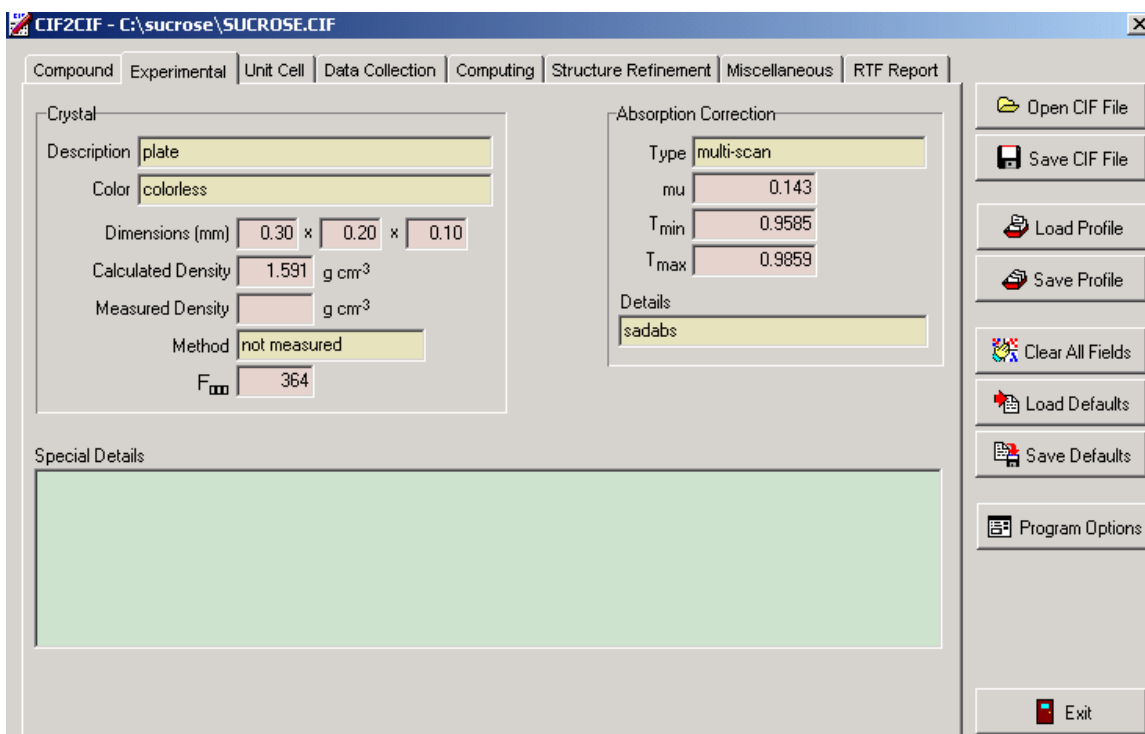


Close POV-RAY and X-SEED.

You should find a file named sucrose.cif and sucrose.fcf. Run the program CIF2CIF
Open sucrose.cif



Under the experimental tab enter



Description : plate Color : colorless Type : multi-scan Details : sadabs

Save the CIF file.

Generating Reports