X-seed Practical

A LAPTOP university course

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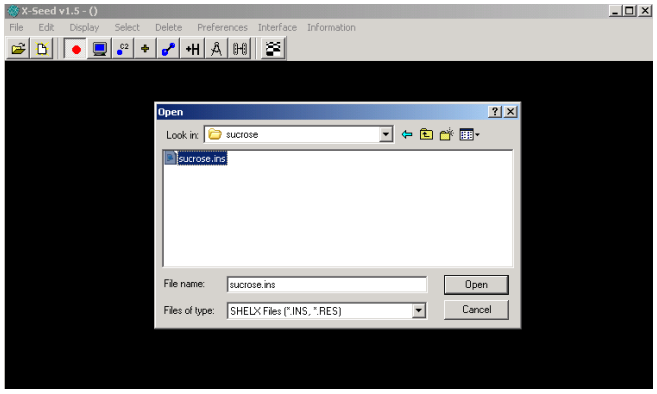
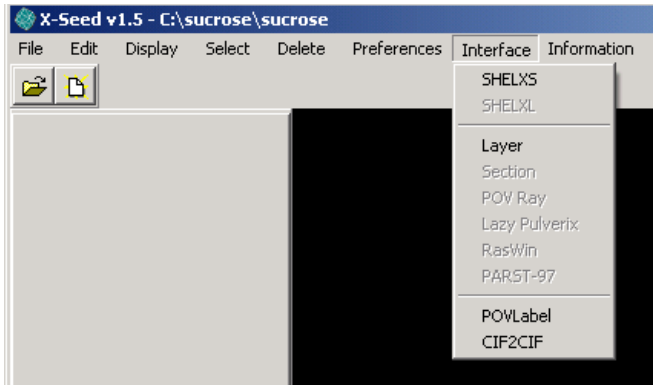
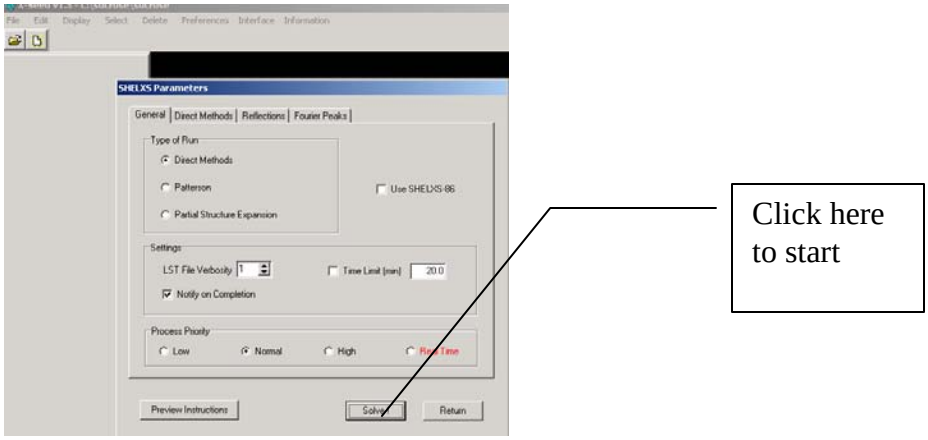
Start the program X-SEED and open “sucrose.ins”.



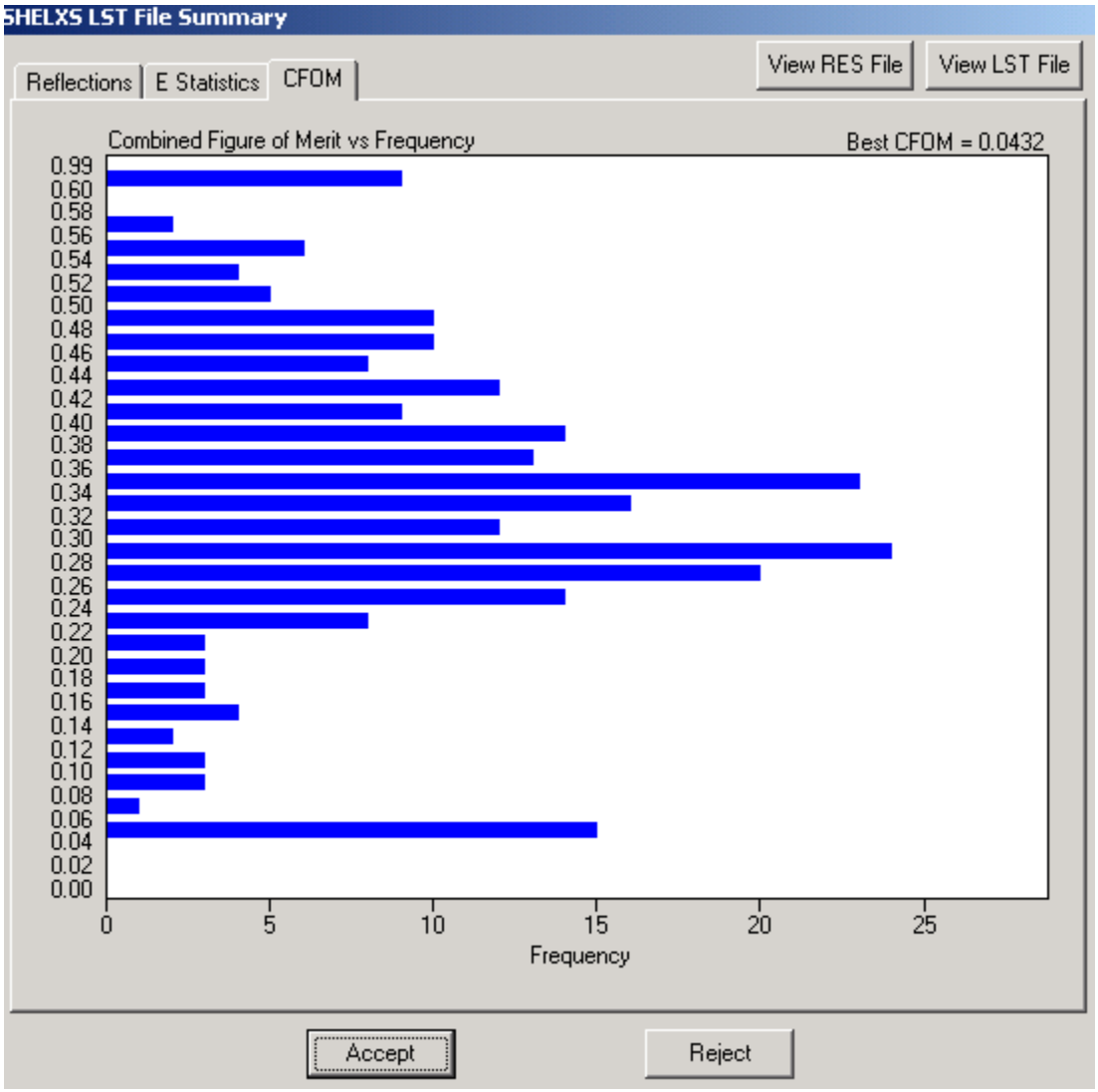
Point to interface and then SHELXS



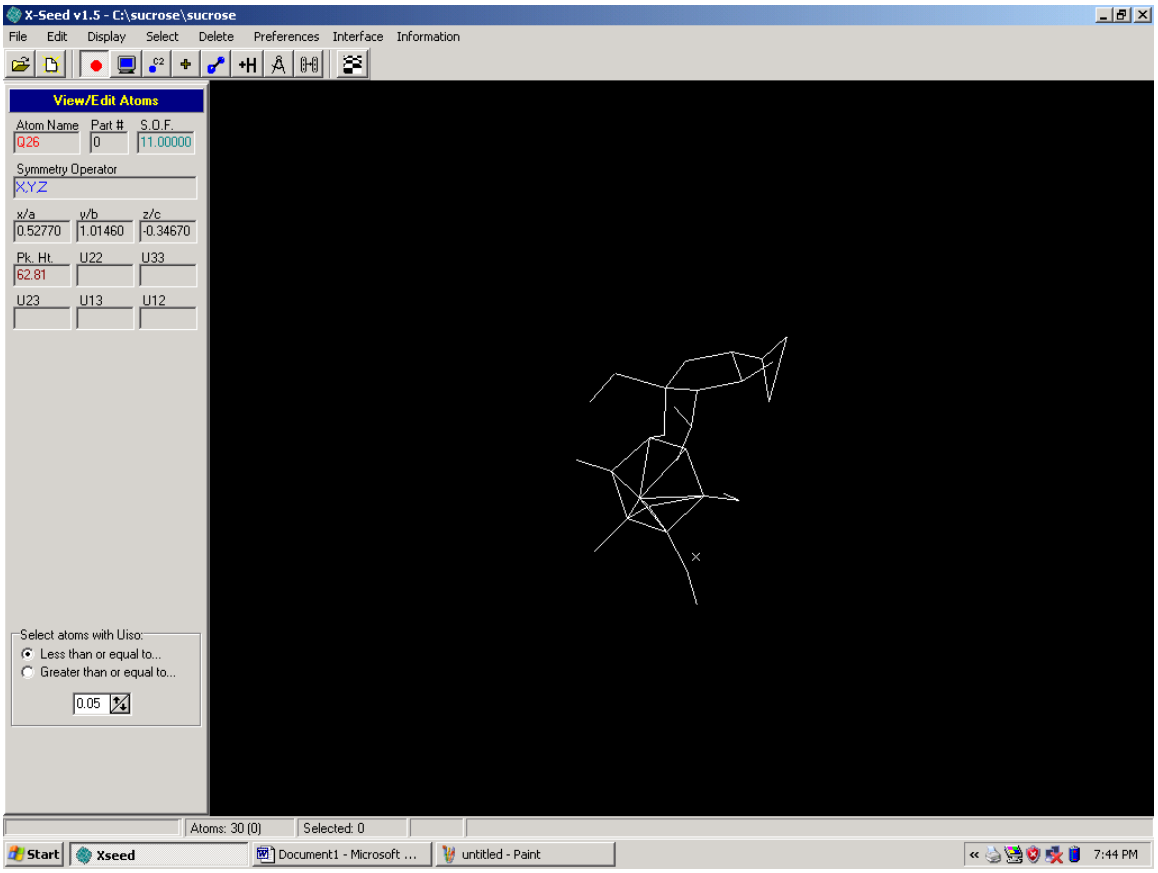
You will see the Direct Methods radio button checked, with a low output verbosity (summarized results) and normal priority on the processing. These are the defaults and are good for the first run. You should only change these settings if you cannot solve the structure on the first run.



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.

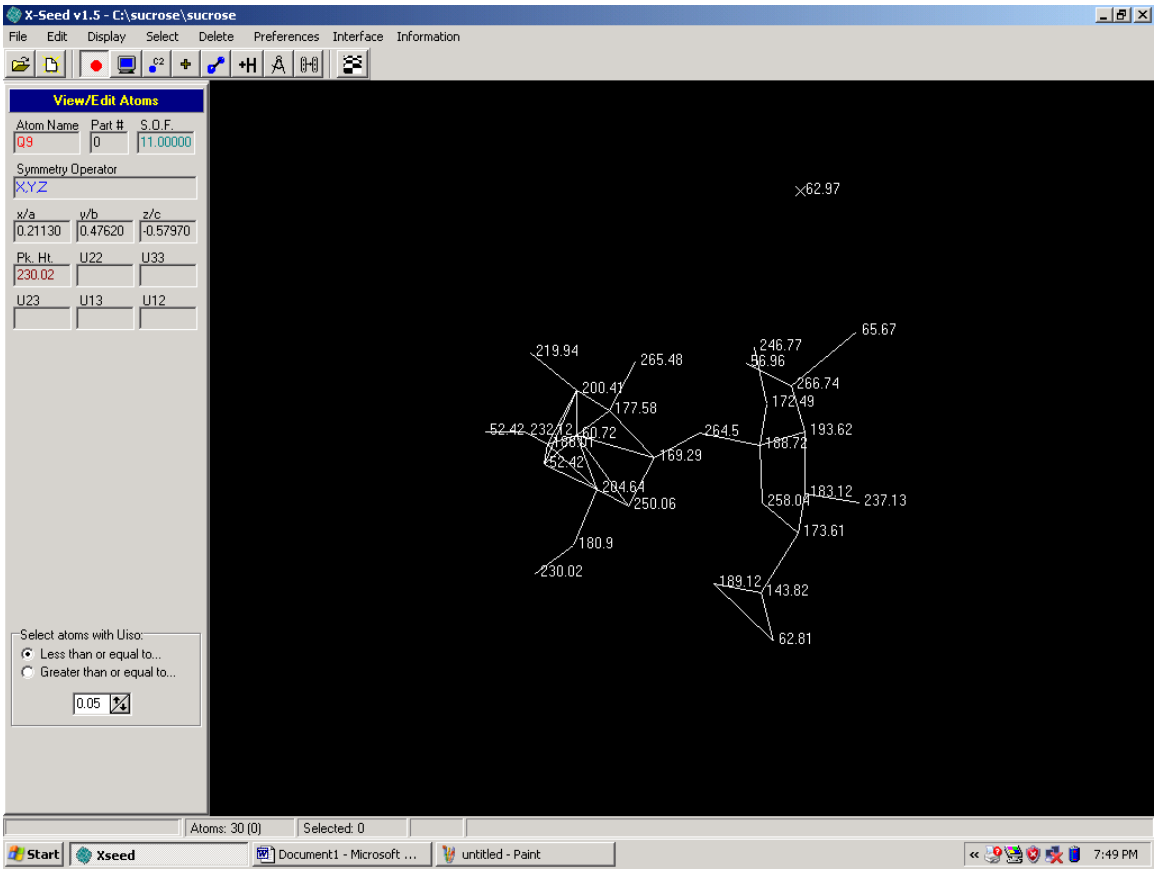


Point to the Accept button and continue. You should see something like this



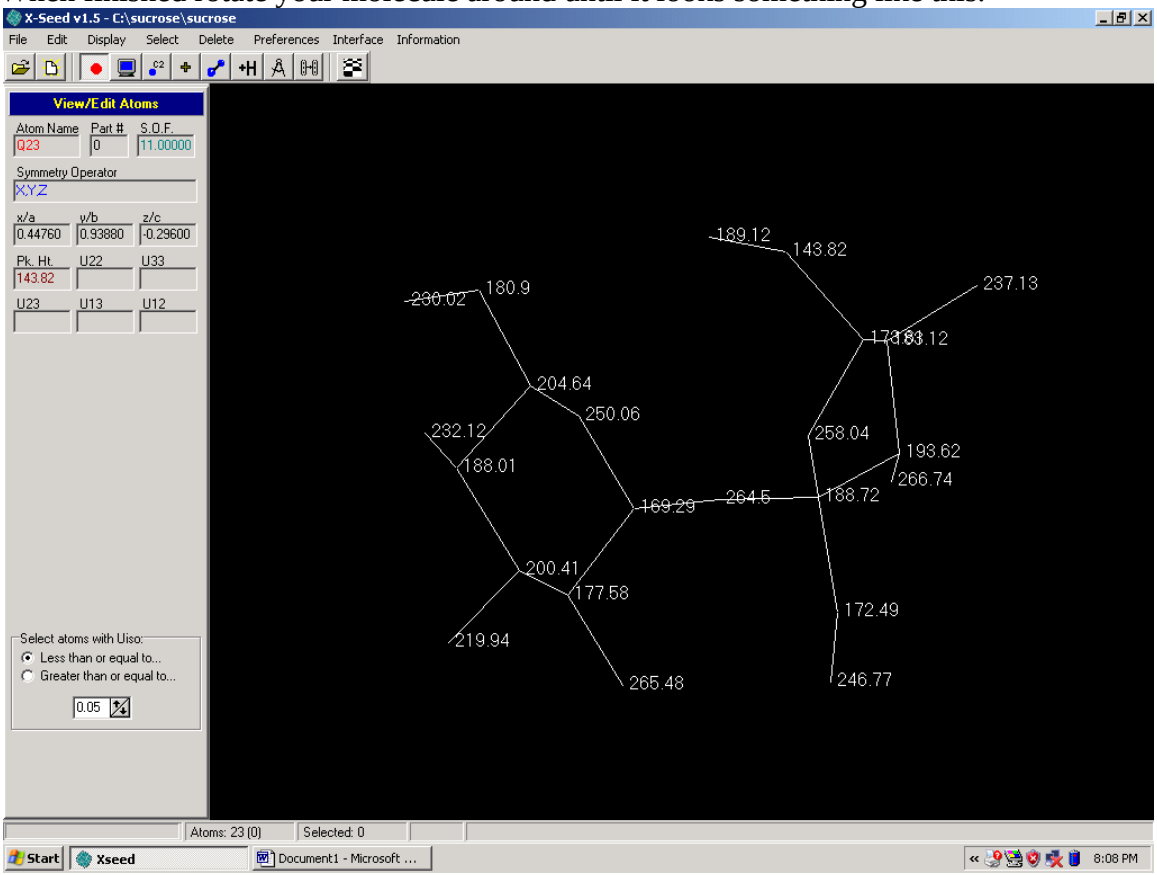
Hold the left mouse button down and move the mouse. Now hold the right mouse button down and move the mouse. Note what happens. Try the cntl and shift buttons with the mouse movement. Experiment until you feel comfortable and you know how to move and rotate the molecule.

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

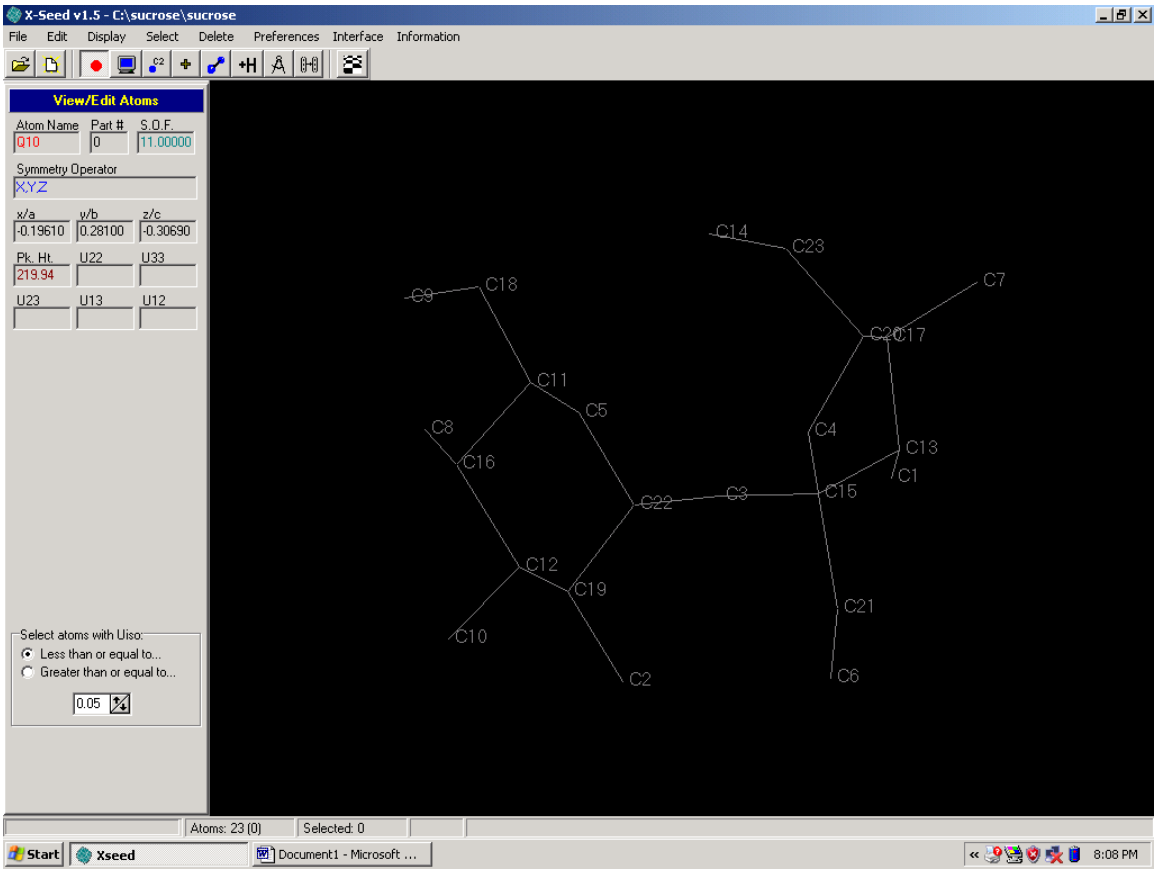


Now clean up your model. With the mouse choose and peak position that does not make good chemical sense. For example peak position 60.72 makes too many bonds. Move the cursor over that peak and double click. Repeat this procedure for all nonsensical peak positions.

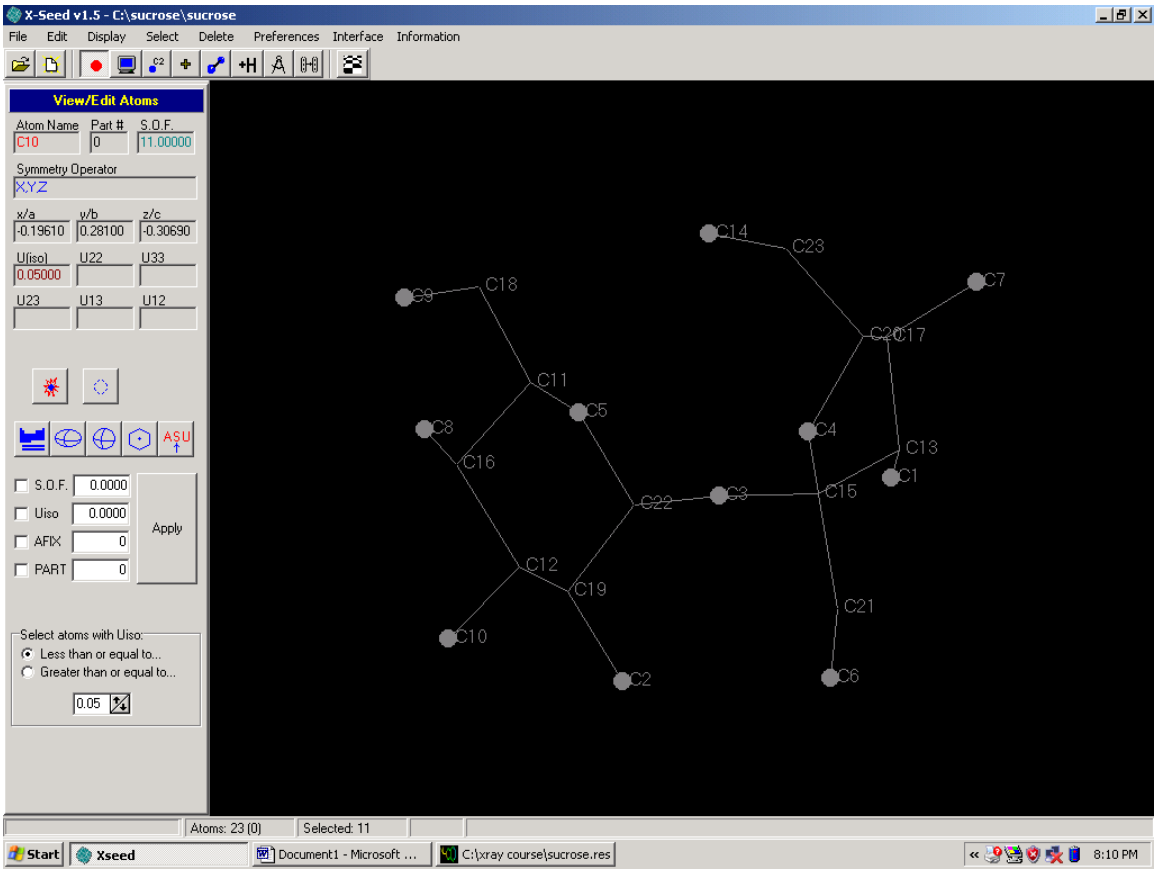
When finished type the delete (or backspace) key. The selected atoms will be erased. When finished rotate your molecule around until it looks something like this.



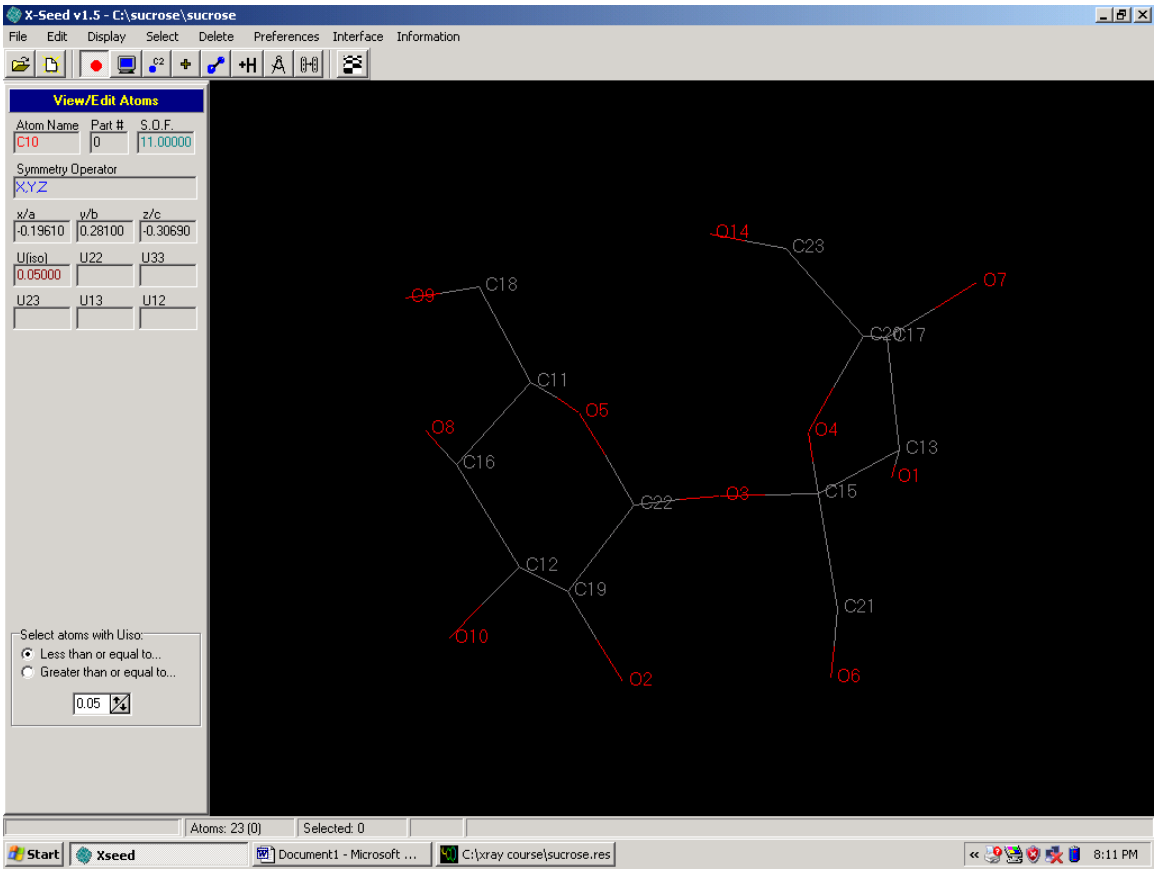
Now let's start naming atoms point to edit and choose "change Q's to C's". You should find all remaining peak positions now named Cij, where ij are integers. Don't worry if some of the carbons should be oxygens, we will change them next. For now your molecule should look something like this.



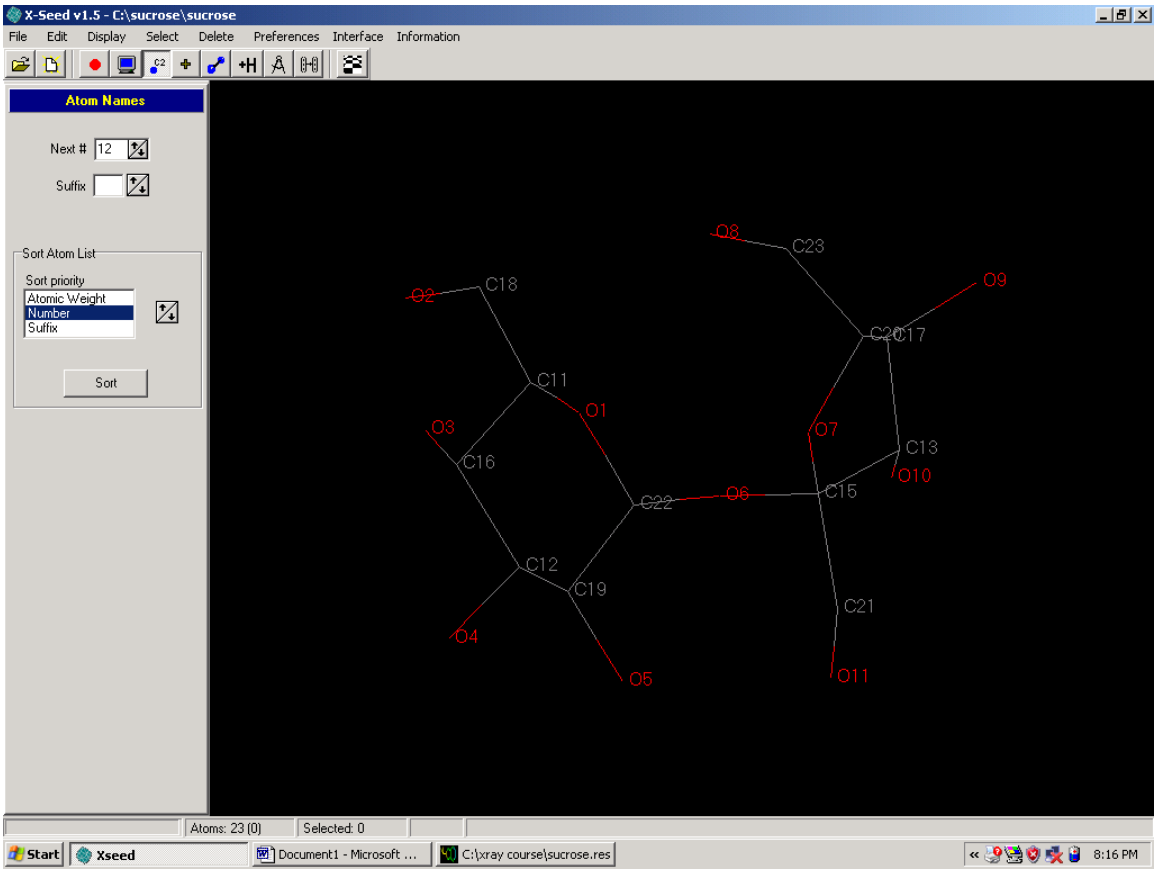
Now time to pick the oxygens. From your diagram you should know where the oxygens are placed in the molecule. You could also pick the oxygens by their peak heights, but this could lead to some errors in judgment.



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

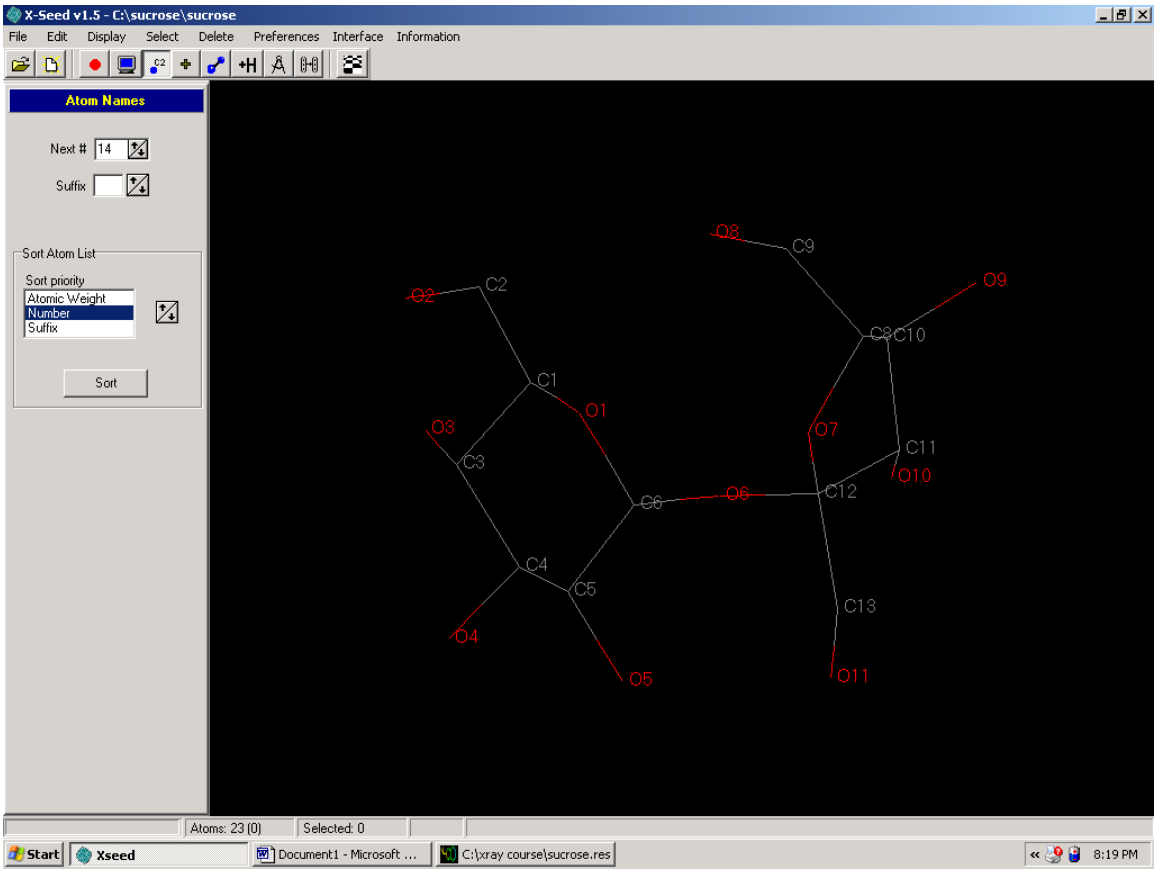


If everything looks alright then lets renumber the atoms. Point to the .C2 icon move your cursor to O5 (glucose oxygen) and click the mouse. The O5 will now read O1 . Following a counter-clockwise direction click on each oxygen of the glucose ring. Go next to the bridging oxygen (O3 above) and click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

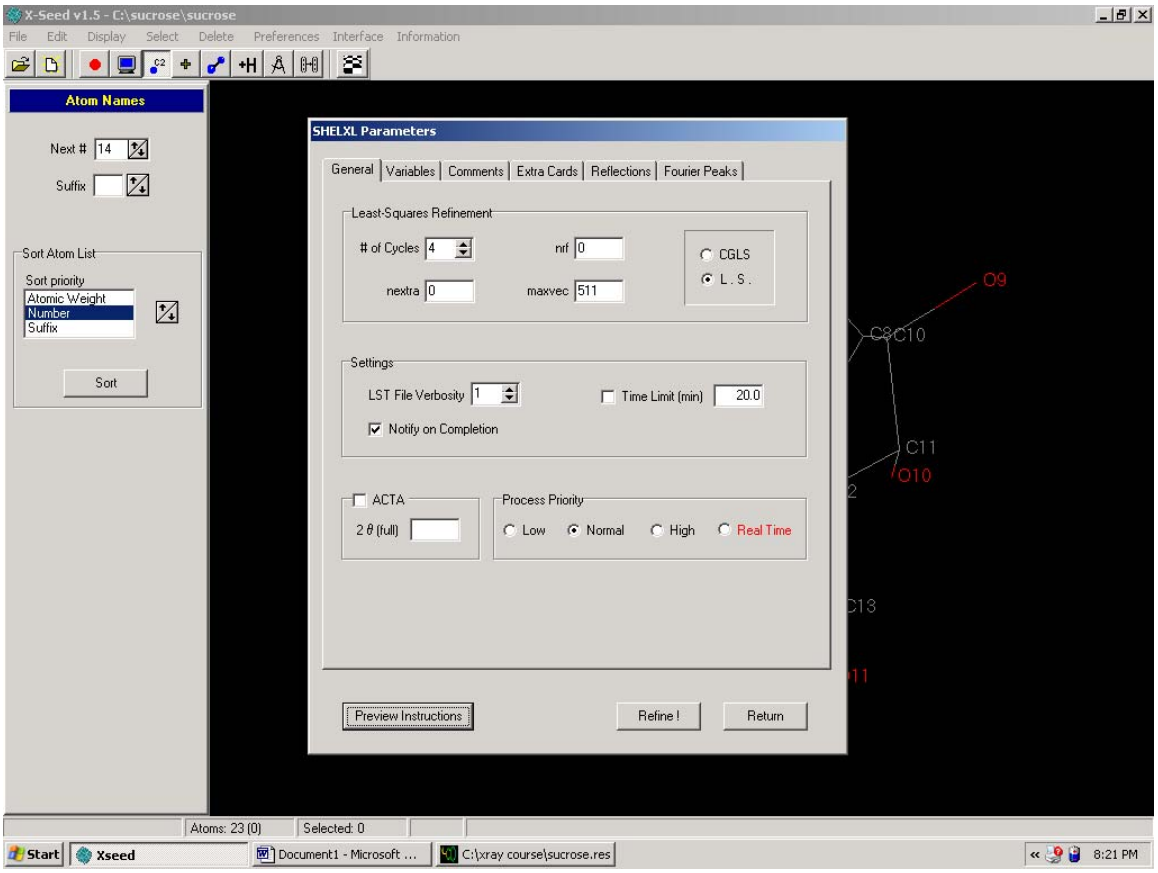


If you make a mistake then just point to the Next # box, type in the number where you made the mistake and then go back to the atom where you made the error and continue.

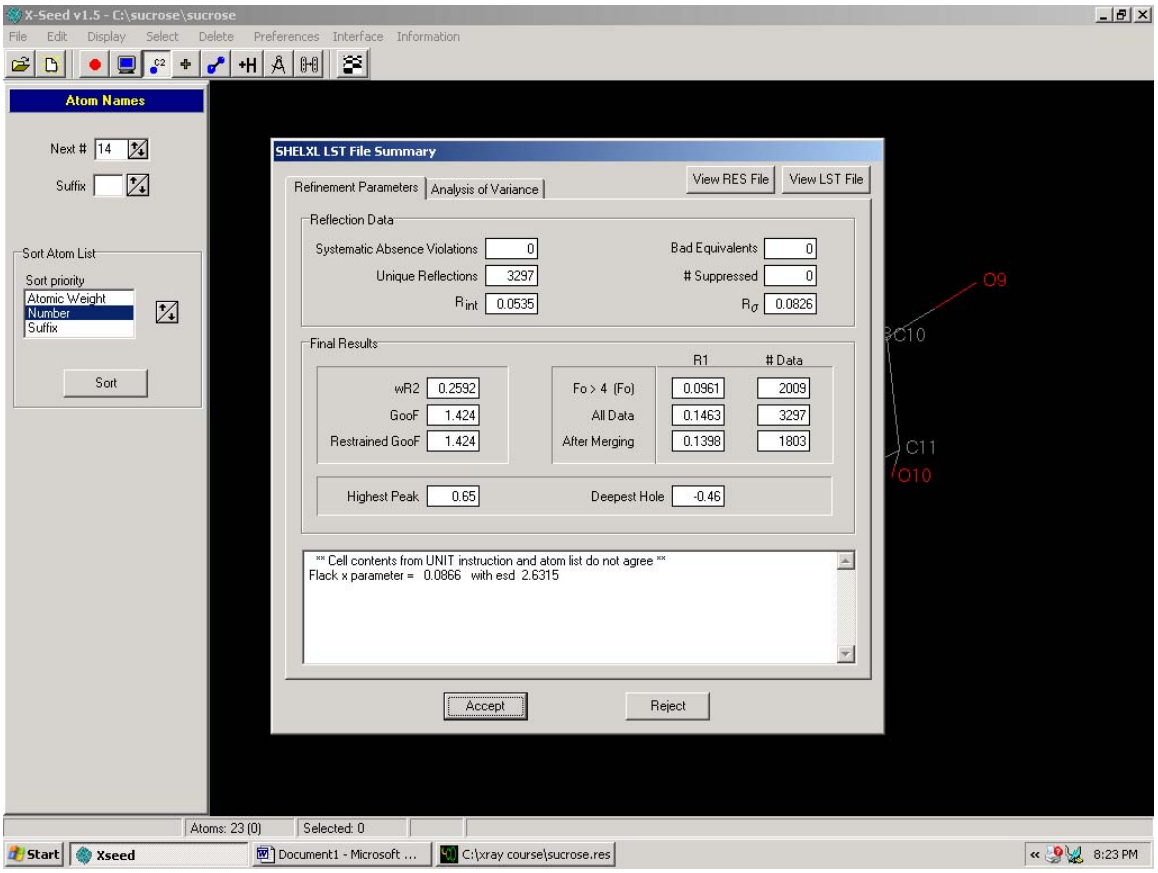
Now do the carbons. Go to the Next# box and type in 1. Go to the carbon next to O1 (C11 above) and click. Continue in a manner similar to the oxygen labeling.



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

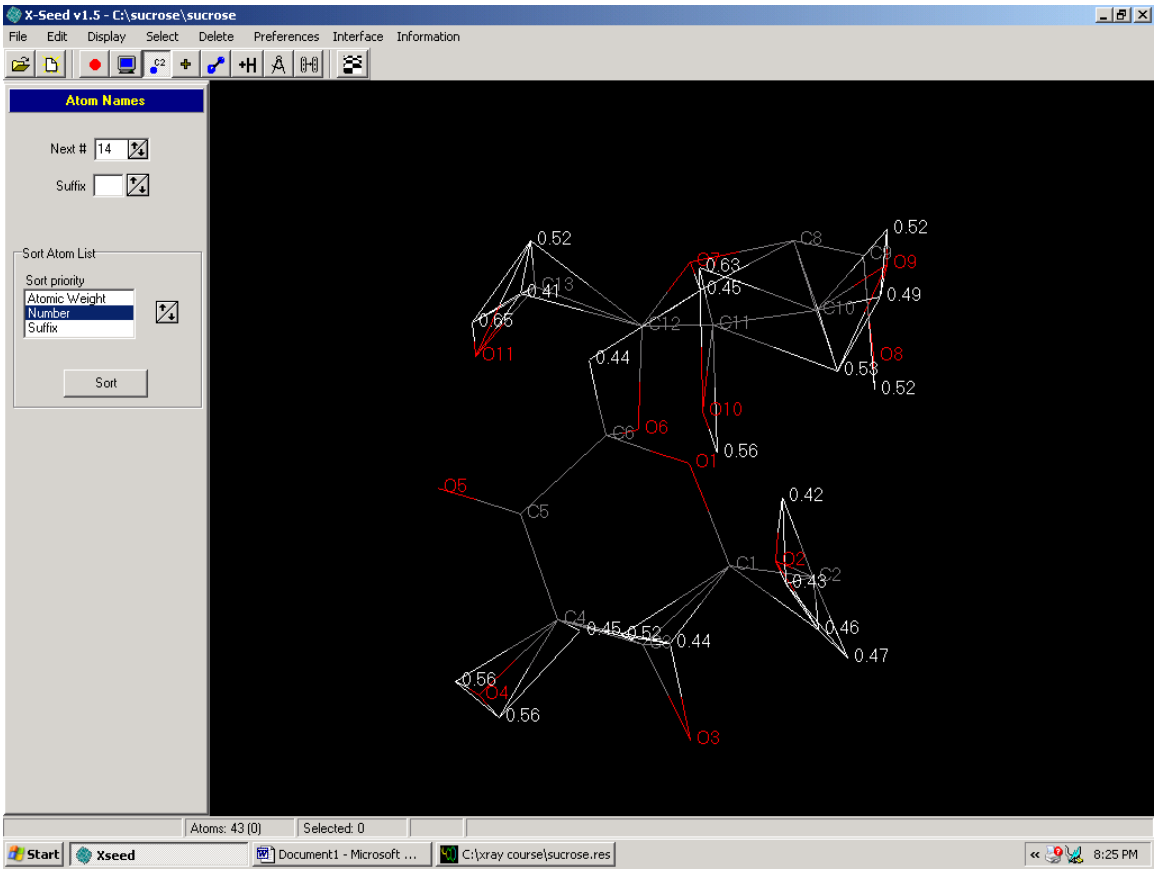


As before leave the defaults alone for now and select Refine!
The SHELXL window will open and refine for a while. When finished a summary window will appear.



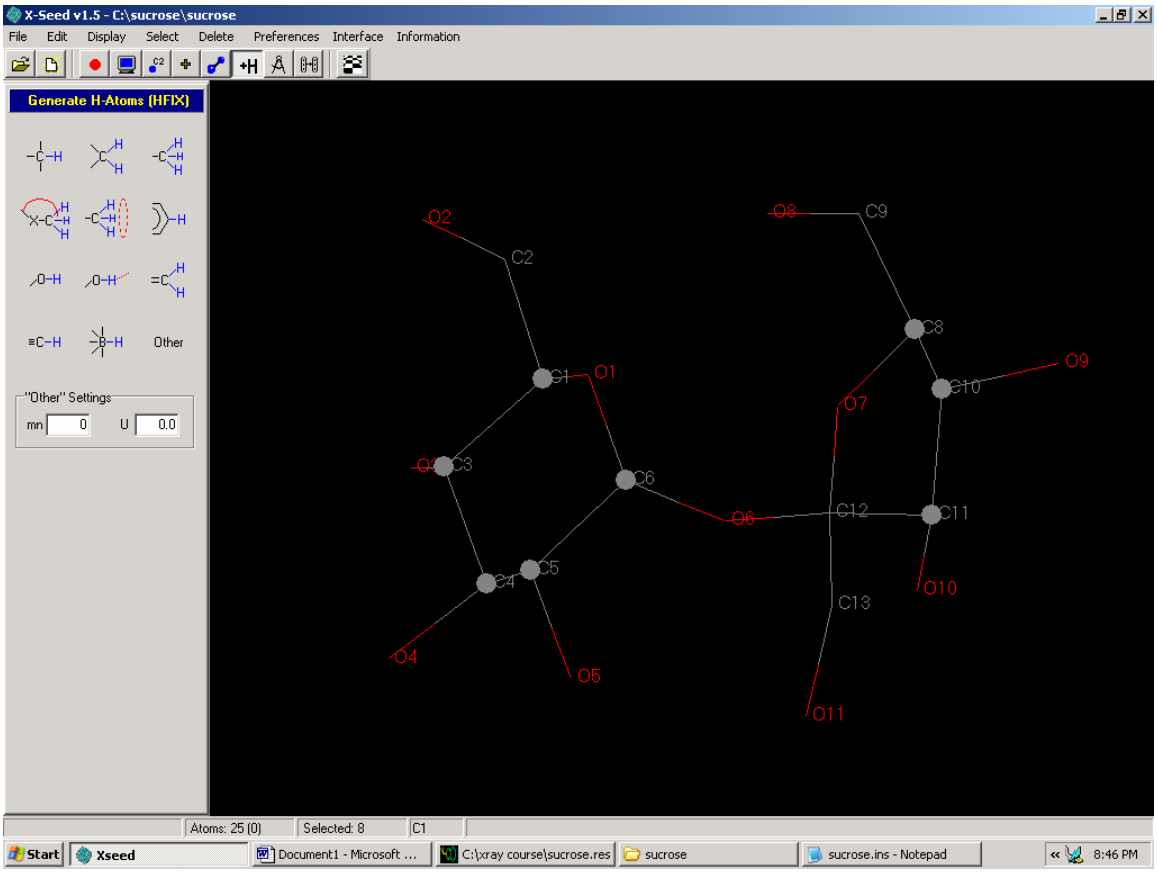
In this window you will see the R factors as well as a few comments. Were not finished with the refinement so don't worry about the R factors for now. Type Accept.

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

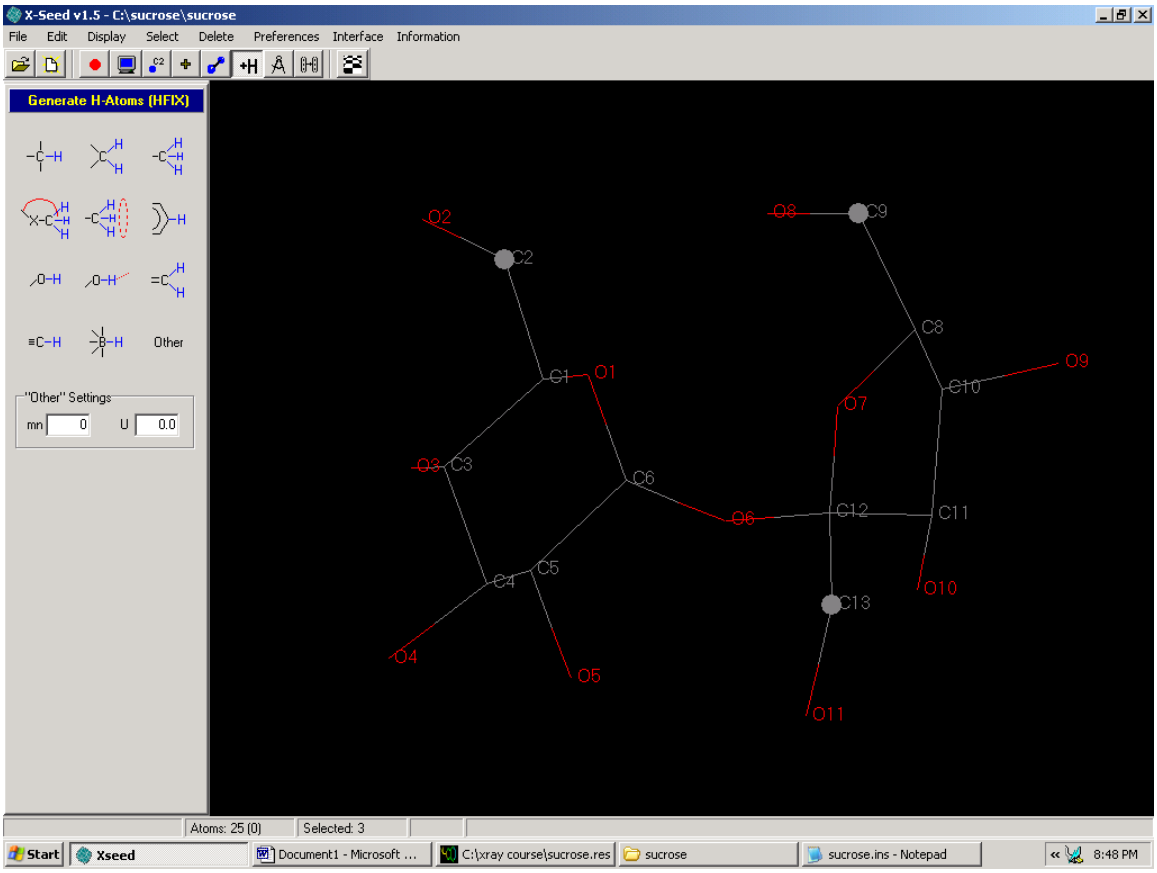


For now point to Delete and choose “all Residual Peaks” or just type a Q. If any residual peaks are left then goto the red dot icon, select the residual peaks and type the delete key.

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

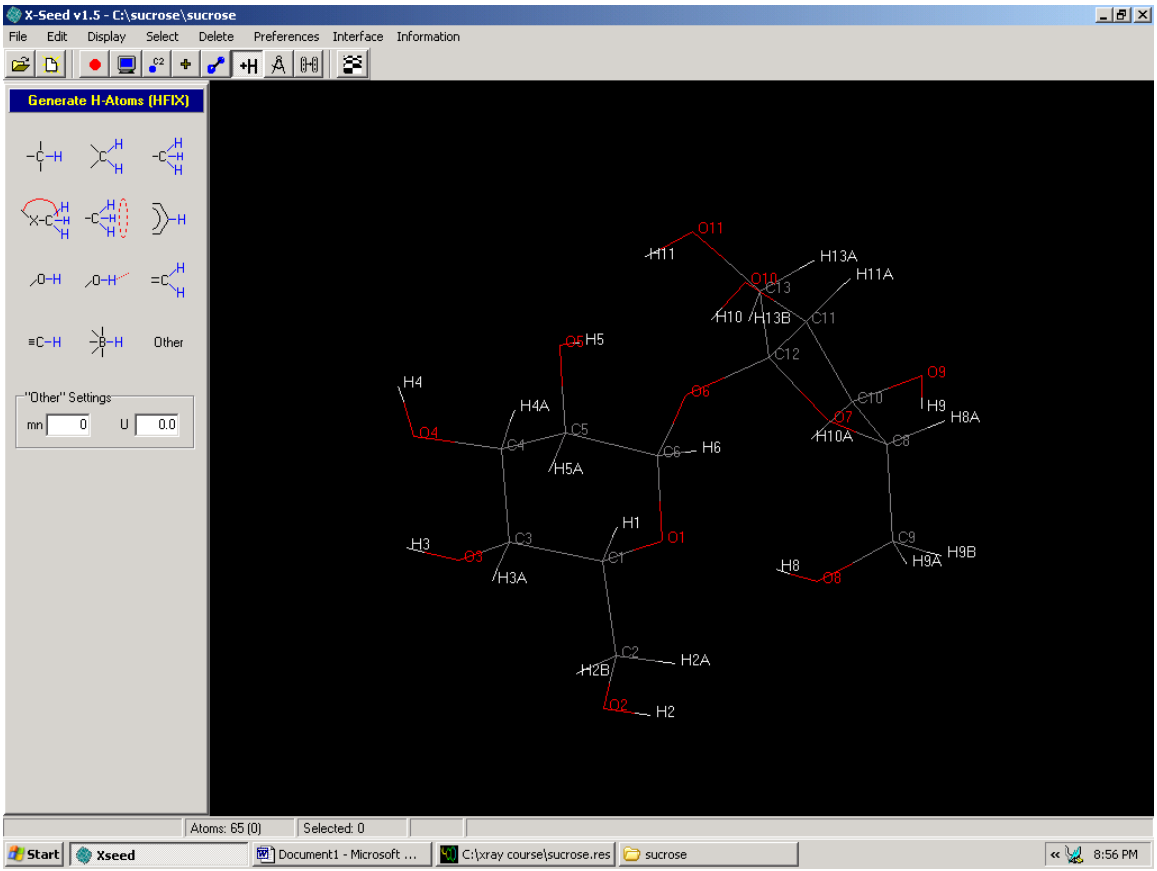


When complete type the –C-H icon. The selection will disappear. Now select the secondary Carbons



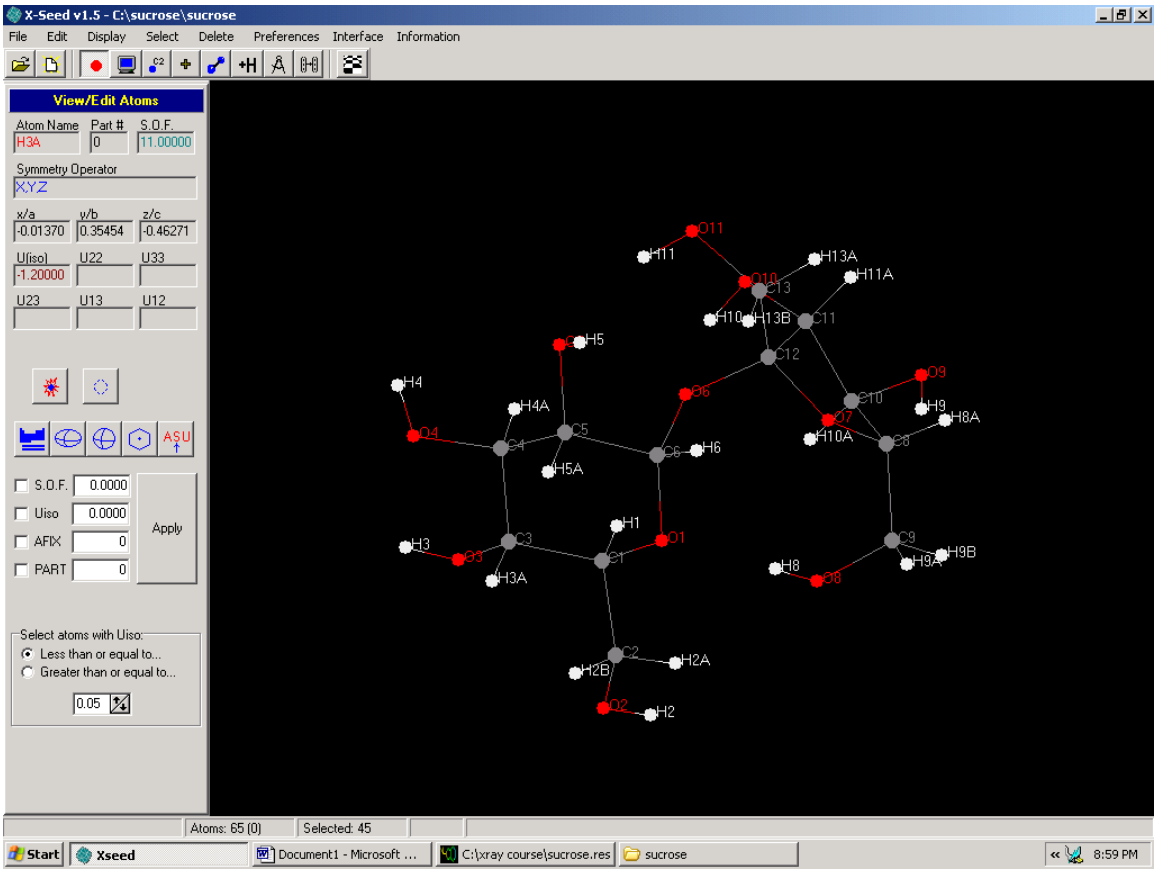
Type the CH2 icon. The selection will disappear. Now select the terminal oxygens. Type the –O-H icon (not the –O-H…. icon). The selection will disappear.

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.

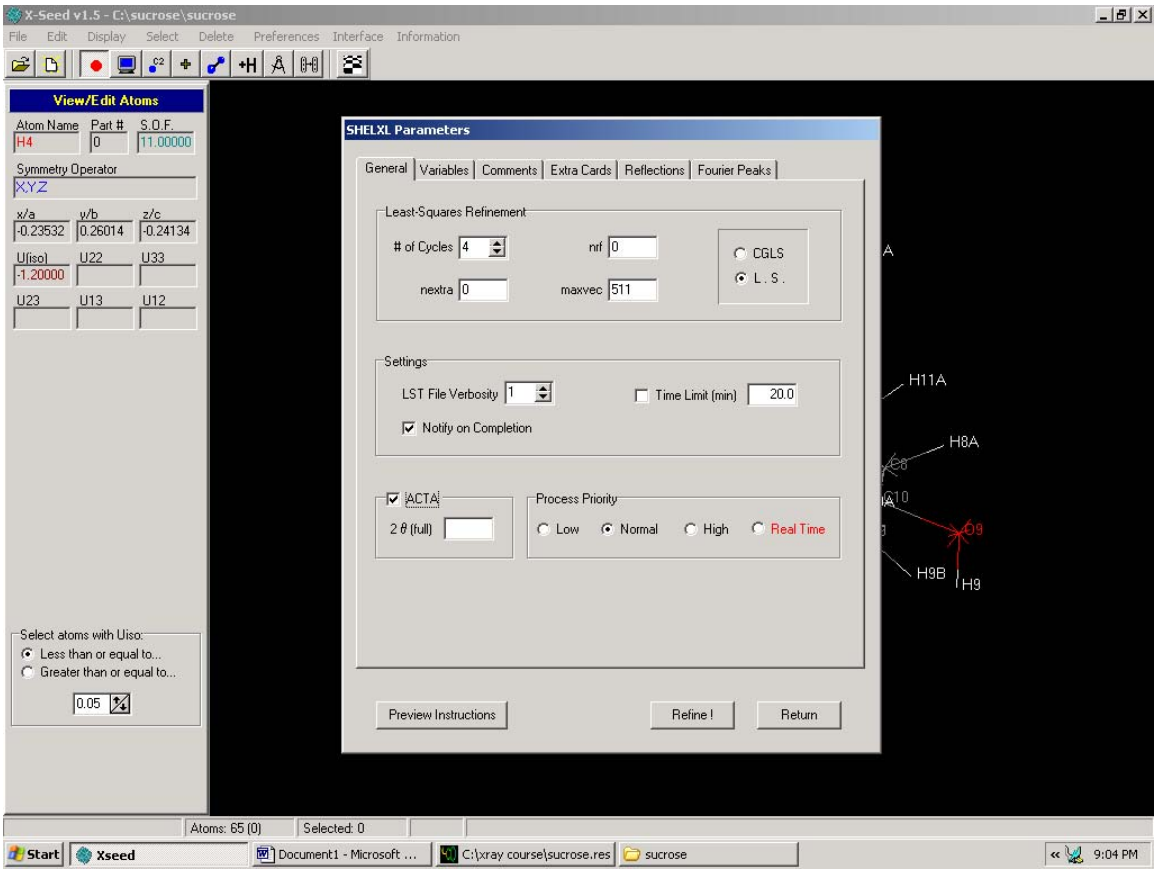
Next let's go anisotropic on all non-hydrogen atoms. To do so select all atoms. Under the select menu goto to select all and atoms. Return to the red dot icon and select the icon that looks like a football.



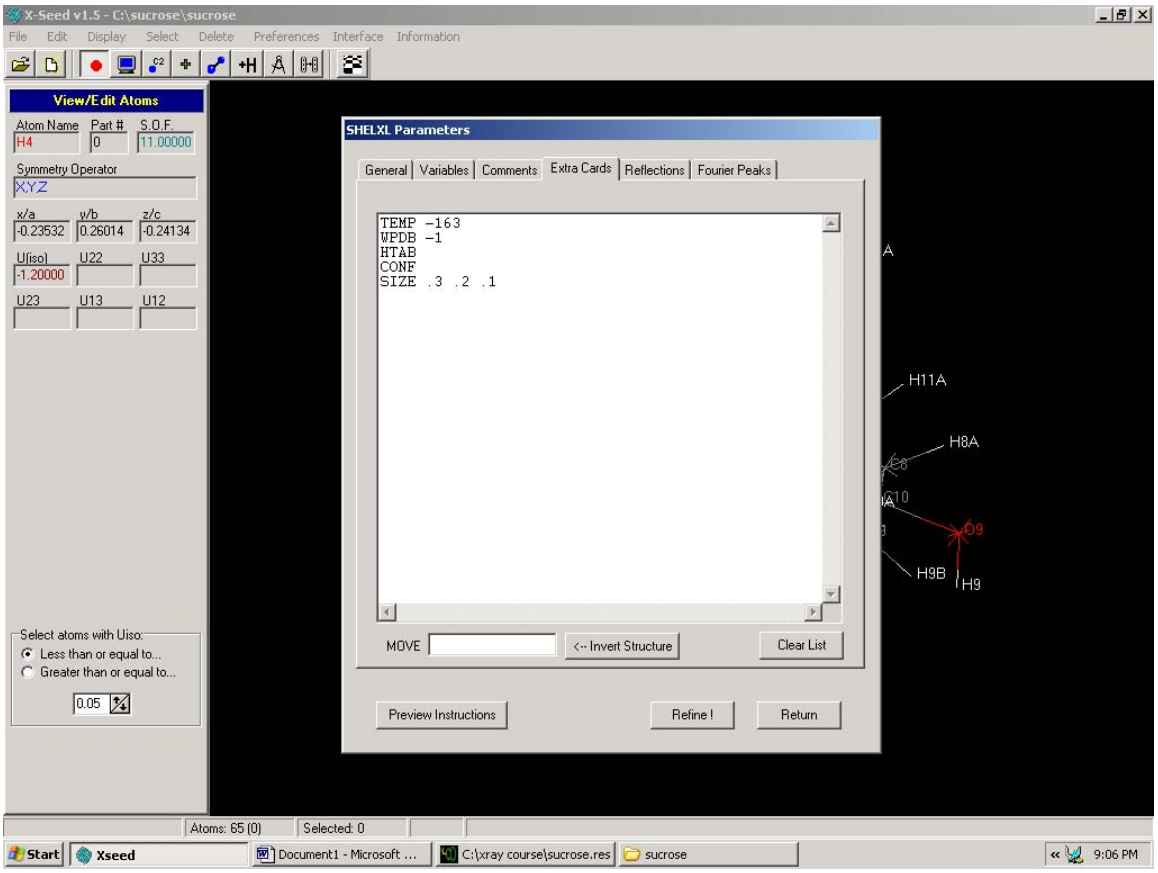
Your selections will disappear. Now go back to interface and SHELXL again. Refine the structure. Notice the R1 factor should be near .06.

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

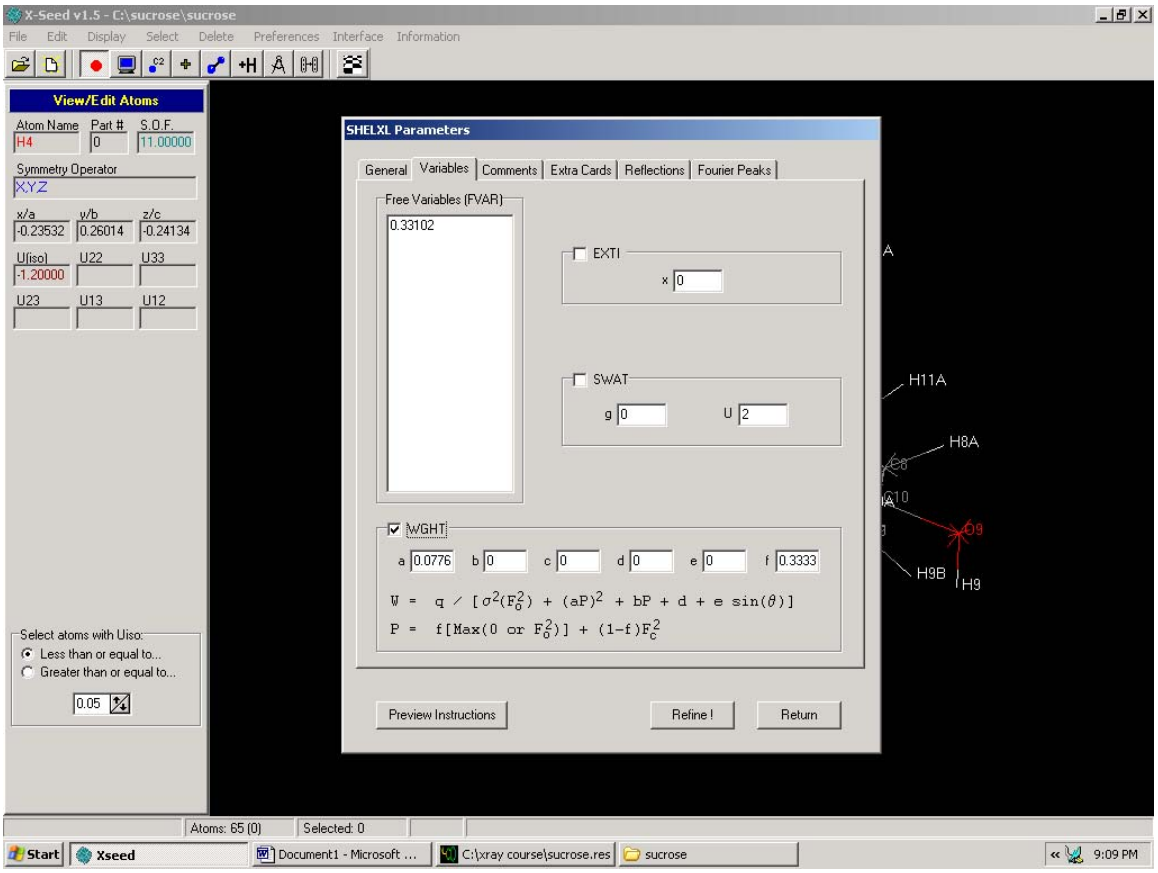


goto the Extra Cards Tab and add these commands

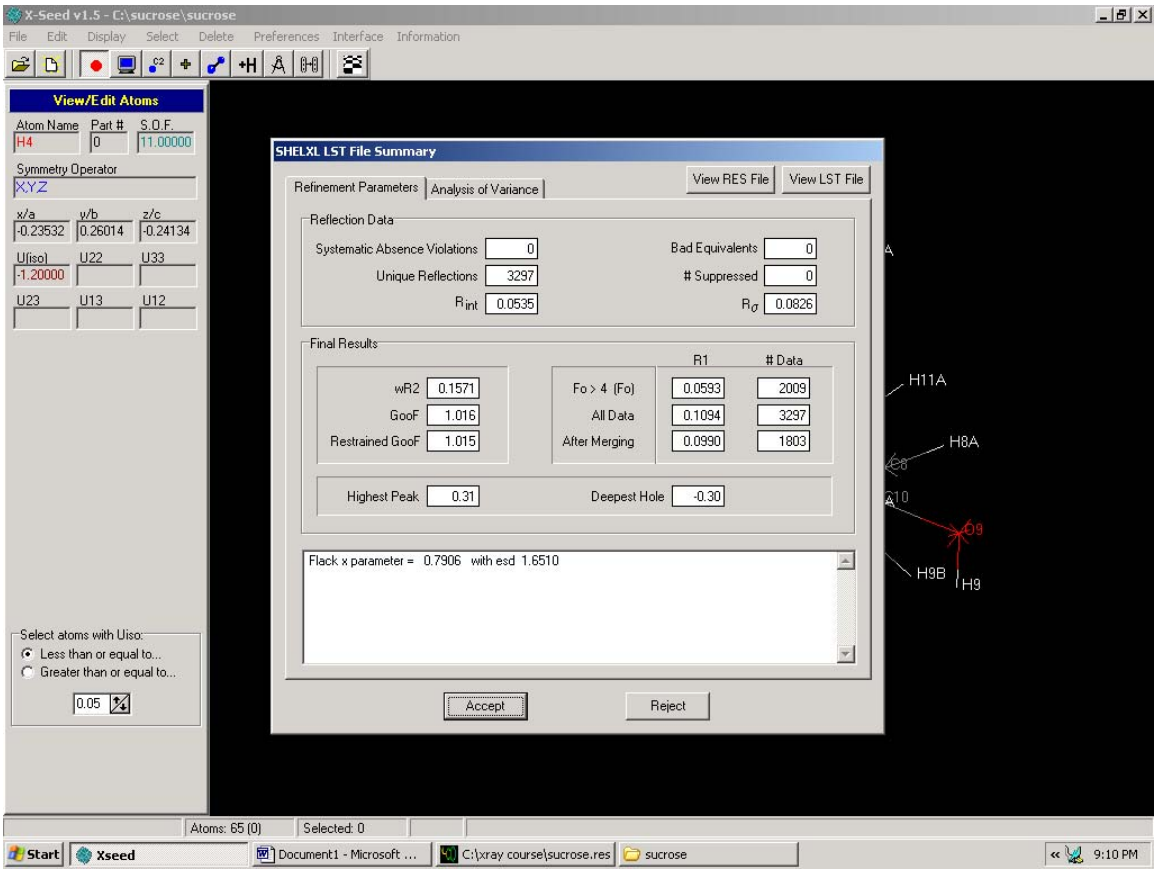


TEMP –163
WPDB –1
HTAB
CONF
SIZE .3 .2 .1

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

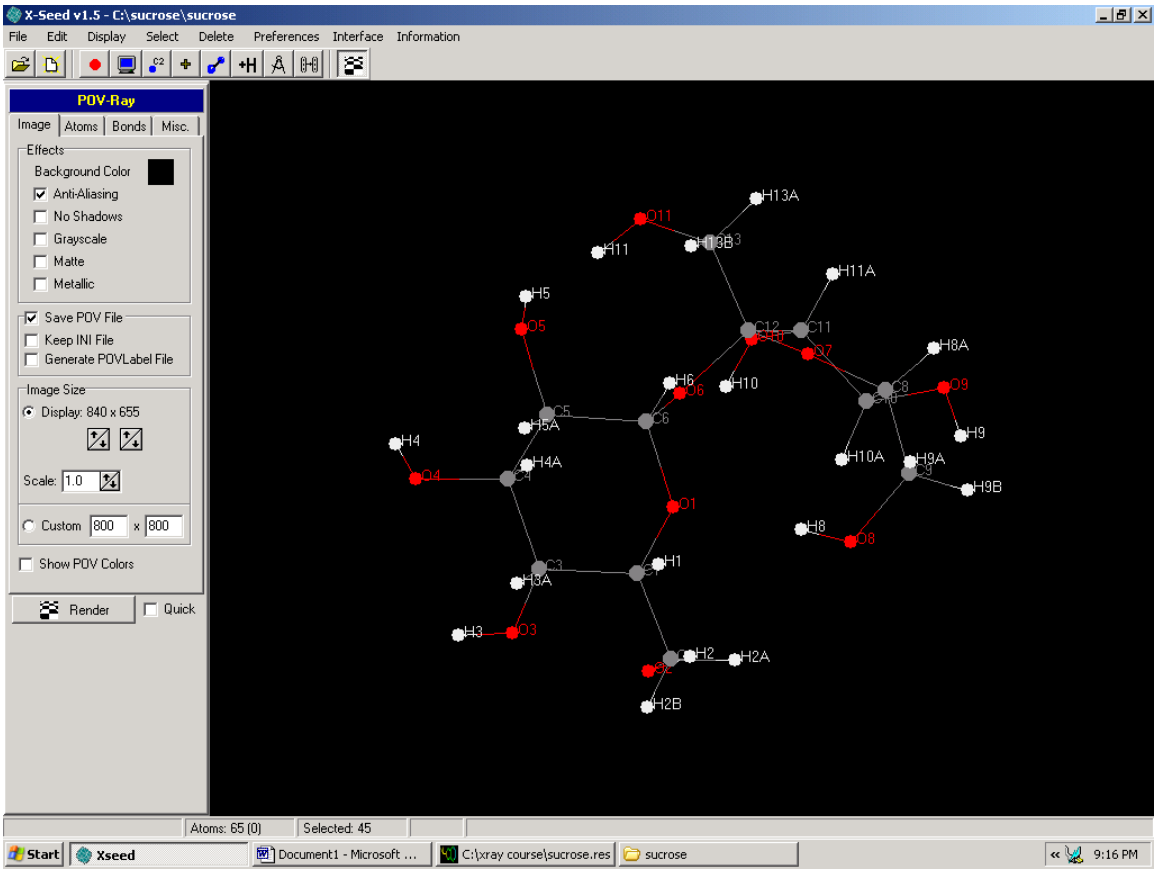


Now refine. The summary should look like this

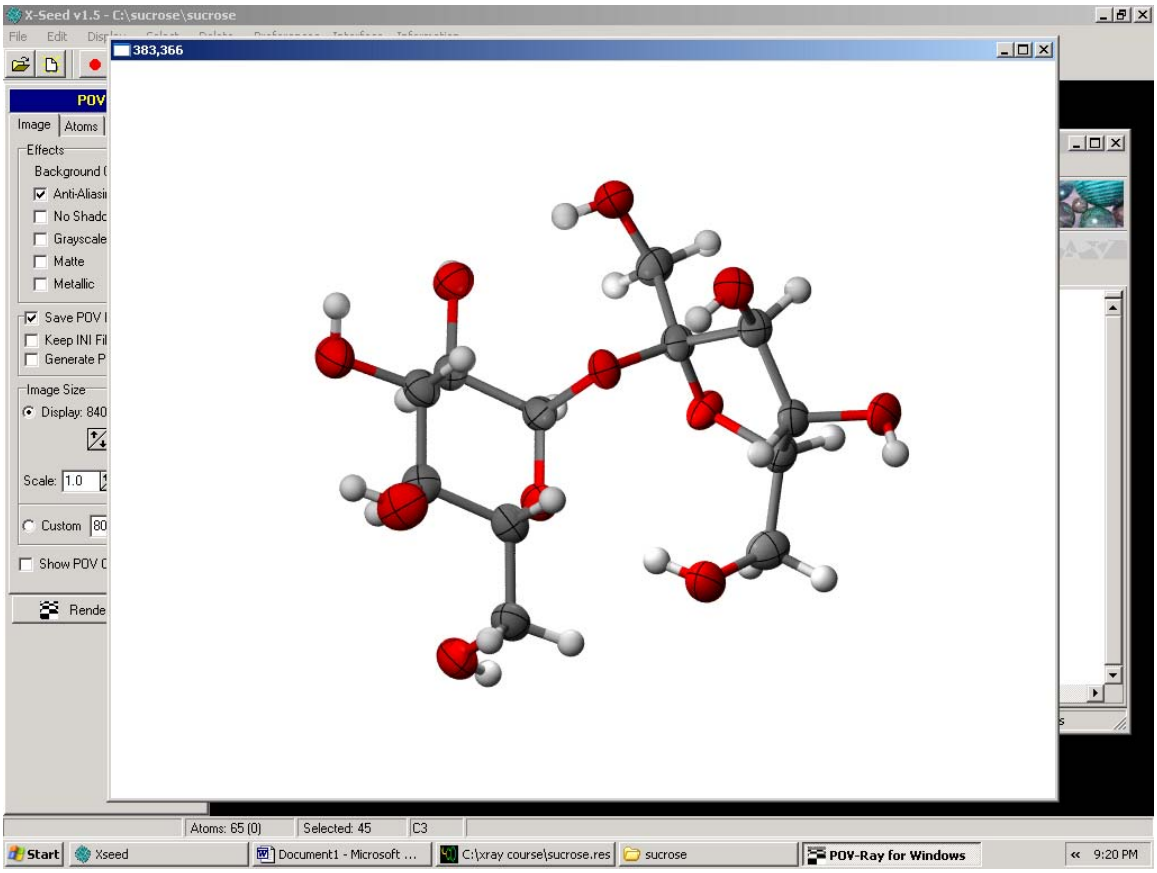


The R1 factor is 0.0593 and the wR2 is 0.1571. The highest peak is 0.3 electrons Accept the results and delete the residual peaks.

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

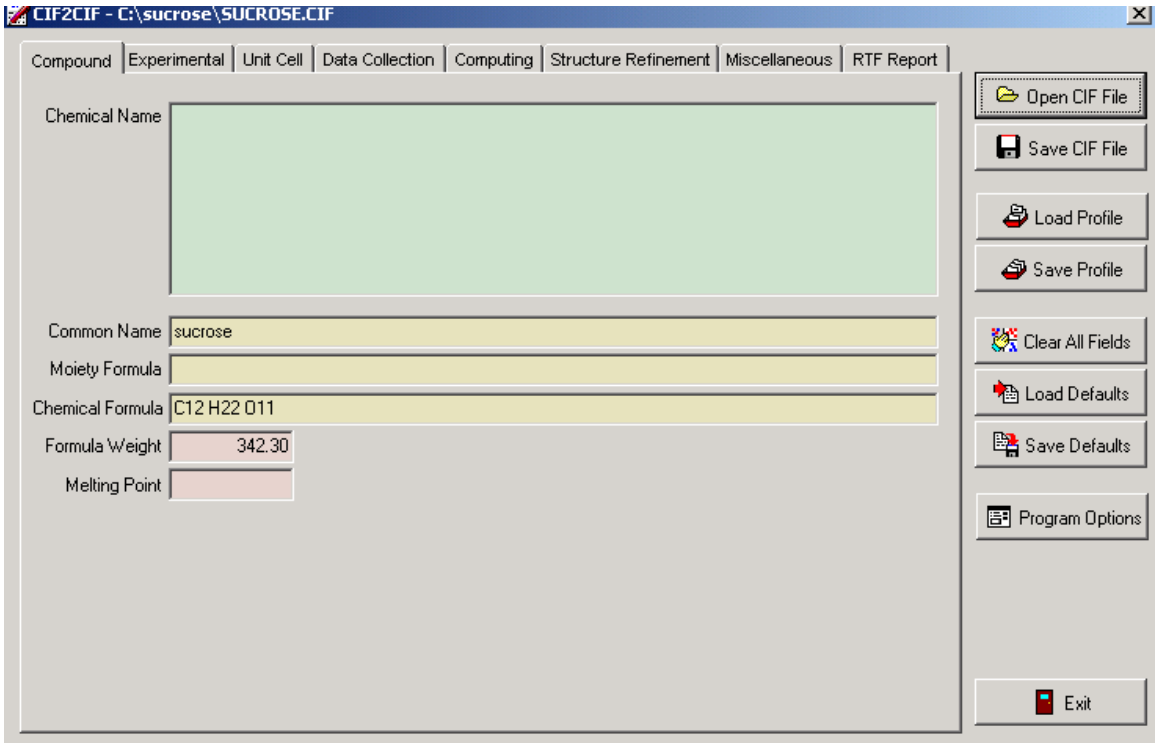


Save the file

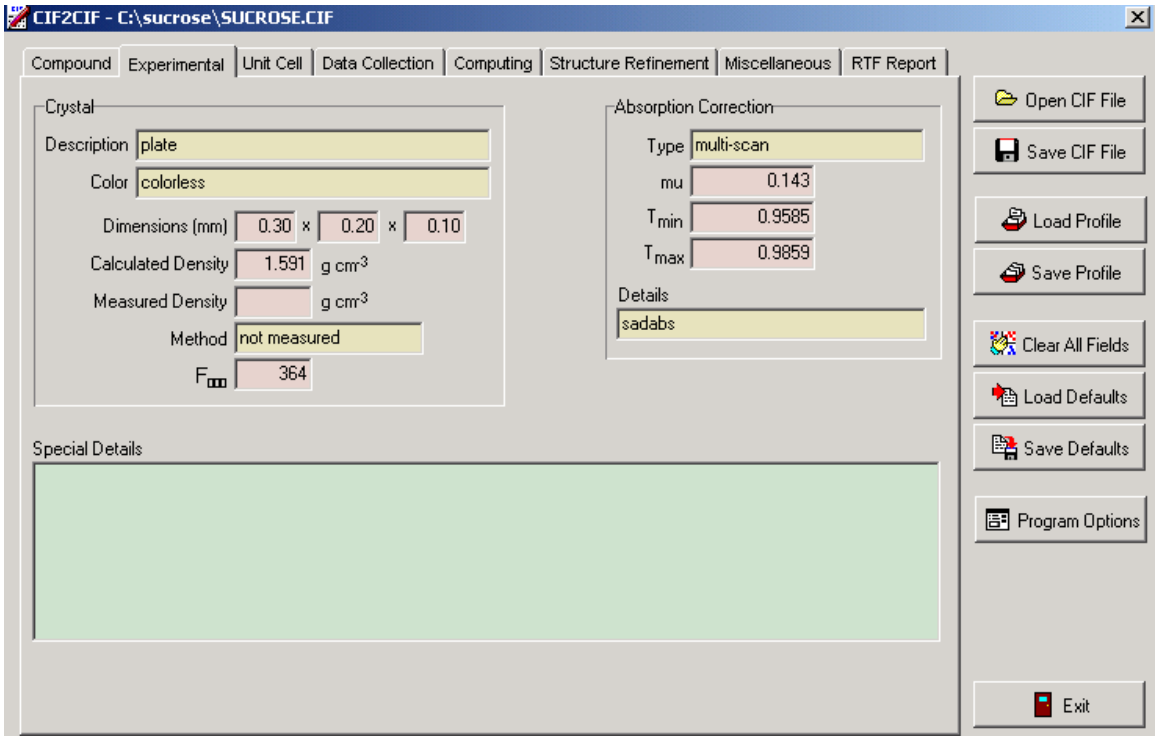


Close POV-RAY and X-SEED.

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